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THE MANUFACTURE OF
EXPLOSIVES

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THE MANUFACTURE OF EXPLOSIVES

A THEORETICAL AND PRACTICAL TREATISE ON THE HISTORY,
THE PHYSICAL AND CHEMICAL PROPERTIES, AND
THE MANUFACTURE OF EXPLOSIVES

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The figures 1, 15, 16, 80 to 82 incl., 100, 101, 111, 117, 118, 133, 138 to 140 incl., 146, 147, 263 to 266 incl., 270, 271, 273, 274, 277, 278, 281 to 289 incl., 295 to 298 incl., are taken from Dr. J. Upmann, 'Das Schiesspulver.'

" " 169, 175, 176, 245 to 251 incl., from Dr. E. v. Meyer, 'Die Explosivkörper und die Feuerwerkerei.'

" " 3 to 6 incl., 18, 19, 31, 158, 159, 194, 211, 219, 222 from Muspratt, 'Theoretische und praktische Chemie.'

" " 8 to 13 incl., 17, 87, 104, 108, 123, 132, 145, 268, 302, 303 from E. Désortiaux, 'La Poudre, les Corps Explosifs, et la Pyrotechnie.'

" " 20 to 30 incl., 151 to 157 incl., 167, 168 from German patent publications.

" " 32 to 36 incl., 66, 218, 235 to 240 incl., 319, 320 from English patent specifications.

" " 241 from the Austro-Hungarian patent publication.

" " 37, 38 from Dr. G. Lunge, 'Coal Tar Industry.'

" " 149 from Dr. G. Lunge, 'Sulphuric Acid.'

" " 39, 40, 304 to 309 incl., 311, 321, 322 from the 'Mittheilungen des k. k. techn. u. adm. Militär Comité's.'

" " 106, 107, 327, 328 from the 'Mémorial des Poudres et Salpêtres.'

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" " 98, 99, 276 from the 'Preussisches Handbuch für Kriegsfeuerwerkerei.'

" " 86 from Witte, 'Waffenlehre.'

" " 79, 83 from Böckler, 'Theatrum Machinarum Novum.'

" " 269 from Furttenbach, 'Büchsenmeistereischule.'

" " 141, 272, 294 from Post, 'Chemische Technologie.'

" " 211 to 213 incl., from the 'Journal of the Society of Chemical Industry.'

" " 258 to 262 incl., from Karmarsch u. Heeren, 'Technisches Lexikon.'

" " 279, 292, 293 from Maudry, 'Waffenlehre.'

" " 274 from Castner, 'Das Schiesspulver.'

" " 290, 291 from Sebert, 'Essais d'Enrégistrement.'

" " 310 from Quinan, 'Crusher and Cutter Gauges.'

" " 121, 122, 183, 187 to 190 incl., 242 to 244 incl., 323 to 326 incl., from English official publications.

" " 85, 95, 124, 128, 148, 163, 196 to 198 incl., 223, 224 are blocks of the makers.

Of the remaining 142 figures, 41 were re-drawn from sketches and plans of makers and others, whilst 101 figures were made from own drawings.

THE MANUFACTURE OF EXPLOSIVES

CHAPTER VIII

GUN-COTTON

1. MANUFACTURE

The manufacture of gun-cotton and of nearly all nitro-compounds consists essentially in immersing the prime material (cotton, glycerin, and similar materials) in a mixture of nitric and sulphuric acids and allowing the nitric acid to act upon it for a more or less prolonged time. The explosive body thus formed is then separated from the spent acid, and washed, with the addition of alkalies as a rule, until it has lost all traces of acidity.

As an example to illustrate the action which occurs, the following formula expressing the formation of gun-cotton may be taken:—

$$C_{12}H_{20}O_{10}+6HNO_3=C_{12}H_{14}O_4\ (O.NO_2)_6+6H_2O.$$

From this it will be seen that six atoms of nitrogen are replaced by six equivalents of the nitro-group NO_2, six equivalents of water being at the same time formed. In order to prepare a nitro-compound, it would seem at first sight that nitric acid only is necessary for the reaction; but water is formed at the same time which would constantly dilute the remaining nitric acid, from the action of which lower nitro-compounds would be obtained. It is therefore necessary to add to the nitric acid a considerable quantity of sulphuric acid, the object of which is solely to take up the water formed during the reaction, and so maintain the nitric acid at its original state of concentration.

The inventor of gun-cotton, Schönbein, used at first a mixture consisting of three parts by weight of sulphuric acid of 1·85 specific gravity and one part of nitric acid of between 1·40 and 1·50 specific gravity. Into twenty to thirty parts of this mixture contained in a porcelain vessel he dipped one part of cotton at a temperature of from 10° to 15° C., and allowed the reaction to proceed for an hour. The liquid was then poured off, the gun-cotton thoroughly washed, and in order to eliminate the acid, again washed with a dilute potash solution. It was then again washed with water in order to dissolve the salts formed, squeezed out and soaked in a 0·6 per cent. solution of saltpetre, squeezed out again, and finally well dried at 65° C. Later Schönbein modified this process by using 14 parts of a mixture of equal volumes of nitric and sulphuric acids to each part of cotton. Dr. Gladstone obtained by Schönbein's process 173 to 175 parts of gun-cotton from each 100 parts of cotton treated.

Otto dipped cotton for half a minute into concentrated nitric acid made from 10 parts of saltpetre and 6 parts of sulphuric acid. The product was thoroughly washed and picked to pieces, since it became strongly felted together in the dipping process. Karmarsch and Heeren, and also Knop, worked in a similar way to Schönbein.

According to Walter Crum's method, the carded cotton was first purified in a dilute solution of soda, then bleached with a solution of bleaching powder, and after being dipped for a short time into diluted nitric acid, it was well washed with water. By this process the cotton lost 5·6 per cent. of its weight. A sample of the cotton so purified gave only 0·9 per cent. of ash. The cotton was then dried on a steam stove and well distributed in a mixture of 1 volume of sulphuric acid of 1·84 specific gravity and 3 volumes of nitric acid of 1·517 specific gravity. For each part of cotton 48 parts of the acid mixture were used. After the gun-cotton had been carefully washed, it was dried in the open air. By this process 178 parts of gun-cotton were obtained from 100 parts of dry cotton.

Kerkhoff and Reuter used 1 part of cotton to 48 parts of a mixture of equal parts by weight of strong nitric and sulphuric acids, by which means they obtained 176 parts of gun-cotton from 100 parts of cotton treated.

At Le Bouchet, the following process was formerly used:—100 grammes of cotton in the form of wadding were dipped into 1 litre of a mixture of 3 volumes (or 1 part by weight) of nitric acid of 1·500 specific gravity and 7 volumes (or 2·86 parts by weight) of concentrated sulphuric acid contained in an earthenware vessel. After having been dipped, another litre of the acid mixture and another 100 grammes of cotton were added. The vessel was then covered, and the acids allowed to act on the cotton for an hour, after which the gun-cotton was squeezed in a press, which removed about $\frac{7}{10}$ of the acid. It was next washed in wicker-baskets placed in running water for one and a half to two hours. After being again pressed, it was first washed in water made alkaline by wood-ash lyes, and then with pure water, again pressed and finally dried on linen cloth in a slightly-heated current of air. The yield was on an average, 165·25 parts of gun-cotton for each 100 parts of dry cotton.

Von Lenk's process was as follows:—The cotton was spun into hanks of about 85 grammes, and suspended on hooks in a hot solution of potash. An under-heated iron boiler was used for the purpose, the water in the boiler containing sufficient potash to give it a density of 3° B. (1·022 specific gravity). The cotton remained in the boiling solution for from two to three minutes, according to the percentage of grease it contained. After having thus saponified the grease, the potash solution was driven out of the hanks of cotton as completely as possible by means of a centrifugal machine. They were next purified from the soap-liquid formed by swinging them to and fro in water in perforated zinc baskets, and then again wrung out and finally perfectly dried.

The acid mixture was made by allowing nitric acid of 1·48 to 1·49 specific gravity (at 17·5° C.) and sulphuric acid of 1·835 specific gravity to run in thin streams from two taps into an earthenware vessel. The proportion was usually 1 part of nitric to 3 parts of sulphuric acid. The mixture so prepared was allowed to cool in earthenware jars in which it was stored.

The nitration was done in cast-iron trough-shaped dipping-pans, divided into three compartments, with a grating fixed over the middle one. Two hanks at a time were put into 300 times their weight of the acid mixture in the first two compartments of the dipping apparatus, and were turned over and squeezed by spatulas until the acid had thoroughly penetrated the cotton hanks. They were next transferred to the grating and freed from the excess of acid by squeezing, the cotton being allowed to retain about 9½ parts of acid. When about 2 kilogrammes of cotton had been treated, the acid mixture was completely renewed. When six hanks had been nitrated, they were put into an earthenware pot standing in the third compartment of the dipping apparatus. A weighted disc was put into the pot to submerge the cotton in the acid, and the pot itself closed with a lid and allowed to stand for between twenty-four and forty-eight hours in a special room, the temperature of which was not allowed to fall below 5° nor rise above 25° C. In order to maintain the temperature within these limits, the room had to be heated in winter, and the exterior of the pots cooled by running water in summer. During the first two to six hours the pots had to be watched, and heating was prevented either by adding some fresh acid or passing cold water round them.

After the nitration was finished, the crude gun-cotton was freed from the excess of acid in centrifugal machines, then washed with a large quantity of water in copper washing-drums, and finally treated in running water in special washing-boxes for six weeks. It was then wrung out in a centrifugal machine, treated with a boiling potash solution of 1·02 specific gravity, then again "centrifugaled," washed with water, centrifugaled, and then dried. After this it was dipped into a solution of sodium silicate of 1·072 specific gravity, centrifugaled, and finally exposed to the air for three days.

During this time the sodium silicate was decomposed by the action of the carbonic acid in the air, and silica or an insoluble silicate precipitated on the fibre of the gun-cotton (the so-called "silicating process"). Finally the product was again washed in running water of as soft a nature as possible, again centrifugaled, and then dried in the open air, or in a drying-house, at a temperature not exceeding 35° C., direct rays of the sun being avoided. The yield of gun-cotton, according to the experience of many years, was, on an average, from 165 to 167 parts for each 100 parts of dry cotton. The structure of the finished gun-cotton was carefully examined and the gun-cotton tested by the rod gauge for strength. On the latter 0·39 gramme (6 Austrian grains) should give 42°. A product of inferior strength was only issued for use in blasting operations.

In the examination of the physical properties the retention of the original structure was considered of importance, and hanks containing torn threads were eliminated as unserviceable.

All the above processes, however, only gave imperfect gun-cotton. Although the original processes by Schönbein, Otto, etc., were only used for manufacture on a small scale, considerable quantities were made by Von Lenk's process; but since the gun-cotton maintained the structure of the cotton used, its purification was of necessity imperfect, and several explosions which happened later on caused the manufacture by Von Lenk's process to be given up.

The real initiative to the successful modern manufacture of gun-cotton was given by Sir Frederick Abel, who insisted on the necessity of a thorough division of the gun-cotton in order to free it completely from every trace of acidity. His process is still the one in use, although many modifications have been made in the apparatus and operations of manufacture.

(*a*) PREPARATION OF THE RAW COTTON.

The cotton, which is either supplied in a purified state or purified in the factory itself by means of soda solution, etc., is first hand-picked, in order to eliminate nails and other foreign bodies which might accidentally have got into it, and is then teased in an "opener" or "willow."

This machine consists of a large drum, on the periphery of which are a large number of sharp-pointed teeth. Opposite to the main drum stands a smaller one, also provided with similar teeth. Another pair of small rolls feed the cotton waste to the toothed drums, which tear its threads apart and deliver the cotton in the form of wadding. A second pair of carding rolls are frequently provided behind the toothed ones, in order to completely separate the fibres of the opened cotton.

A willow made by Mr. James Tomlinson, of Rochdale, which is illustrated in Fig. 148, is used in several large factories. Over the main drum are several small rolls for removing the teased cotton from the drum. All these rolls are actuated by an endless chain on this machine, which is also provided with a fan for exhausting the dust formed. A similar machine with six drums is said to be used in the German Government factory at Hanau.

Another form of willow is also used in Germany. It consists of a large wooden disc, on which a number of saw-toothed blades are arranged radially. The cotton is pressed by a small roll through a slot in the cover against these blades, which, as they rotate, tear the cotton asunder. At Waltham Abbey the cotton, after having been teased, is cut into short lengths, about 2 inches, by means of a kind of guillotine machine, so that it may pass through the drying machine more easily, and also because in this form it is more convenient to nitrate.

The cotton is then dried at about 212° F. until it contains a minimum of moisture. The simplest method of drying is in wooden cupboards containing a number of wire-gauze trays on

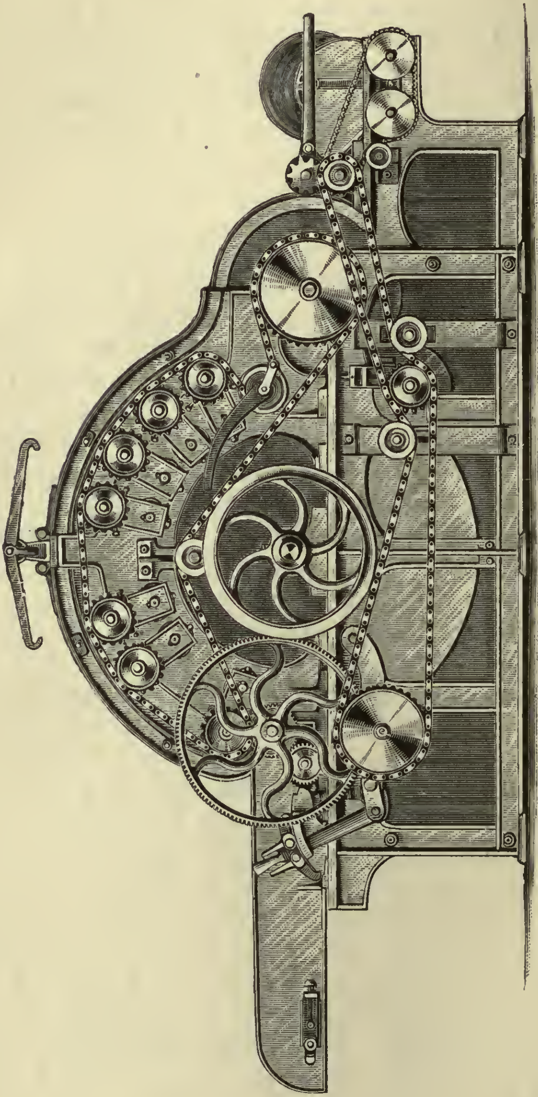

Fig. 148.

which the cotton is laid, the heat being supplied by steam-pipes placed in the lower part of the cupboards. Sheet-iron cupboards heated direct by a fire have been used, but they are rare.

At Stowmarket and Moulin Blanc, the cotton is put into iron cylinders fitted with a lid. A pipe passes from the lid to a vacuum pump, by which means the drying is done very rapidly, although at a somewhat greater cost.

At Waltham Abbey a mechanical drying-stove is used. It consists of a high oblong rectangular casing of sheet-iron, in which, at short distances from each other, endless bands are stretched over two rolls, one of which is movable for tightening the band, while the shaft of the other passes through the casing and has a small pulley fixed on to it. The endless bands are so arranged that one end of them stands about 2 feet further from the end of the casing than the band immediately under it. By this means the cotton falls from the upper band on the one below. The pulleys are all geared together by an endless band in such a manner that they travel alternately in opposite directions. The consequence is that the cotton falling from the one band on to the next is carried back in the opposite direction to its travel on the first one, and so on. A double partition formed of two plates is placed between each pair of bands, through which steam passes and heats the stove, while at the same time a fan blows air into the casing from below. A dust-settling chamber is placed in front of the casing to prevent fine particles being carried away. The lowest band delivers the cotton to an endless band-conveyer, which carries it to the cooling-chamber. The rate of travel of the cotton in the stove is about 6 feet per minute, and the total length of travel 126 feet, so that the time of drying is about 21 minutes. In the cooling-chamber the cotton is put, in charges of 1¼ lbs., into tin boxes with tightly-fitting lids, in which it remains over-night to ensure perfect cooling.

A simple and very effective way of drying, especially for large quantities, has been used by the author. It consists of drying the cotton in cupboards made of wood or sheet-iron, provided in the usual way with wire-gauze trays. Each frame is separated from the other by a fixed horizontal partition. On each side of the cupboard pipes are fixed, or channels left open in the cupboard itself, which communicate on the one side with the free space below the frames, and on the other with that above the frames. Hot air is then blown in by means of a fan, while the moisture thus driven out of the cotton on one frame cannot get to the other frames on account of the horizontal partitions. The air is also forced to penetrate the cotton from below, and can only leave the cupboard above the cotton. The current of hot air introduced into the cupboard is produced by a fan, and passes over a steam stove, or other heating apparatus, before entering the cupboard. The author has found Koerting's gilled pipes best for such steam stoves, since they have a large heating surface, and can be arranged in any number together, according to the quantity of air blown in. They may be enclosed in an iron casing lined with some non-conducting material, or may stand in a brick chamber. With a proper arrangement and sufficient quantity of heating surface, it is possible to introduce air of 90° to 95° C. (194° to 203° F.) into the cupboard. In order to further hasten the drying operation, the air itself may be first dried over calcium chloride, or sulphuric acid, for which the apparatus illustrated in the chapter on Black Powder, vol. i. p. 228, may be used.

After drying the cotton is weighed out in charges of suitable weight, and at once put into tightly-closed receptacles. The weight of a charge of cotton varies in different factories. At Waltham Abbey, as before mentioned, it is 1¼ lbs.; in Germany, France, and Italy, as a rule, 4·4 lbs. (2 kilogrammes); or, when centrifugal nitrating machines are used, 17·2 lbs. (8 kilogrammes).

(*b*) PREPARATION OF THE ACID MIXTURE.

The proportions of the acid mixture used by Von Lenk, namely, 1 part of nitric acid and 3 parts of sulphuric acid, both of maximum concentration (at least 93 per cent. of monohydrate for nitric acid, and at least 95 to 96 per cent. for sulphuric acid), are still generally adopted. As a rule, weighed quantities of the acids are put into large cylindrical vessels of cast-iron fitted with a stirring-gear, which immediately mixes them together. The stirring-gear is moved by an eccentric, giving it a forward and backward movement. From the mixer the acid runs into a number of cast-iron vessels, in which it is cooled down and drawn off for use as required. In some factories the acid mixture is prepared by simply placing weighed quantities into a mixing vessel made of lead or cast-iron. If the whole of the nitric acid is put into such a vessel, and then the sulphuric acid run in on the top of it, the two acids will mix instantly; and experiments made by the author show that they do not separate again, but that the proportions of the two acids in the upper layers are exactly the same as in the lower ones. The acid mixture is run from these mixing vessels through a cooling-box into a storage vessel, or if it is to be carried to a higher level or to a great distance, it is run into an acid elevator (*montejus*).

Such acid elevators are illustrated in Figs. 149 and 150. Fig. 149 is an acid elevator as used by many chemical and explosives works. It was first described by Professor Lunge, in his 'Handbook on Sulphuric Acid and Alkali.' It is a horizontal cast-iron cylinder (A), hemispherical at one end, and closed at the other by a blank flange (a). The walls of the cylinder are, as a rule, from 1½ to 3 inches thick, and the ends rather more. There are three sockets on the upper part, through one of which (f) an iron or lead pipe (g) passes into a small pocket (c) at the bottom of the vessel. This pocket is cast on to the cylinder, in order that the whole of the acid may be pumped out. The pipe (g) is connected with the main acid pipe. A second socket (d) serves for running in the acid mixture, and is closed by means of a blank

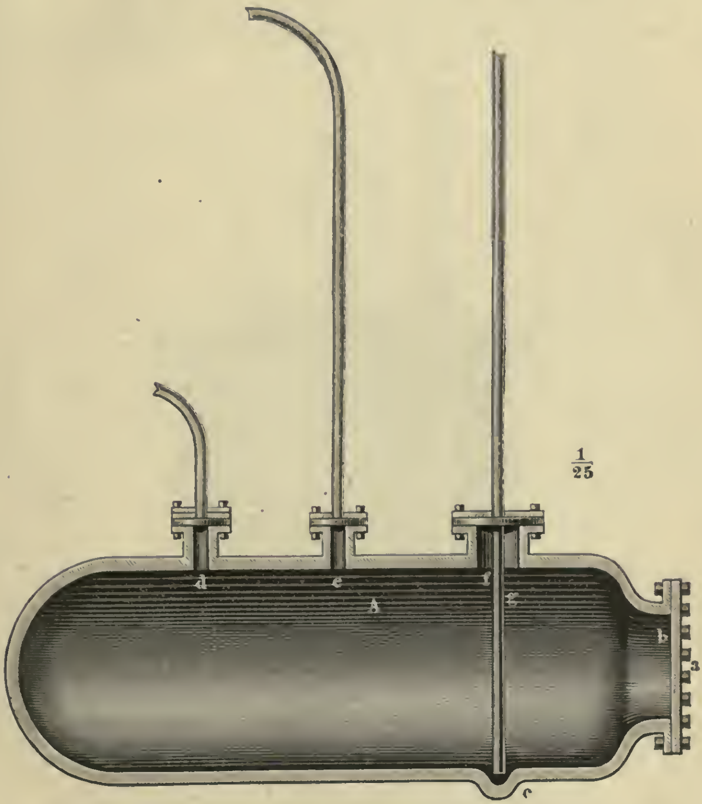

FIG. 149.

flange when the vessel is full, or it is permanently connected with a storage vessel by means of a tap. The third socket (e) is for the compressed air inlet, which has at some suitable point a small T-piece attached to it. Both the main air-pipe and the T-piece are fitted with taps. After the vessel has been filled with acid the charging-hole is closed, the tap on the T-branch of the air-pipe is shut off, and that on the main air-pipe opened. The compressed air enters the apparatus and forces the acid out into the main acid pipe. As soon as all the acid has been ejected, the air inlet pipe is closed, and the tap on the T-branch opened, to allow the compressed air in the vessel to escape.

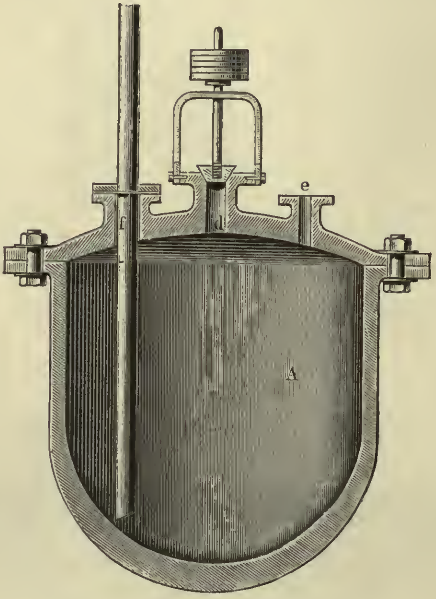

Fig. 150.—One-25th full size.

Another kind of acid elevator which is much used is illustrated in Fig. 150. It is a vertical cylinder fitted with a safety-valve to relieve the vessel in case of excessive pressure. It is closed by a dished cover which can be removed for cleaning out the apparatus. The best method of making the cover joint tight is to use a length of thin lead pipe as packing between the cover and the vessel. The pressure pipe does not reach to the lowest point, so that any dirty matter which may have collected at the bottom is not forced into the acid main.

Acid elevators are very convenient means for moving acid from one place in a factory to another. The air can be introduced into them either directly from an air-pump, or, which is more convenient, from an air storage vessel in which case it will enter the elevator in a uniform stream and no excessive sudden pressures are set up. On account of the fluid friction in pipe lines, the high specific gravity of the mixed acid, and the very high viscosity of the sulphuric acid, the mixed acid, requires a pressure of at least 60 lbs. per square inch to lift it to from 40 to 50 feet high. If the acid has to be lifted beyond this height, it is more convenient to use two acid elevators and effect the lift in two stages, since compressed air can be conveniently carried to very great distances and considerable heights, without serious loss of pressure, which is not the case with acids.

Thus compressed air and acid elevators, placed at various intermediate points, form the best means for conveying liquids such as acids to considerable distances and heights. Great care has to be taken to lay the pipe-lines in a uniformly ascending slope. Bends and sharp curves should be avoided as much as possible, and at the highest point an arrangement must be provided for automatically carrying away any air that may have collected there, and which might otherwise give rise to dangerous shocks. What great trouble may arise from this cause was shown in a pipe-line which, though otherwise well arranged, bent downwards at its upper end to discharge the acid into the vessel. The air collected at this point from time to time, and the acid elevator repeatedly broke down, and these accidents only ceased when the pipe was led into a blow-out hood. The walls of lead pipes for conveying the acids should be at least $\frac{3}{8}$ inch thick. The initial cost is, of course, greater, but the pipes last very much longer, and are much less likely to get bent or otherwise damaged. Such pipe-lines are, as a rule, carried on planks placed on edge and fixed on poles at about 10 feet centres. They are secured by lead straps nailed to the planks. In places where severe winters occur it is necessary to protect the pipes with some non-conducting material, or to lay them in channels filled with cinders or similar material, since otherwise, probably on account of the dilution of the acid by the access of air when out of use, the pipe gets frequently frozen.

In a few special cases, where smaller quantities of acids have to be dealt with, earthenware acid elevators can be used. Professor Lunge has also published descriptions of continuously-acting acid elevators, but their use presents no advantage for pumping the acid mixture used for nitration, because the mixed acid has to be stored in vessels, as it is not used continuously.

The acid mixture is drawn from the storage vessel, either directly, or by allowing it to run through a pipe-line into the nitrating vessel up to a fixed mark. If large quantities are used at the same time, the acid mixture is run into a tank standing on a weighing machine. The filled box is weighed, and then weights corresponding to the quantity of acid to be used are taken off and the acid allowed to run out of the tank through a tap until the weighing machine is again balanced.

The acid mixture must be stored in well-closed vessels or at least be so protected that rain and organic matter cannot get in, since it would otherwise decompose. Experiments made by the author have shown that as soon as the temperature of the acid mixture rises above 50° C., the probability of a decomposition, especially on introduction of organic matter, becomes very great. A decomposition is accompanied by the rapid development of large quantities of hyponitric acid fumes, violent boiling up of the mixture, and sometimes by a blackening of the sulphuric acid remaining, whilst at the same time the temperature increases rapidly.

(c) NITRATION.

For the nitration of cotton a larger quantity of acid is always required than for that of other organic bodies. The cause of this is that after carding the cotton becomes very bulky, and since it is necessary for the acid mixture to thoroughly penetrate and always cover it, and since it takes some time for the acid to be completely absorbed, it follows that the acid mixture must always be a multiple of the quantity of cotton. In general, about 30 times the weight of cotton will be required, but it is of advantage to work with 50 times the weight, provided the waste acid be utilized again in a rational manner. In English and French factories the quantity of the acid mixture used is even as much as 176 times the weight of the cotton.

The nitration is done either in iron or lead vessels, or in a special nitrating apparatus, or in centrifugal nitrating machines.

The iron vessels used are square cast-iron tanks standing in a second larger tank, cold water being run through the space between them. The inner tanks have, as a rule, a grate at the back into which a ring or a cross-bar is fixed. An iron lever, bent into a hook at one end, is inserted into the ring or bar. At about one-eighth of the length of the lever a square corrugated cast-iron plate is so fixed that it just stands above the grate; this plate serves for pressing the acid out of the nitrated cotton.

As a rule, the troughs stand along a wall, and above them hoods are arranged, which are connected with a main earthenware pipe. Into this a steam jet is blown which sucks away the gases and carries them either to an absorbing tower, or, when permissible, into the open air.

At Waltham Abbey, the dipping-pots, as they are called, are filled with 220 lbs. of acid mixture, into which 1¼ lbs. of cotton are rapidly dipped in small portions by means of a three-pronged wrought-iron fork. After five or six minutes the cotton is taken out, put on to the grating, and squeezed by means of the lever. It then still contains about 14 lbs., or about eleven times its weight, of acid.

The nitrated cotton is then put into an earthenware jar with a tightly-fitting lid, and the jar itself is put into cooling-pits through which cold water continually circulates. The cotton remains in the soaking-pot, together with the eleven times its weight of acid that it contains, for about twenty-four hours, during which time the "after nitration" takes place.

Nitrating in lead vessels is comparatively more simple. A number of leaden pots strengthened by a wrought-iron frame-work stand in cooling-pits, through which cold water circulates. The cotton is, as usual, dipped into them with iron forks. The pots are either left open or provided with lightly-fitting covers, and the cotton is left in the acid mixture for from two to three hours. Here, also, care has to be taken that the fumes developed are carried away, which is best done by exhaustors.

A nitrating apparatus, which has been repeatedly used, but which is very much subject to wear, consists of a longitudinal square cast-iron trough, over which an arched cover is fixed by means of bolts. The trough stands either in a wider cast-iron trough, or in a trough made of bricks through which water circulates. At the front the cover has an opening for charging and discharging the cotton and for introducing the acid. This opening can be closed by means of a sheet of glass. On to the cover are fixed cast-iron stands which unite at the top, forming a cross-head. Through this and the cover a screw-spindle passes, to which, inside the trough, a perforated iron plate is attached. At the lowest end of the trough there is a socket for discharging the acids. The apparatus is worked in such a way that when the screw spindle is left down, the perforated plate is at the bottom of the apparatus, the acid is then introduced, the cotton put in, and the sheet-glass cover closed. After the nitration is finished, the spindle is lifted, whereby the whole of the cotton is taken out of the acid and pressed against the cover, so that all the acid which can be pressed out is extracted. Then the acid is run out, and the cotton removed by means of scissor-like tongs.

Another apparatus patented by the Rheinisch-Westphälische Sprengstoff-Actiengesellschft of Cologne is illustrated in Fig. 151. It consists of a cast-iron pan (A), in which a perforated false bottom (S) is inserted, and which stands in a cooling-tank (B). A channel is cast on the side of the pan, which on one side is connected with the discharging socket of the pan (A), and on the other with an acid storage vessel. After the nitration is finished, a vacuum is created in the storage vessel by an air-pump, the discharge tap is closed, the inlet tap opened, and the acid drawn out from the cotton by the vacuum.

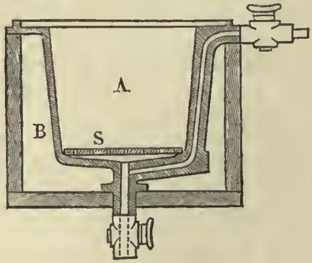

Fig. 151.

The apparatus does not seem to have been much used, as sucking off acid gases by an air-pump, the probability of the side channel being blocked, and the possibility of decomposition are not very inviting.

More recently the so-called centrifugal nitrating machines, which have evidently many advantages, have come into use. A centrifugal nitrating machine, patented by Selwig and Lange of Brunswick, is illustrated in Fig. 152, and is said to work with good results at the Troisdorf gun-cotton factory. It consists of a centrifugal machine of the usual construction, having inside the casing (A) a perforated iron basket (B) of 2 feet 11 inches diameter. The cover is provided with an opening (C) through which the cotton is introduced. It also has a discharge pipe (D) of 8 inches diameter to carry away the fumes, which are as usual drawn off by means of an exhaustor, into an earthenware pipe, and from there into an absorbing tower. The basket is suspended in the usual manner on a collar-bearing (G), and this is connected with a pulley (H) by means of a shaft passing through the frame. A band brake (I) is provided for quickly stopping the machine. When a charge is to be nitrated, the discharge-tap at the bottom of the casing is closed, and the acid poured in, the centrifugal being charged with acid to about $\frac{3}{4}$ inch from the upper edge. The basket is then slowly revolved, and meanwhile 17·2 lbs. of cotton are gradually introduced. This raises the level of acid about $\frac{3}{8}$ inch. The apparatus is then left standing with the charging-hole open, which can be done without fear, because the use of the exhaustor prevents any vapours from issuing. After about thirty minutes the nitration is finished, the acid is

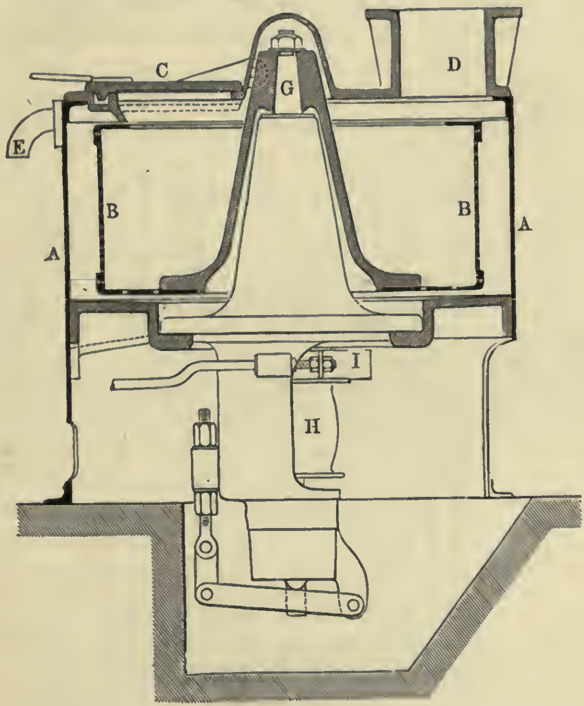

Fig. 152.

drawn off, the cover closed, and the centrifugal set revolving rapidly, which wrings the acid out. After the wringing is finished, the cotton is removed by means of tongs of suitable shape.

Another centrifugal nitrating machine, made by the Sudenburger Maschinenfabrik, is illustrated in Fig. 153. It has a vessel (a), a little wider at the top than at the bottom, fixed on a shaft, which rotates in a casing (b). The casing can be closed by a cover fitted with a counterweight. The vessel (a) fits into the casing by means of a diaphragm (c), and is thus used during nitration; if the vessel (a) rotates, then the diaphragm is lifted off the casing, but it is not necessary that it should be closed, because the centrifugal force prevents the gases from getting out there. In the vessel (a) a basket (d) is placed which receives the charge of cotton. The cover is then closed, and a weighed or measured quantity of acid run through the pipe (e) into the vessel (a) until the cotton is covered with acid.

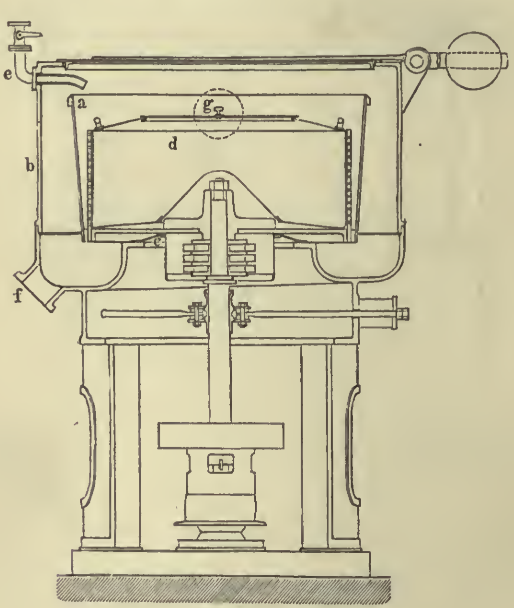

Fig. 153.

After nitrating, the centrifugal is rotated at a slow speed, whereby the greatest part of the acid is carried up the conical sides of the vessel by centrifugal force, and issues through the discharge socket (f). The velocity is then increased, and the rest of the acid thrown out. The basket containing the gun-cotton is then lifted out, and either put into water with its contents, or the gun-cotton is taken out in small quantities. The acid vapours are exhausted through the socket (g), and the acid may be run into an acid elevator and used again.

Opinions as to the suitability of the various apparatus are divided. Nitrating in small vessels has the advantage that if a decomposition does take place, the quantity of cotton and acid lost is not large, and if necessary the decomposition can be made harmless by tipping the pot or quickly removing it, or even by simply stirring with an iron rod. It will be seen later on that in the English system of using large quantities of acid for nitrating small quantities of cotton, the re-utilization of acid may be effected in a very convenient way, since the after nitration is done in a smaller vessel. On the other hand, the utilization of the remaining acid is always a matter of estimation only, and the gun-cotton differs with each nitration.

Nitrating in lead vessels has the advantage that the whole of their contents, acid and cotton, can be carried to the acid centrifugals without removing the cotton from the acid. The latter always leads to the development of acid fumes, which are injurious to the workmen, and also sometimes to decomposition of the gun-cotton through contact with the moist air.

The disadvantage of nitrating apparatus in the form of presses is that both the cover and the spindle are soon destroyed by the acid vapours, and that in general they require careful attention in order to make them work well. Apart from the convenience of pressing out, they have no advantage over ordinary iron nitrating pots, although they are much more expensive than the latter.

Centrifugal nitrating machines have, no doubt, the advantage of allowing nitrating and centrifugaling to be done in one apparatus, but it may not be possible to keep them in good order for a prolonged period unless they are very carefully attended to, and the initial outlay is heavy.

Iron vessels for nitrating have advantages over lead ones, from the fact that the sides of the vessel are less readily attacked, and that the gun-cotton made in them has a smaller percentage of ash. On the other hand, lead vessels can easily be mended, and if they are completely destroyed, they have a certain considerable value as old lead, whereas an iron vessel simply becomes valueless.

The Actien-Gesellschaft Dynamit Nobel have patented a new process of nitration. It is their intention to drive out the air from the interior of the central core of the cotton fibre, and thereby cause a more complete penetration of the acid. In order to do this, the nitration is performed in vacuo. The general arrangement is shown in Fig. 154, and the details of the apparatus in the Figs. 155, 156, and 157. The vessel (A, Fig. 157) is a lead-lined wooden vat, or an iron or earthenware receptacle placed on a framework. The nitrating vessel (B, Fig. 155) is made of cast-iron, $\frac{1}{2}$ inch thick, and into it are inserted perforated cylinders (R and R_1). It is placed on a base in such a way that the tap (b) is easily accessible. C (Fig. 156) is an ordinary cast-iron acid elevator, sunk into the ground. The taps (a, b, c, d) are of earthenware lined outside with lead, and are attached to their respective sockets on the apparatus by flanges. The cover of the nitrating vessel is fastened on with bolts and a leaden flange, and the cover of the charging-hole (O) is secured by means of a bridle and screw. The quantities of nitric and sulphuric acid required for the nitration are either mixed directly in the vessel (A) or forced in from the acid elevator by means of compressed air, the small tap (a) being closed. The nitrating apparatus is uniformly charged through the charging-hole (O) with suitably prepared cotton, which is pressed tightly, after which the discharging-hole is closed. The tap b is then opened and a vacuum produced in C and B by means of an air-pump, whereby the air is removed from the hollow core of the fibres.

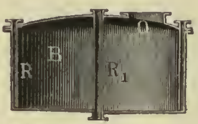

Fig. 155.

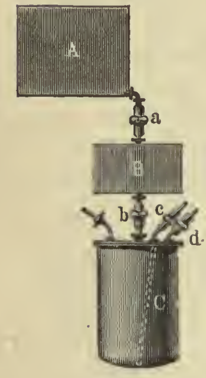

Fig. 154.

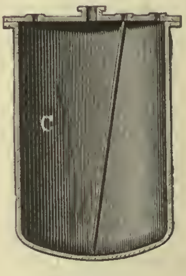

Fig. 156.

Fig. 157.

As soon as the vacuum gauge shows an almost complete vacuum, b is closed, and c is opened. By this means the acid enters into the vacuous space in B containing the cotton, and very soon fills it with acid. In order to let the acid enter at several points, the pipe for the admission of acid can, according to the size of the apparatus, be divided into two or more branches. Experience has shown that a slight, or intermittent, opening of B is sufficient to replace the spent acid by fresh, which, being cold, prevents an over-heating, and also gives the operator a strong control over the process. The last quite fresh acid is allowed to remain over the cotton after b has been closed, until nitro-cellulose of the required percentage of nitrogen is obtained, which will be the case much sooner than with other methods. The taps a and b are then opened, and the excess of acid is drawn out by means of the vacuum. The waste acid coming into C is mixed with the necessary quantity of acid for revivifying it and is then pumped either into A or into a second apparatus for working another charge. The nitro-cellulose is taken out through the charging-hole (O), the air-pump being kept working to draw off the acid vapours. Instead of taking out the nitro-cellulose, water and, if necessary, soda solution can be introduced, and the nitro-cellulose thereby washed and neutralized; but this will give rise to some trouble, since the apparatus will be strongly attacked, whilst suitable vessels which are acid proof and yet able to stand a high vacuum are not easily made. It is said that with this apparatus a hundred-weight of cotton can be nitrated at a time, and that the gun-cotton produced is of much greater uniformity than usual.

(*d*) Extraction of the Acid.

As soon as the nitration is finished, it is necessary to remove as much as possible of the acid adhering to the gun-cotton.

It has already been mentioned, when speaking of nitrating machines, that the cotton is taken out from the acid mixture by means of scissor-shaped tongs, after it has been squeezed against the cover. With the nitrating centrifugal, the extraction of the acid is done in the nitrating apparatus itself. With the ordinary cast-iron nitrating troughs a large part of the acid is squeezed out on the grate, and that part contained in the soaking-pots is fairly well absorbed by the cotton.

The contents of the soaking-pots are put into the so-called acid centrifugals, six pots at a time, so that one centrifugal machine holds gun-cotton equivalent to 7½ lbs. of raw cotton. If the nitrating has been done in lead pots, with about 4·4 lbs. of cotton, the weight of the pot, the cotton, and the acid together is not too large for two men to comfortably carry; so, instead of taking out the gun-cotton from the acid mixture and pressing it (in which case, moreover, inflammation might occur), it is preferred to lift the pots out of the cooling-pits and put them on a trolley running on rails, and to throw the contents of several pots at a time into the centrifugal.

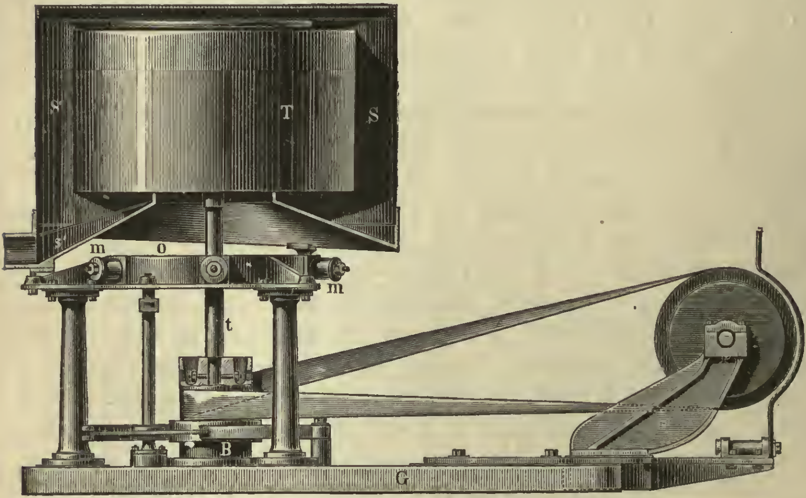

Fig. 158.

The action of centrifugal machines depends on the centrifugal force. They have a perforated or wire-wove drum, the so-called basket (T, Figs. 158 and 159), which is rotated rapidly, whereby the solid material is strongly pressed against the sides of the basket, whilst the liquid is separated from it partly through this compression of the solid material and partly through the action of centrifugal force on the liquid itself. The liquid passes through the holes in the basket and is carried away by a discharge-pipe (S¹) at the bottom of the casing (S). In acid centrifugals the basket is made of finely-perforated wrought-iron. Cast-iron would be more advantageous, but a basket made of this material would be too heavy, and would not have sufficient strength to resist the action of the centrifugal force. No other metal will effectively resist the action of acids. Wrought-iron baskets have to be renewed about once in two years. Acid centrifugals should be provided with a lid and fume-pipe for carrying away the acid vapours developed. As a rule, centrifugals are fitted with a governor for keeping the basket in equilibrium. In spite of the great circumferential velocity, it may happen that more of the solid material accumulates on one side of the centrifugal machine than on the other, and the consequence would be a one-sided movement of the basket, which, at a high velocity, would give rise to accidents.

There are various types of such governing apparatus. With the centrifugal machine here illustrated, the shaft (t) runs in a foot-step (B), fixed to the base-plate (G), and is guided by a plate (K) carrying the upper bearing. The latter is held by six tie-bars (m) passing through a ring (o) fixed on three columns and tightened by means of india-rubber discs, so that the shaft and the basket are free to oscillate a little. Another regulating apparatus, made by the Sudenburger Maschinenfabrik, which is shown in Fig. 153, works very well. It consists of three brass rings of large diameter put round the basket shaft and enclosed in a box. By the rapid revolution of the shaft these rings are thrown into such positions that they counteract any unequal loading of the basket, and thereby restore equilibrium.

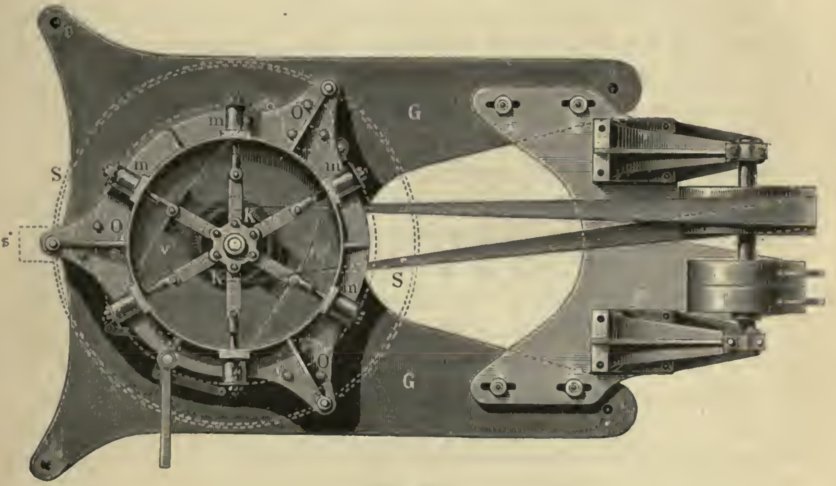

Fig. 159.

Centrifugals may be driven from the top or from the bottom, but the underdriven type is usually preferred, since the driving-belt in this arrangement is well out of the way. Each centrifugal machine has a brake, as a rule of the band form, for quickly stopping the basket when the wringing is finished, otherwise the basket would continue to revolve for many minutes longer.

The number of revolutions of a centrifugal machine depends upon the diameter and degree of extraction required. As a rule the circumferential velocity of the basket is about 140 feet per second; thus, with a basket 40 inches diameter—a usual size for acid centrifugals—a velocity of 800 revolutions per minute is required, or for a basket of 2 feet 8 inches, 1000 revolutions per minute. Such a high speed is not easily produced on the centrifugal machine; therefore a counter-gear is provided for multiplying the number of revolutions of the main shaft. On account of the acid gases developed, it is necessary to drive acid centrifugals either with impregnated cotton, or, better still, with india-rubber belts. The acid extraction in a centrifugal takes about five minutes, and about 70 per cent. of the acid absorbed by the cotton is thereby expelled. Acid centrifugals are, as a rule, located in a separate room to prevent the acid fumes spreading in case of the cotton in the centrifugal firing.

Fires in centrifugals are a special peculiarity, and their cause has not yet been perfectly elucidated. Experiments made by Dr. Dupré, in connection with H.M. Inspectors of Explosives, have shown clearly that the accidental falling of drops of water, oil, or similar foreign liquids almost immediately causes decomposition of the acid, and thereby ignition of the whole charge. On the other hand, it is known that without such accidents inflammation frequently takes place, especially on hot, moist days.

The author also has made experiments on the decomposition of waste acids, especially from the manufacture of nitro-glycerin, and, as will be seen later, has found that they decompose at a temperature exceeding 50° C., especially when violently agitated in contact with air. Of course it is not possible to absolutely exclude the possibility of water or lubricating oil getting into the acid centrifugals, but, as a rule, it is not a frequent occurrence, since the centrifugals are covered. It is much more probable that on a warm summer's day, when the temperature of the atmosphere is about 84° F., local friction may take place by the violent throwing and pressing of the gun-cotton against the sides of the basket, and this may bring the contents of the centrifugal to a higher temperature than that of the surrounding air. The development of nitrous fumes may also give rise to formation of water, which would tend to heat the acid by diluting it. If we further take into consideration that a considerable quantity of moist air is able to enter the basket through the fume-pipe, then all elements for initiating decomposition of the acid are present; and if various favourable circumstances act in concurrence, it will probably occur.

The acid extracted by the centrifugals is either stored in special storage tanks, or is carried straight to the de-nitrating apparatus, the latter of course only when it is not intended to use it again. If the acid has to be revivified, it is run into special mixing-vessels, or direct into acid pumps, whence it is drawn for further use. An average composition of the acid coming from the centrifugals is about—

	FROM THE GUN-COTTON MANUFACTURE.	FROM THE COLLODION COTTON MANUFACTURE.
	PER CENT.	PER CENT.
Nitric acid, monohydrate	10	30
Sulphuric acid, monohydrate	80	50
Water	10	20
	100	100

It is turbid from the presence of fine particles of gun-cotton, and must, like all waste acids, be carefully guarded from external influence, since it decomposes easily.

(*c*) REVIVIFICATION OF ACIDS.

Since, as has been already stated, about fifty times the quantity of acids is, as a rule, taken for making gun-cotton in order to ensure complete nitration of the cotton, it would be a very costly process to use fresh acids for each new batch of cotton. It has, therefore, always been considered important to re-utilize the acid extracted from the gun-cotton. It is clear, according to the composition of the waste acids given above, that if we consider the 80 per cent. sulphuric monohydrate to be highly concentrated sulphuric acid, say about 96 per cent. monohydrate, there would be only 83 per cent. of serviceable sulphuric acid in the waste acid; whilst the existing 10 per cent. of nitric acid monohydrate would be mixed with 7 per cent. of water, which would lower its concentration to a specific gravity of 1·370. Such nitric acid is of no use for further nitration; in the most favourable case it would, after many hours' nitration, only give a low nitrated cotton, and more probably the cotton would be partly attacked by the sulphuric acid. For these reasons it was long ago thought advisable to revivify these waste acids.

When describing the nitrating process at Waltham Abbey, it was mentioned that immediately after nitration the gun-cotton holds about 11 parts of absorbed acid; the same quantity, or a total of 14 lbs. of fresh acid, has consequently to be added to the 206 lbs. remaining in the dipping-pots. In German factories it is customary to take away one-fourth of the total acid, and to add one-fourth of fresh acid mixture. In the English method of revivifying the acids, increasing quantities of water must remain at each succeeding operation in the acid mixture, and the same, although not to the same extent, takes place in the German method. Since the same mixture of acids is added, whilst the waste acid contains three-fourths of all the water formed, it follows that the gun-cotton produced contains less nitrogen at each succeeding operation, and that it therefore contains larger quantities of soluble gun-cotton. It is necessary, therefore, to mix the gun-cotton produced from various charges, in order to obtain a uniform product; moreover it is unadvisable to keep on revivifying the waste acid for more than about twenty charges in the English method, or for three to four charges in the German one. To revivify acids in a rational manner, it must not be done by guessing, but by making the percentage of nitric monohydrate in the revivified mixture up to about the same as in the original mixture. Experiments made by the author in connection with Dr. Abelli have shown that by suitably altering the proportions of the acid mixture used for revivification, the percentage of monohydrate can be regulated in such a way that even after revivifying and nitrating some twenty times, and using fifty times the quantity of acid, the gun-cotton only loses a few tenths of a per cent. of nitrogen. Of course, since the composition of such revivifying mixtures depends mainly upon the quantity of acid originally used, the normal strength of the acids, the quality and dryness of the cotton, etc., definite rules cannot be given.

To revivify the waste acids indefinitely is impossible—firstly, because on contact with the vessels the sulphuric acid forms sulphates which deposit, and in still larger measure because fine particles of gun-cotton remain suspended in the acid and make it thick and turbid, so that it can no longer properly penetrate into the cotton. It is advisable, therefore, to do away with the waste acids after a certain number of charges, which has to be determined by experience.

The German method of nitration and revivifying, where the whole of the acid mixture used except that part absorbed by the cotton is extracted in the centrifugals, is more advantageous than the English method, since it is quite possible to exactly regulate the quantity of acids, and to give it for very large quantities an exactly-defined composition. As a rule, the acids from one shift's or from a whole day's working are run into storage vessels, and after their percentage has been examined, the composition of the revivifying mixture is arranged to suit it.

(*f*) WASHING.

After extracting the acids, the gun-cotton is taken out of the centrifugals as quickly as possible by means of scissor-shaped tongs, or forks. For this purpose a small sliding window is made in the wall of the centrifugaling room, from which a gutter lined with sheet-iron leads into a washing-tub, or a washing machine. A workman standing on the other side of the washing machine draws the cotton down the gutter as quickly as possible and plunges it under the water. Sometimes the gun-cotton is put into tins and carried to the washing machine. Washing-tubs are rarely used, but as a rule washing or immersing machines, somewhat of the shape of beaters, are employed. They consist of a longitudinal oval wooden tank of about 10 feet 6 inches in length, 5 feet in width, and 2 feet in height, having in the middle a wooden partition, so that a nearly equally wide space is left between the sides of the tank and the partition wall. In order to increase the velocity of the water, one side is, as a rule, left a little narrower than the other, so that on one side the water is swelled and rushes down more quickly from the paddle-wheel. Plummer blocks are fixed on to the sides of the tank, in which a wrought-iron shaft runs. This carries on the narrower part of the tank a wooden paddle-wheel which catches the gun-cotton, dips it into the water, and at the same time agitates the water itself, thereby constantly carrying fresh cotton to the paddle-wheel. Sometimes there is also, as is the case with paper beaters, a "swelling" below the paddle-wheel, of which more will be said later on, when dealing with the pulping of the gun-cotton. This swelling forces the gun-cotton to ascend an incline, and when it arrives at the top, to be submitted to a certain squeezing pressure by the paddle-wheel, which effects a better washing. Sometimes the washing machines have two paddle-wheels moving in opposite directions, which is, of course, a still more advantageous arrangement. At Waltham Abbey it was formerly arranged that the gun-cotton, on coming from the centrifugals, should be placed under a hood of sheet-zinc, from which it was pushed into a tube at the bottom, where it met a large current of water which carried it instantly into the immersing machine; this plan has, however, been abandoned. The gun-cotton is worked in the washing machine, through which a current of water constantly flows, until it is no longer acid to the taste. The discharge of the water is effected through a sieve on the side which is either a perforated board or wicker-work, and, as a rule, a wicker basket is also put in front to prevent pieces of gun-cotton from being carried away with the waste water. It is better, and quicker, to put the whole of the gun-cotton into a second washing machine after it has undergone a preliminary treatment in the immersing machine. Sufficient time can then be given to the washing, without disturbing the working of the centrifugals, the gun-cotton being thus purified until it does not redden litmus paper.

The washed gun-cotton is either wrung out in ordinary centrifugals, or the immersing machine is emptied and the wet gun-cotton carried to the boiling-rooms.

(*g*) BOILING.

In spite of careful washing in the immersing machines, the gun-cotton still contains traces of acid, especially in the interior of the tubular fibres, which have maintained the structure of the cotton. In order to still further eliminate these traces of acid, the gun-cotton is boiled, and in most factories this is done with a 2 per cent. soda solution. It has been found at Stowmarket that the soda solution partly decomposes gun-cotton, and that the yield in consequence becomes smaller. In England, therefore, the gun-cotton is boiled in water only.

For the purpose of boiling, large wooden vats with perforated wooden false bottoms are used. Steam is introduced between the false bottom and the bottom of the vat, this is filled with gun-cotton and pure water, and the boiling continued for a period, depending on the quantity treated, of from eight hours to four days.

The advantage of using soda solution is that the boiling is done in a shorter time. At Moulin Blanc, the gun-cotton after nitrating is washed once in pure water, then five times in water containing 28 grammes of ammonium carbonate per litre, and then again twice with pure water. In some factories it is found advantageous to steam the gun-cotton after boiling, and similar vats to those for boiling are employed; but the gun-cotton is introduced without water and steam only admitted, the condensed water being constantly discharged through the false bottom. It is said that gun-cotton so treated has a higher degree of stability.

(*h*) PULPING.

In spite of washing and boiling the gun-cotton still retains traces of acid within its fibres, which has in former times given rise to its spontaneous decomposition on storing. The credit of having pointed out that the thorough purification and stabilization of the gun-cotton is the only means of preventing such decompositions, and of having found the means for such purification, by pulping the gun-cotton in beating engines, belongs to Sir Frederick Abel. Figs. 160 and 161 show a suitable pulping machine for this purpose, which is made by J. and R. Shorrock, of Darwen. It consists of a cast-iron trough (A) about 13 feet 6 inches long, 6 feet 8 inches wide, and 2 feet high, with a cast-iron partition (B) dividing the pulper into two slightly unequal parts. In the smaller half is placed the swelling (C), which ascends towards the knife-roll (D) at an angle of 15°, then curves concentric with the roll, and finally suddenly drops at an angle of 45°. On the side of the tank two bearers (d) are fixed, in which a wrought-iron shaft (d_1) is carried on plummer-blocks, the knife-roll (D) being fixed on this shaft. It extends about the whole width of the partition, and is made entirely of cast-iron, while on its circumference a series of knives or bars are fixed parallel to its axis. In the swelling below the roll, at a short distance horizontally from the axis of the roll, is the so-called "knife-plate" (F). It consists of a number of knives similar to those on the roll, but standing closer together, and held fast by means of bolts. As the knife-roll rotates over the knife-plate, it carries the gun-cotton between the two, and simultaneously subjects it to a cutting and a crushing action.

The carrying levers, and with them the knife-roll, can be raised from or lowered to the knife-plate by means of the adjusting gear (G), which is actuated by the hand-wheel (H). In this way the degree of fineness of pulping is regulated. On some other place in the beater, as a rule at the end of the partition, a second wrought-iron shaft (i) is placed, which carries in the other half of the trough a washing-drum (I). This is driven by a cog-wheel (i_1) and a belt (i_2) from the knife-roll. It can be lifted out or lowered into the water by means of the adjusting gear (k), the arrangement of which can be clearly seen from the drawing. The washing-drum consists of an iron frame covered with fine wire gauze. Within this wire cylinder, and fixed on to the shaft, are scoops (l) of sheet-iron bent into a spiral, which during rotation constantly take up the water and carry it into a gutter outside the cylinder, whence it runs away, the object of the wire cylinder being to admit water only and no gun-cotton into the scoops.

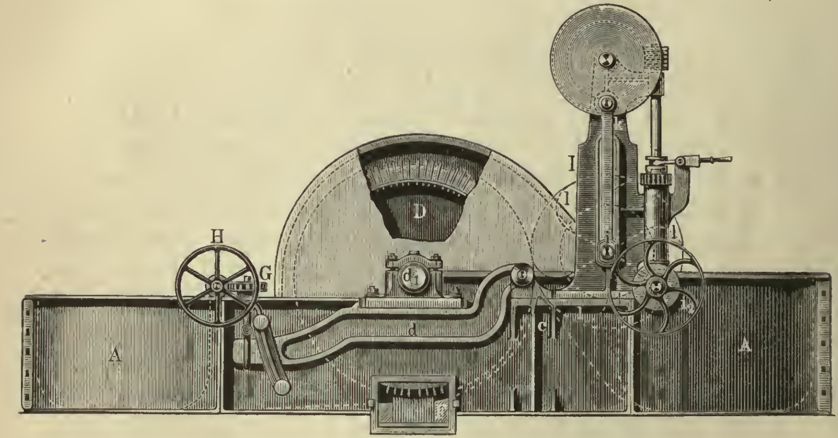

Fig. 160.—One-40th full size.

At the bottom of the beater is fixed a large disc valve (m), which can be lifted from above for discharging the beater. It is good to place a wire-gauze cylinder over the valve to prevent the gun-cotton from coming out, or better still, a movable basket can be placed below the valve. After having discharged all the water, the gun-cotton can be either scooped out or discharged through the outlet. In some places a centrifugal or rotary pump is used for transferring the pulped gun-cotton from the beater to the centrifugals, an arrangement which saves much work.

Fig. 162 shows a pulper made by Messrs. Bertrams, Ltd., of Sciennes, Edinburgh, of somewhat different construction to the pulper above described. The tank (A) has a rounded bottom, so as to allow a better circulation of the pulp. The bearings (d) of the shaft (d_1) are hung on bars, the ends of which are screwed. The drum is lifted by turning the hand-wheel (H), which is geared to the nuts on top of the suspension bars. The washing drum (I) is driven as usual by cog-wheels (i_1) and a pulley (i_2). It is not lifted automatically, but by means of a hand-wheel (R) with a pinion gearing into a rack. The drawing shows very clearly the working arrangements, and the pulper is one well adapted for its work.

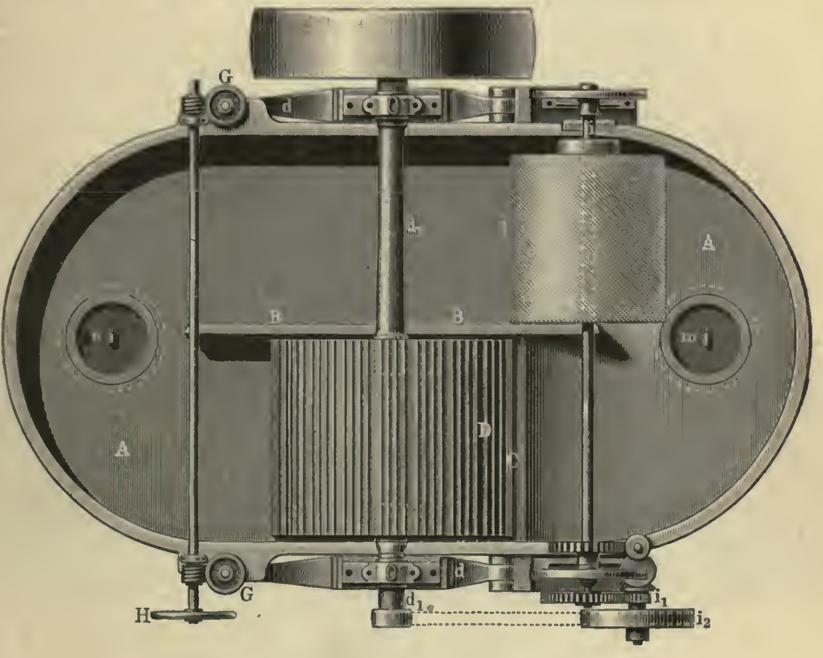

Fig. 161.—One-40th full size.

The beaters hold, as a rule, about 4 cwt. of gun-cotton with the corresponding quantity of water. At the beginning the gun-cotton is only roughly cut, the knives being placed wide apart, then the distance between them is very gradually decreased as the pulping process goes on. Of course great care must be taken to prevent foreign matter, nails and such like, from getting into the gun-cotton, as it would at once cause damage to the knives. At Waltham Abbey they prefer to pulp the gun-cotton in beaters without washing it in them or changing the water, and then to subsequently wash the finished gun-cotton for six hours in washing machines; but it is more general to constantly change the water in the beater, because any traces of acids which may be removed from the gun-cotton are then at once carried away.

It is of advantage to wash in warm water, which can be easily done by introducing a steam-pipe. If calcium carbonate has to be added to the gun-cotton, and if the latter has to be used in a loose state, it is usual to add the alkali in the pulper. When making large compressed slabs, especially when a definite percentage of calcium carbonate is specified, it is more convenient to add the alkali before pressing. The degree of pulping varies according to the requirements specified for the gun-cotton. If the slabs are to be compressed, the pulping must not be carried too far, because in that case a much higher pressure will be required for compression. For smokeless powder and similar materials the finest possible state of division is desirable, because in this state the gun-cotton dissolves more rapidly.

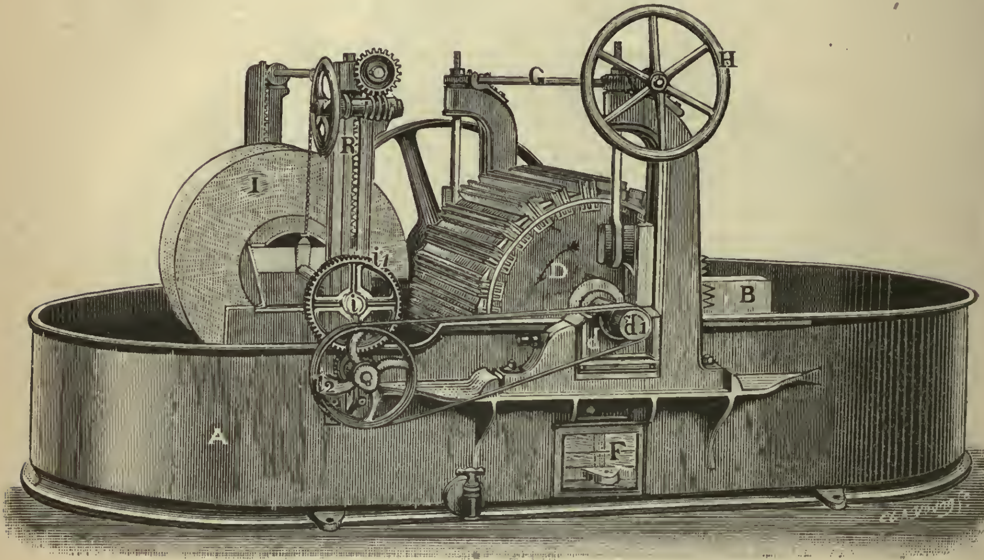

Fig. 162.

At Waltham Abbey the pulped gun-cotton is sucked into a "stuff-chest," standing at a higher level, by means of a vacuum. After pulping, gun-cotton should stand the heat test perfectly. Sometimes it may happen that this is not the case, and then the gun-cotton is boiled several hours in lead-lined wooden tubs with the introduction of steam and constant stirring; but this should not be necessary with careful work.

(*i*) WRINGING.

The gun-cotton coming from the beater is, as a rule, not worked up immediately, but is stored wet. The contents of the beaters are therefore treated in water centrifugals. These are similar in construction to acid centrifugals, except that the basket is made of perforated sheet-copper, over which a layer of wire gauze and then one of flannel is laid. At Moulin Blanc the pulp is put into pits, the bottoms of which are built of perforated bricks, over which a layer of cloth is laid. After the greater part of the water has drained off the gun-cotton is centrifugaled. In spite of all precautions, the water issuing from the beaters and water centrifugals always contains small quantities of finely-divided gun-cotton in suspension, and this has to be carefully removed. For this purpose settling-tanks are arranged. They are, as a rule, either brick pits or wooden boxes divided into several compartments by means of partitions, with communicating holes alternately at the bottom and at the top, so that the water constantly circulates through one compartment after the other. The gun-cotton collected in these settling-tanks is usually unserviceable on account of the presence of lubricating oil and other impurities, and is destroyed. The gun-cotton coming from the centrifugals contains on an average 25 to 30 per cent. of water. In this state it is stored in zinc-lined, tightly-closed boxes, and is examined from time to time for its percentage of moisture, this being generally kept at about 30 per cent.

(*k*) PACKING LOOSE GUN-COTTON.

For railway or other transport, loose gun-cotton is packed into wooden boxes lined with zinc, or tough paper. In order to pack as large a quantity as possible into one box, it is necessary either to ram the gun-cotton into the box, or to press it in by means of screw presses. The presses used are generally either simply strong screw presses or toggle-lever presses, with a block which presses the gun-cotton into the box; or else about 8 inches of gun-cotton are put into the box, and a block of wood corresponding in size to the box is put on it, and the whole is placed under the press, the process being repeated until the box is filled. The boxes are closed by means of a sheet of zinc soldered with a solder of low melting-point on to the bent edges of the lining. This operation requires care, and it is necessary to let the solder rather drop on to the lining than to go over it with a soldering-iron, because otherwise the gun-cotton immediately under the zinc might get unduly heated. As a rule, a box holds between 170 and 224 lbs. of dry gun-cotton. The gun-cotton is sold by dry weight, but it is frequently specified that it should not contain less than 30 per cent. of moisture. The dry weight is determined by taking small samples out of each box from the top, centre, and bottom. Those from four boxes are then examined together for moisture in the usual manner. The quantity of moisture thus found serves for calculating the total weight of gun-cotton in the boxes.

(*l*) SEPARATING KNOTS.

In some cases the process of pulping can be shortened if the gun-cotton pulp is first passed through an apparatus called a "knotter," the object of which is to catch any knotted lumps. The elimination of even a very small percentage of knotted cotton in the material to be pulped would necessitate a comparatively prolonged pulping, and the previous removal of it by some efficient mechanical appliance will, as a rule, effect a considerable saving of time in the pulping process.

Such knotters are largely used in paper manufacture, and various types of them are to be found in the trade. As a rule, they consist of a finely-slotted metal plate fixed on a frame, which receives a short up-and-down shaking motion. In order to further quicken the process, the bottom of the frame is connected with a box from which a centrifugal pump, or a vaccum pump, continually draws out the pulp, in order to prevent the fine openings in the metal plate from getting clogged, and also to make the pulp, which is put in together with a large quantity of water, run through more rapidly. At Moulin Blanc the gun-cotton enters a vat in which a brass-wire sieve of $\frac{1}{24}$ inch mesh is kept oscillating. If the pulping is done with great care—that is to say, when the knives of the roll are kept properly ground, and when sufficient time is devoted to pulping—a knotter is superfluous.

2. COMPRESSED GUN-COTTON.

For military purposes gun-cotton is almost exclusively used in a compressed state—that is to say, it is moulded into blocks of regular shape by means of high pressure. By this means an increased specific gravity, a more regular propagation of the explosion within the charge, a stronger action in a given space, and easier manipulation are obtained.

(*a*) Preparation of the Loose Gun-cotton.

In order to make gun-cotton suitable for the preparation of such blocks (cartridges, torpedo charges, etc.), it is in the first instance necessary that the whole quantity to be used should be of uniform quality, and this has to be determined in the laboratory. Further, it is important that the gun-cotton should be put into the moulds in which it is compressed in a state of perfect subdivision, so that the pressure exerted by the press shall be distributed uniformly throughout the whole mass. The most suitable means for attaining this is to suspend the gun-cotton in warm water.

At Waltham Abbey and in other factories this is done, as previously said, in a stuff-chest, into which the gun-cotton, to which the necessary proportion of calcium carbonate has been previously added in the pulper, is drawn by means of a vacuum. This stuff-chest is a round iron vessel holding about 18 cwt. of gun-cotton and the corresponding quantity of water. In it a shaft fitted with paddle-arms rotates, thus keeping the pulp constantly in agitation, so that the pulp in all parts of the stuff-chest contains about the same proportions of water and gun-cotton. Small measuring vessels provided with a gauge-glass are connected with this stuff-chest, and a suitable quantity of gun-cotton pulp can be drawn off into them, the quantity being read off on the index-plate of the gauge. Similar stuff-chests are used in almost all factories.

Since it has been proved that, in spite of all precautions, including the most careful stirring of the gun-cotton pulp, the quantity required for the cartridge cannot be measured with sufficient accuracy, it is more advantageous to determine the percentage of moisture of the gun-cotton kept in a storage bin and to weigh off separately, with due consideration of this moisture, the quantity required for each slab. With well-pulped material the quantity weighed off can be easily and uniformly suspended in water by mixing it in a small bucket by means of a wooden rod. Gun-cotton made in this way shows only quite insignificant variations in weight after pressing and drying.

(*b*) PRELIMINARY PRESSING OR MOULDING.

In most factories the gun-cotton is compressed in several stages. First of all the greater part of the water is extracted in suitable moulds, which gives the slab its proper form as far as diameter goes, although the height will still be considerably larger than that of the finished slab. At the same time it obtains sufficient cohesion to stand being taken out and transferred to the mould in which it is finally pressed, without crumbling to pieces.

This moulding is done either in hand-presses or by hydraulic pressure. At Stowmarket tubes of sheet zinc are used which widen out at the top into a funnel shape. They are closed at the bottom by a wooden piston, on to which a hinged bottom-piece is put; there is also a lever attached to the wall, carrying a wooden piston. The gun-cotton pulp is placed in the tubes, and by pressing down the lever the first moulding is done. At Waltham Abbey a small hydraulic press is used. A bronze mould containing a number of holes is put on to a box which is covered with fine wire gauze and connected with a vacuum pump. On to the bronze mould a cylindrical filling-box with an agitator is put, and the gun-cotton pulp coming from the before-mentioned measuring vessel is run into the mould by means of an india-rubber hose, the vacuum pipe drawing off the water at the same time as the pulp runs in. After this the bronze mould is run on wheels under the press. This is fitted with a number of pistons, which first compress the slabs and then press them out of the mould. The pressure applied is about 38 lbs. per square inch.

In more recently-built factories, hand-pressing is combined with aspiration, by putting the mould on to a special aspirating table, and at the same time, placing on top of the mould the so-called "stone," which is pressed on to the gun-cotton by means of a lever. This apparatus will be more fully described further on.

The most suitable process, in the author's opinion, is probably one which he designed in which the moulding and final pressing are done in the same mould, so that it is not necessary to transfer the slabs from one mould to a second one. This is an operation requiring great care, as it is liable to damage the slabs, which also always expand a little, so that when two moulds are used the second must always be a little wider than the first one.

(*c*) Final Pressing.

The moulded slabs are about three times the height to which they are finally compressed. It follows from the manner in which pressure is propagated through a pulverulent substance when applied on one side only, that it will not be transmitted through the whole mass with the same intensity, so that the parts at a greater distance from the point at which the pressure is applied are less acted on by it than those near it. Theoretically, the pressure decreases as the square of the distance. In reality this is not exact, because the shape and size of the slabs have considerable influence on the amount of friction against the sides of the mould, and as the friction increases a great part of the pressure is rendered inoperative. The more irregular the surface of a slab is, the greater the pressure required to obtain a certain density. For these reasons gun-cotton cannot be pressed into slabs to any considerable thickness. It is preferable to make them not thicker than 2 inches, and to build up large slabs by laying several of these one on the top of the other.

Since a pressure of between 7500 and 15,000 lbs. per square inch is required for pressing gun-cotton, it is done exclusively by hydraulic presses.

Fig. 163 shows a press built by Taylor and Challen, of Birmingham, for the Royal Gunpowder Factory of Waltham Abbey for pressing torpedo slabs. The press itself does not differ materially from other hydraulic presses,[1] only the ram (A) is guided on the sides on two columns (B) by means of claws (a). The upper part of

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[1] Full information on hydraulic presses in general will be found under Compressed Black Powder, vol. i. p. 247.

the ram has a rim for catching the water expelled from the moulds during compression. About half-way up to the cross-head (F) is placed the mould (C), which is guided on the columns by four arms

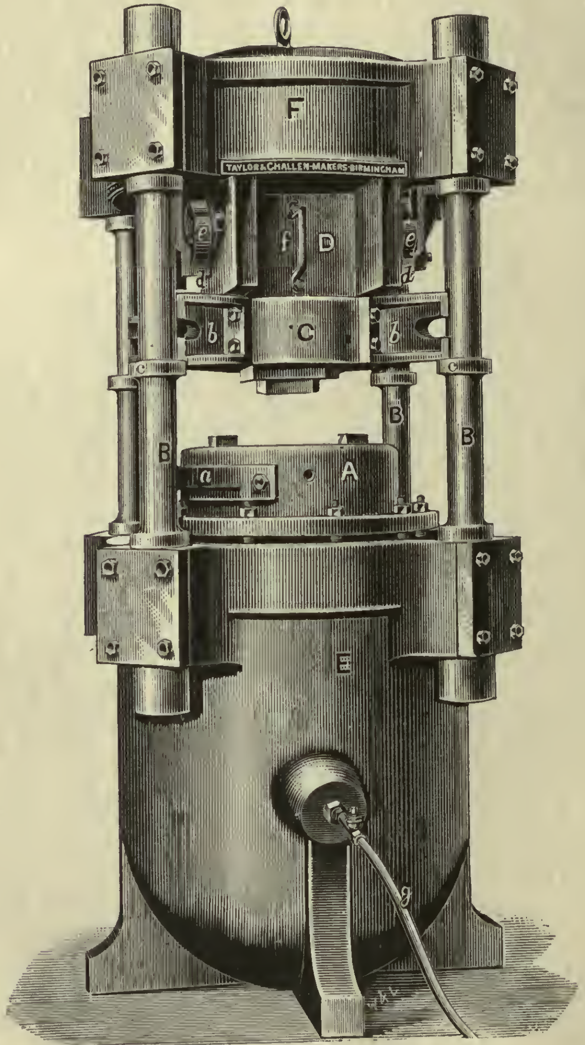

Fig. 163.

(b) screwed on to it, and rests on rings (c) forged on to the columns. Two rails (*d*) are fixed on the cross-head, and on these a small trolley (*e*), carrying the press block (D), runs. This consists of a solid and a hollow part, the latter being formed by two projections cast on to the pressing block. A handle (f) is fixed to the front of the hollow part for moving the press block, together with its trolley, to and fro. To press a slab, water is introduced into the cylinder (E) through the pipe (g), the ram with the piston held by two rails ascends, and the material in the mould is compressed between the solid part of the press block and the piston. After the pressing has been effected, the ram is lowered and the press block pushed back, so that the hollow part stands above the mould. The mould is then again raised by admitting water to the cylinder until it comes in contact with the sides of the hollow part of the press block, which stop it moving any further. The ram, however, continues to ascend, and in doing so pushes the slab out from the mould.

With this press it is necessary to have a different mould plate for each form and size of slab, and to dismount it and fix a new one whenever another form has to be pressed. In most English and German factories it is usual to press in such a manner that the mould containing the moulded slab receives two so-called "stones" on the top and the bottom. These are steel plates, 2 inches high, of the shape and size of the bore of the moulds, and are so made that they fit absolutely exactly into the mould. They are perforated with a large number of fine holes, $\frac{1}{24}$ inch diameter at most, widening towards the exterior. Thus one stone lies at the bottom of the mould, and another one on the slab. The mould so arranged is put on to the ram, and a press block revolving round one of the columns is swung over the mould, so that the ram on rising presses the mould against this press block. Sometimes the mould is put on to a revolving arm, and a bottom piston presses the slab against the smooth crosshead of the press.

Great care must be taken in making the moulds and "stones." The best material for them is cast steel, since it has the requisite strength to withstand the high pressure, and is also sufficiently hard not to be worn out too soon, and also because on account of its great tensile strength the thickness of the moulds can be smaller than if other materials were employed. How important these circumstances are is shown by the fact that even the best kinds of steel—and so far the best has proved to be a special steel made by Krupp of Essen—wear out gradually within a few months on that part where the slab is submitted to the highest pressure, and that in this place an enlargement of the mould takes place. This wear can give rise to such great friction as to cause the gun-cotton to burn or explode in the mould. It may also give rise to breakage of moulds and presses, through jamming occurring.

The best shape for moulds is cylindrical, as is self-evident, since by this form material is saved, and the stress upon the material is uniform. The core in moulds for cylindrical slabs is made by drilling and turning and afterwards carefully smoothed by filing; for parallelopipedic or irregular slabs they are made by drilling out cores and afterwards filing out to shape. Since such slabs have to be of exactly specified diameter, very exact gauges are used for making them; as a rule, one that just enters the mould, or sometimes two are used, one of which can be put in easily, the other one with difficulty only. Exact gauges have also to be used for stones and pistons.

The filing out of such moulds—for instance, those for making circular segments for torpedo charges—requires special skill and extreme patience, and takes many days. The stones, or pistons, should fit so exactly into the moulds that they will suck like the piston of a pump. There should not be the slightest friction at any point. The holes in the stones are made conical, so that the water may run off easily, and any gun-cotton particles that may have passed through be easily carried away. If pistons are used instead of stones, the body of the piston is of slightly smaller diameter than the real pressing head, which is in reality nothing but a stone screwed, or otherwise fixed, to the piston. In this case some of the perforations in the stone are made parallel to its axis, and others diverging towards the sides, so as to clear the projecting parts of the stone. Sometimes there are channels made between the stone and the body of the piston corresponding to the axial holes of the stone. As previously mentioned, a special steel of Krupp's has proved the best material for moulds and pistons. It has a coarse structure, but is of extraordinary tenacity, and can be worked to a very fine and smooth surface. Its hardness is so considerable that moulds made from it last a very long time. For preliminary moulding, moulds of ordinary or manganese bronze are sometimes used. The pistons used for preliminary pressing, or aspiration, are mostly made of bronze. The stones for the final pressing are always made of the best steel, as are also the heads of the pistons. For the lower parts of the pistons, steel of lower quality—or better, ordinary chilled cast-iron—can be used, since the pistons are only working under pressure.

As before mentioned, the pressing is, as a rule, done by admitting hydraulic pressure under a ram. With this arrangement the gun-cotton is compressed from one side only, and the consequence is that the side of the slab farthest from the piston is the least compressed. A considerable pressure is therefore required to give the gun-cotton the requisite minimum density in all parts. For a density of 1·00 to 1·10, a pressure of about 15,000 lbs. per square inch is required for slabs of not too irregular shape, and a surface of about 28 square inches. For pushing out the compressed slab from the mould, a pressure of about 3000 lbs. per square inch is required, because the finished slab adheres so firmly to the sides of the mould that it can only be detached by using considerable force.

The maximum friction takes place on pressing out, and any inequality, and also any jamming of the stones, may become dangerous, because the compressed slab has to travel a long distance. Pressing slabs by stones has the disadvantage that they can only be made of a comparatively small height, and the larger the surface of the slab, the easier it is for the stones to lean on one side. Especially will this be the case when the stone and the mould are worn, when jamming will take place, as the axis of the stone is then no longer identical with that of the mould, and the consequence will be friction at a few points, and this, on rapid pressing, and especially on pressing out, may cause the evolution of considerable heat. Such jamming will of necessity be unfavourable to the smooth surface of the mould, and is consequently the cause of rapid wear. Moreover, from the frequent putting in and taking out of the stones and moulds, it is impossible to avoid their becoming damaged, since their weight is considerable and their surfaces highly polished and wet, which makes them difficult to handle.

The pressure required for pressing is supplied exclusively by accumulators. Sometimes two accumulators are used. One of these serves for giving a kind of preliminary pressing, and thus eliminating the largest part of the water, and at the same time to rapidly lift the ram as long as the pressure to be applied is not yet considerable. As a rule, such an accumulator is arranged for a pressure of about 7500 lbs. per square inch. The second accumulator then applies the high pressure, for which only a very small additional quantity of water need be forced into the cylinder. A specially-constructed inlet valve allows the alternate introduction of low or high pressure water into the press and the discharging of the water from the cylinder.

The compression of gun-cotton slabs is generally done in isolated rooms, where the workman is protected either by a stone wall or by a stout wooden partition, or, as at Waltham Abbey, by a double curtain made of stout rope. The maximum quantity worked at a time in the press is about 2½ lbs. This corresponds to a surface to be pressed of about 30 square inches, and a total pressure to be applied by the press of about 200 tons.

In a gun-cotton factory which he built, the author introduced a special method of compressing gun-cotton. The chief feature of this is that the gun-cotton pulp when once put into the mould does not leave it until the finished slab is pushed out from it. In this way the objectionable pushing out and putting in of the moulded slabs and the crumbling away of the gun-cotton are avoided, and, moreover, considerable time is saved.

Fig. 164 shows the author's press and the aspirating apparatus used in conjunction with it. The gun-cotton is weighed separately for each mould, due allowance for the percentage of moisture it contains being made; it is then stirred up with warm water in a copper vessel and poured into the mould which stands on the aspirating apparatus. This consists of a number of round cast-iron vessels (a) connected with a pipe (b) by means of a socket and tap, leading to a water-catcher (B) in connection with a vacuum pump (A). On the vessel a bronze plate (C) is screwed, the surface of which is formed to the exact shape of the slab to be pressed. This plate is perforated with concentric holes, and is covered with fine wire gauze secured by screws. An india-rubber ring is first put on to the bronze plate, and then the steel mould (d) placed on this, the weight of the mould being quite sufficient to make the joint between it and the brass plate in a satisfactory manner. As the gun-cotton pulp is poured in, the water is continually drawn off, and the pulp settles into a fairly tough mass in the mould. Since the atmospheric pressure due to the aspiration has to traverse the whole of the rather soft mass in the mould, it would by itself only give a very irregular compression. To get over this difficulty, the author contrived the following arrangement for applying mechanical pressure from the top, whilst the aspiration is going on:—

Two strong brackets (e) fixed to the wall of the room carry two iron shafts (f). A cross-head (g) sliding on these shafts carries along a pressing lever (h), with a counter-weight at one

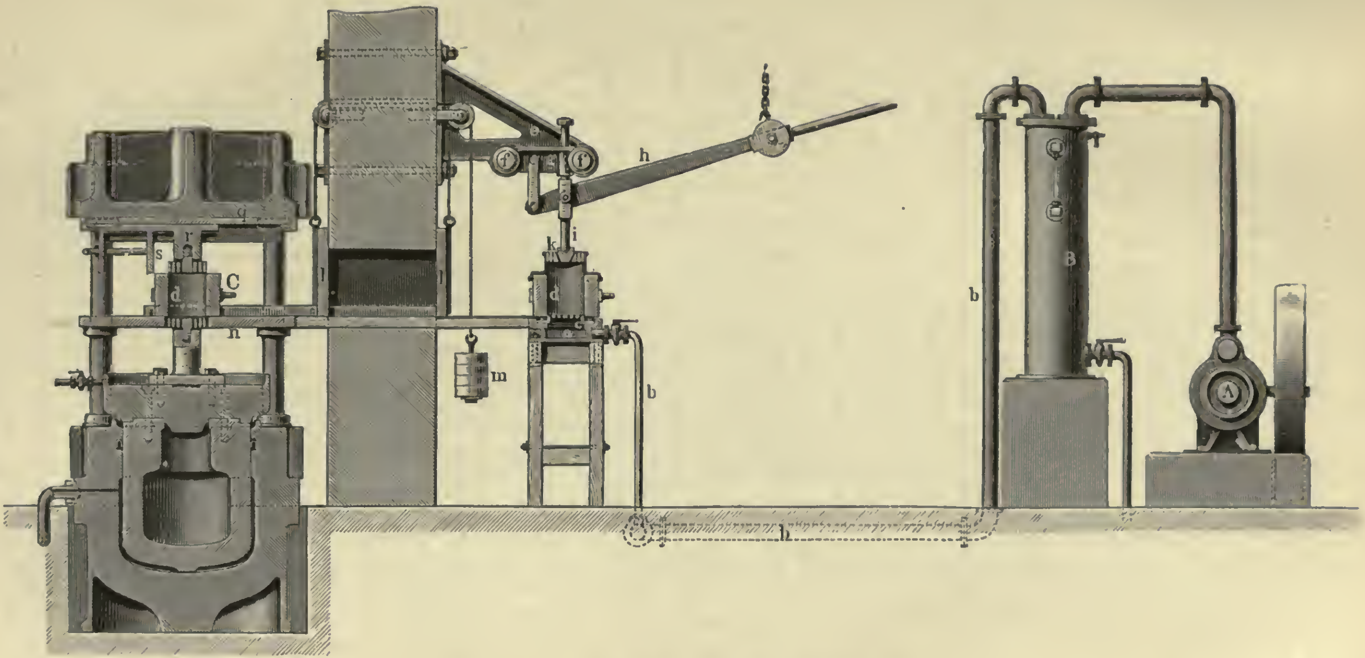

Fig. 164.—One-36th full size.

end, and near the other where it is hinged a vertical piston (i). The lever can be suspended by a hanging chain, and a hook on the weight. A bronze "stone" (k) with a boss on its top is put then into the mould. The piston of the hand-press is always on the same line as the stone, it is only necessary therefore to place it over the mould to apply a pressure, which can be regulated at will by drawing down the lever.

In this manner a large part of the water is expelled from the slabs, and they become sufficiently solid to prevent gun-cotton falling out when the mould is lifted. The mould is now lifted off the aspirating table (which can be done without difficulty, since the "stone" is very low) and pushed along a small table through a window on to the final press. This table is at the same height as the aspirating table, so that the moulds need not be knocked about, and it is lined with sheet-iron in order to make the moulds slide along it more easily. The window between the aspirating and the pressing-room is closed by means of stout wooden shutters (l), which can be rapidly lifted or lowered by aid of a counter-weight (m). In order to facilitate work, at least four aspiration tables are, as a rule, used to one press. If the slabs to be pressed have only a small surface, three and more moulds may be grouped on one aspiration table.

It has been previously mentioned that a water-catcher is inserted between the aspirating apparatus and the air-pump. The water from the aspirating apparatus enters it at the top and issues from the bottom, whilst the air is exhausted from another socket on the top. The water in the water-catcher must be drawn off from time to time, and this can be done by either opening an air-valve, or, where local circumstances permit, by leading the outlet pipe to the bottom of a pit or down the slope of a hill for about 30 feet vertical depth; with this arrangement the vessel will empty itself automatically. In both cases care has to be taken that the issuing water is freed in settling tanks from finely-suspended particles of gun-cotton, which it will always contain.

The final pressing itself is done in the hydraulic press (c), also designed by the author. In this press the use of stones, which is dangerous, is avoided, and the pressing is exclusively done by means of two pistons, an upper and a lower one. Between the four columns of the press, on which are forged collars for limiting its stroke, a pressing plate (n) is guided. It has suitable openings for large slabs in the centre, and for smaller ones in various suitable places, so that as large as possible a number of pistons can pass through them, and yet the mould can safely rest upon it. On the sides of these openings guide-rails are fixed, in which the mould slides. The mould is flattened on two sides, and has two guide-bars screwed on to it. On the back of the mould a stop-piece (o) is screwed, to enable it to be placed in its proper position. The bottom pistons (p) are screwed on to the table of the ram, and are made of chilled cast-iron with the exception of the upper or moulding face proper. To the cross-head a cast-iron plate (q) is attached, which is fitted with two angular guide-rails, in which the upper pistons (r) and the pressing-out tools (s) slide. The latter, as a rule, consist of two iron plates connected with each other by means of a cross plate. The travel of the pressing-out tools and of the pistons are limited by means of stop-pieces. The arrangement is such that if the pressing-out block is against the right-hand stop, the piston is exactly concentric with the mould; but if the piston is over to the left as far as it will go, then the pressing-out plate is in its correct position above the mould. The upper pistons and the pressing-out plate are so arranged that they only clear the mould by $\frac{1}{50}$ inch. This is sufficient to allow the piston and pressing-out plates to move freely, whilst at the same time it closes sufficiently well to prevent particles of gun-cotton being forced out, and yet lets air pass out. When the ram is forced up, it presses the mass of gun-cotton in the mould against the upper piston until the friction on the sides of the mould is sufficient to lift the mould itself, and with it the pressing table. From this moment the mould has a continuous tendency to fall down by its own weight, and is again constantly lifted by the increasing pressure, and therefore it is in a permanent state of oscillation. The consequence is that the upper and lower piston alternately exert the pressure, although the latter is movable, and the slab is thus pressed from both sides at the same time. The great advantage of this is that in order to give the slab a certain density, a much lower pressure (only about half of what is usually required) has to be applied, and the zone of least pressure is in the middle of the slab, as can be seen on the finished slab with the naked eye. As soon as the pressure is finished, the ram is allowed to fall down until the mould and the table have returned to their original position. The upper piston is then pushed aside, and the pressing-out plate brought over the mould. The ram is again lifted and pushes the slab out from the mould. The water pressed out from the gun-cotton is carried away continuously by means of a tap fixed on the side of the ram table.

If several slabs have to be pressed at the same time, the moulds are fixed in special sliding rails with sufficient play for each to be perfectly independent of the other, and an unequal filling does not influence the pressure exercised on the gun-cotton. Each receives a fraction of the total pressure corresponding to the number of the moulds and their surface. Moreover, since both the moulds and the pistons are always in the same relative position, no jamming, nor excessive friction, need be feared, even when the mould and pistons are worn. The manipulation of the press, therefore, is without danger—in fact, no explosions have as yet happened with this type of press. Nevertheless, in order to give the workman suitable protection in case of an explosion through breakage of the press or from the presence of foreign matter in the gun-cotton, the press is isolated from the inlet valve by means of a wall made of 2-inch planks in two layers, with a filling of coke-dust between them. A tube of about 1 inch diameter let into the wall allows the workman to comfortably watch the whole process of pressing. To ensure the moulds running exactly in between the rails of the press, the latter are prolonged through the window and bent out to a circular segment at their outer end. With this press it is possible to press cylindrical slabs with a surface of about 30 square inches to a gravimetric density of between 1·2 and 1·3 with a pressure of 3·5 tons per square inch.

If the pressing is done by aid of two accumulators loaded for different pressures, it is best to let the low pressure act until water ceases to be pressed out from the slabs, which occurs in about half a minute. In this way the elasticity of the gun-cotton fibres is also overcome. The high pressure is then allowed to act for another half-minute. At Waltham Abbey only one pressure is used, and it is kept on for 1½ minutes. The time is checked by an exactly adjusted sand-glass. As a matter of course, it is necessary that the packing of the ram and the pipes should be perfect. This should be taken into consideration when the presses are designed, by selecting such size for the ram that the strain on the cylinder is not more than about 1500 lbs. per square inch.

Sometimes slightly conical slabs are made direct by pressing. In this case the full pressure of the accumulator must not be allowed to act on the piston, but the pressing must be done by measure—that is to say, the piston is only allowed to penetrate to a certain depth, otherwise the stone would jam in the mould. The distance of pressing is gauged by a pointer and index scale. This process, however, is always dangerous, and is but rarely employed. When the finished gun-cotton slab issues from the mould, it alters in size slightly. The expansion in diameter is quite insignificant, but in height it may reach, according to the pressure it has been subjected to, as much as $\frac{1}{24}$ inch. In consequence of the elasticity of gun-cotton, slabs of irregular form expand irregularly. Ring segments are slightly higher in the centre than at their circumference. Moreover, gun-cotton slabs show on their surface small dot-like projections which correspond to the holes in the stone, and also a fine projecting rim which is due to the fact that the pistons never fit exactly into the mould, so that some gun-cotton passes up between the mould and the sides of the piston. The more the mould and pistons are worn, the greater will this be. If it is a question of making large charges out of single slabs, these dots and rims must be removed, otherwise too much air space would exist between the various parts of the charge, and its building up could not be done uniformly.

The slabs, as they leave the press, have on an average 15 per cent. of moisture. If a larger amount be necessary, they are soaked for some time in water.

(*d*) Shaping the Slabs.

If the gun-cotton slabs are to be used singly—for instance, as cartridges for military engineers or miners—no shaping of them is as a rule required, except when their weight is too great, in which case a part is removed by planing. If larger charges have to be built up—for instance, charges for submarine mines—then the ends of the slabs must be as smooth as possible, so that the charge can be regularly arranged and the whole fixed into its case. When shaping gun-cotton, the rule is, that it must at the outset contain at least 30 per cent. of moisture, and that the surface to be worked should be constantly rinsed with water from a vessel with a tap; otherwise the heat set up by the mechanical action of the tool on shaping would cause it to fire and explode. The compressed slabs can be easily cut in the direction of their pressing surface, which materially facilitates the planing or scraping of the slabs.

In order to avoid deformation or breakage of the slabs, it is necessary to fix them tightly in suitable clamps.

For this purpose a wooden hollow block of a suitable form or two shaped pieces of wood can be used, the latter being firmly held together by means of cotters. The planing can be done, according to the height to be taken off, either by a chisel or by planing with an ordinary carpenter's plane, or, better still, with a scraper as used by the vat-makers. The amount to be planed off is usually given by the fixing arrangement itself, or better, by putting into it two glass rods which allow the knife to draw easily and at the same time do not injure it. The surface is left fairly smooth after planing, but is improved by filing with a rasp.

If the circumference of a slab has to be diminished, it may be fixed between two boards of the form and size desired for the final slab, and the superfluous material filed off gradually by means of a rasp, or better by turning it down in a lathe.

If gun-cotton slabs have to be put in a case of a certain size, and if their shape is irregular, or if from other reasons the slabs cannot be pressed in the first instance into a shape corresponding to that of the case, then the slab is best shaped by sawing or turning. The sawing is best done by means of a band-saw. Circular saws have also been tried for this purpose, but as a rule they do not permit the saw to penetrate deeply, or inconveniently large saw-blades have to be taken in order to shape larger surfaces. With a circular saw only straight cuts can be made, and therefore a band-saw is preferable, which will produce both round and angular cuts. Of course a continuous stream of water on to the surface of the slab is also required with a band-saw, both in order to keep the saw cool and to prevent the cotton from being excessively heated. For making round slabs it is best to put them in a suitable clamp which allows only so much of the slab free as has to be cut off. This is facilitated if the slab has a central perforation. Frequently conical slabs—for instance, the charges of fish torpedoes—are made by sawing. In this case the slabs are put into the clamping arrangement and the whole laid on an inclined base, which is gradually rotated, so that an oblique cut is made. The radius will be different with each slab, and is determined by means of a gauge, or by drawing circles of different diameter on the slab. The charges when finally built up are then equalized by means of a rasp into a uniformly ascending cone. It is, however, better to make conical charges by turning them in a lathe. The arrangement used for this purpose is similar to that used in wood-working—that is to say, a lathe in which either the tool or the body is guided by a template. In this case, either all the slabs, or several of them, are fixed into the lathe and worked at the same time with a suitably-shaped steel tool.

With the gun-cotton powder made by Wolff and Co., of which more will be said later on, plates of about ⅜ inch thickness are made, and are cut by a band-saw to long strips.

Drilling gun-cotton is an operation which frequently has to be done. In general it is not advantageous to form holes of small diameter in gun-cotton slabs in the act of pressing, because excessive friction is set up in doing so, the pins for making such holes are easily bent, and jamming causes explosions very easily. It is, therefore, preferable to make holes below 2½ inches diameter by simply drilling. Small holes up to ¾ inch diameter are drilled with twist drills. The slab is fixed in a suitable clamp, and whilst the hole is being drilled, water is kept constantly dripping on to it, the drill being frequently taken out in order to cool; otherwise the heat generated by the drill will undoubtedly ignite the gun-cotton. Instead of a vertical drill, a horizontal one can be used in which the drill is taken out from the hole by a suitable shifting-gear. Larger holes up to 2½ inches diameter are drilled with a centre-bit.

For some purposes—for cartridges for military engineering, for instance—it is desirable to have gun-cotton slabs which are always ready for firing and yet are sufficiently strong to resist external damage. It will be seen later that wet gun-cotton by itself cannot explode by ordinary means, but that it is necessary to put on the top of it a slab of dry gun-cotton. On the other hand, dry gun-cotton is sensitive to friction and shock, and also fairly brittle, and therefore dry cotton slabs cannot be issued to troops. These difficulties are overcome if the dried slabs are paraffined. Paraffin wax melts, according to its source and method of production, at between 96° and 150° F. When heated just to its melting-point, it forms a viscous mass, which becomes the less viscous as the heat is increased. If a gun-cotton slab were dipped into paraffin wax heated just to melting-point, then the paraffin wax would not penetrate into the interior of the slab, and moreover it would adhere imperfectly to the outside, on account of the roughness and small protruding fibres of the gun-cotton. The layer of paraffin wax would also be so thick, that the effect of the slab on explosion would be diminished. For these reasons the paraffining is as a rule done at a temperature of about 176° F., and the depth to which it penetrates is regulated by the time the slab is left in the molten wax. The wax is usually melted in iron troughs with steam or hot-water jackets. The slabs before they are dipped must be carefully dried. The drying must be done gradually because compressed gun-cotton has a tendency to fissure, and fine cracks forming inside the compressed body would be filled with more paraffin than the rest of the slab. This would cause more paraffin wax to be taken up than the desired limit. The dipping is done in simple wooden frames, with two handles at the top and covered at the bottom with wire gauze of about 1 inch mesh. The slabs are arranged on the frames so as not to touch each other, and the frames lowered into the liquid. If necessary a second, slightly smaller, frame is put on the top of the slabs to prevent them from tipping over. The time of dipping required must be found by experiment. It depends upon the melting-point of the paraffin wax and on the depth to which it is required to penetrate. If the paraffin at disposal be too soft, it may be hardened by the addition of Carnauba wax, the melting-point of which is between 181° and 206° F. If too hard, it may be softened by adding soft paraffin or purified tallow. When the slabs have been in the wax for the proper time the frames are lifted, and the slabs allowed to cool for a short time in the air. They are then lifted off the wire gauze by pushing them away a little, so that the gun-cotton shall not be bare where it has rested on the wires. The slabs are then allowed to cool completely, and again dipped, this time for only a few seconds. If this were not done, the slab would be coated outside with a more or less irregular layer of paraffin wax, and some fine gun-cotton fibres would project beyond the coating, causing cracks in the latter.

By the second dipping a uniform coat is formed which fills up all interstices and gives the slab a smooth appearance. If the slab have an axial hole, which as a rule it has, for inserting detonators, the hole is covered by a gummed wafer before dipping. If paraffin wax be frequently re-melted, by and by it becomes brown, and the gun-cotton obtains a brownish colour. It is not advisable to re-melt paraffin for use too often, since fine particles of gun-cotton get detached from the slabs, and they will not stand prolonged exposure to a temperature of 176° F. without decomposing.

In exceptional cases the gun-cotton is completely soaked in paraffin wax. It is also necessary to dry these slabs slowly to ensure uniform drying. On the other hand, it is sometimes necessary to permanently combine a determined percentage of water with the compressed gun-cotton. For the mere storage of compressed slabs a certain percentage of water is generally required, which is checked from time to time, and in case the moisture of the slabs falls below a certain minimum, they are again wetted. For submarine mines, shell charges, etc., which for reasons of safety must not have less than a certain percentage of water, and in cases where it is not always possible to keep the vessel absolutely tight, permanent maintenance of the moisture is very desirable. For this purpose J. Alberts proposed soaking the gun-cotton slabs with glycerin instead of water. Glycerin has the tendency of absorbing water from the air, and its influence on the keeping and explosive qualities of the gun-cotton is similar to that of the water. This process, however, has not been adopted in practice.

Wilhelm F. Wolff and Max Von Förster proposed "etherizing," and have made use of it for the manufacture of their gun-cotton powder. They dip the cut cubes or strips of compressed gun-cotton into acetic ether for a brief period, which partially dissolves the surface of the body, forming a kind of collodion on the surface, which is very hard and perfectly water-proof when dry. Gun-cotton cubes made in this way have of course greater solidity, and can be simply put into the shell of the submarine mine, and the interstices between them filled by running in paraffin wax.

Wet gun-cotton when stored in air-tight vessels, especially wooden ones, is frequently liable to get covered with a formation of fungi, of a peculiar grey or black colour and a radially diverging form similar to the well-known electric figures. They are probably chiefly caused by the use of water containing organic matter, which promotes the formation of algæ, or fungi. This formation is further promoted by the fine capillary fissures in the gun-cotton itself, which, of course, chiefly exist on the surface, since the circumferential surface of the gun-cotton is always more tough and smooth. Such growths are always found on the surfaces only. In Great Britain the gun-cotton is dipped into a solution of soda and carbolic acid to prevent the formation occurring. The solution is made by dissolving 1 oz. of soda in 1 gallon of water, adding to it 2 ozs. of carbolic acid, and shaking the whole for half-an-hour. The mixture consists, therefore, of 1 part of soda, 2 parts of carbolic acid, and 160 parts of water.

3. MANUFACTURE OF COLLODION COTTON.

Collodion cotton is the soluble variety of nitro-cotton. In it less than six nitro-groups have combined with the cotton. According to Eder, four varieties of soluble gun-cotton are known, according to whether five or less atoms of hydrogen have been replaced by the nitro-group. They are—

$C_{12}H_{15}O_5(O.NO_2)_5$ Penta-nitro-cellulose.
$C_{12}H_{16}O_6(O.NO_2)_4$ Tetra-nitro-cellulose.
$C_{12}H_{17}O_7(O.NO_2)_3$ Tri-nitro-cellulose.
$C_{12}H_{18}O_8(O.NO_2)_2$ Di-nitro-cellulose.

Hitherto it has not been possible to produce the mono-nitro-cellulose, and it is probable that it does not exist in the free state.

Collodion cotton is, as a rule, a mixture of the above four soluble varieties. By carefully working with certain well-determined acids, and at a fixed temperature, it is possible to make collodion cotton of a definite degree of nitration. Since the chief uses of collodion cotton are for photographic purposes, and for gelatine-dynamite and smokeless powder, the process of manufacture is varied according to the purpose for which it is to be used. For photographic purposes the cotton should be completely soluble in ether, and the solution must be quite clear and water-white. Moreover, the layer of collodion produced on the glass plate must have a certain elasticity and yet a high tenacity. In order to obtain this, it is not desirable to make a highly-nitrated soluble cotton, *i.e.* penta-nitro-cellulose. For gelatine-dynamite and smokeless powders, on the other hand, it is not so much a question of being able to obtain a clear solution, as that the collodion cotton shall be not only completely soluble in nitro-glycerin, sulphuric ether, acetone, and similar solvents, but it must at the same time possess considerable tenacity and especially a power of binding, so as to prevent the nitro-glycerin separating out from it and give the powder made from it great hardness. For this purpose it is again necessary to use as highly nitrated a soluble cotton as possible, and the tetra- and penta-nitro-cellulose, or a mixture of the two, have proved to be the most suitable.

For the manufacture of collodion cotton very many methods have been suggested. According to Mann, 12 parts of nitric acid of 1·512 specific gravity, and 13 parts of sulphuric acid of 1·632 specific gravity, are taken, the mixture is cooled down to 5° C.

(41° F.), and the cotton allowed to stand in it at a temperature not exceeding 8° C. (46° F.) for twenty-four hours.

Another method of Mann's is to take equal parts of nitric acid of 1·42 specific gravity and sulphuric acid of 1·83 specific gravity, and to nitrate the cotton in it for ten minutes at a temperature of 140° F. It has been frequently proposed to use a mixture of nitrate of soda and sulphuric acid, or potassium nitrate and sulphuric acid, instead of nitric acid; thus, for instance, Mann has used a mixture of 20 parts of potassium nitrate and 31 parts of highly-concentrated sulphuric acid, or 34 parts of nitrate of soda and 66 parts of sulphuric acid, and treated the cotton in it for several days at a temperature of 86° F. Vogel took equal parts of saltpetre and sulphuric acid and nitrated the cotton in 30 times its weight of acid mixture during five minutes. Luchs took 20 parts of sulphuric acid and 9 parts of crude potassium nitrate. After about ten minutes he dipped into the mixture 1 part of flocculent cotton and thoroughly stirred it. The nitration was considered to be finished as soon as a sample taken out and washed dissolved completely in a mixture of ether and alcohol. After this the collodion cotton was washed in water, dried by pressing between cloth, and then dipped for twenty-four hours in alcohol, whereby its yellow coloration was destroyed. The liquid was then again extracted by pressing, and the collodion cotton at once dissolved in a mixture of 2 parts of alcohol of 90 per cent. to 15 to 20 parts of ether of 0·73 specific gravity.

It is more generally customary to make collodion cotton by using a mixture of equal parts of nitric acid of 75 per cent. monohydrate (1·440 specific gravity) and sulphuric acid of 96 per cent. monohydrate (1·840 specific gravity), which is kept at a temperature of about 104° F. during the nitration. In other respects collodion cotton is nitrated in the same manner as gun-cotton—namely, either in leaden pots or in iron nitrating vessels or in nitrating centrifugals. The time of nitration is between an hour and an hour and a half.

The extraction of the acid, washing, and pulping of the collodion cotton is done in the same way as with gun-cotton. The revivification of the acid is also done by mixing part of the waste acid with a fresh acid mixture, which may be composed of as much highly-concentrated nitric acid and sulphuric acid as will bring the revivified mixture as nearly as possible to the same composition as the original mixture.

In some factories the manufacture of collodion cotton for making blasting gelatine is done in a way similar to the nitration of gun-cotton, with the difference that the proportions used for revivification are materially altered. Sometimes the process is done in a very imperfect way; for instance, both sulphuric and nitric acids of the highest concentration are often taken and then diluted with water to the required strength, instead of, as would be cheaper and involving less trouble, using at once less highly-concentrated nitric acid. The acid mixture for the revivification also differs for each operation. Its usual composition is 4 parts of fresh nitric acid and 3 parts of fresh sulphuric acid, but this depends so much on the composition of the waste acid that no fixed rule can be given.

More recently it has been found that in order to make soluble gun-cotton of high nitration, the use of weak acids is not convenient, since with them lower nitrated cotton must always be obtained, and from the duration of nitration that is essential, the probability of obtaining insoluble gun-cotton is very great. The nitration must be considered as a function of three factors, each of which influences the final result:—First, the strength of the acid mixture; second, the temperature of the mixture; third, the time of nitration. Of these, an increase of the strength of the acid mixture and the time of nitration have a tendency to increase the percentage of highly-nitrated gun-cotton, whilst an increase of temperature gives rise to lower nitrated ones. It is, therefore, quite possible to obtain collodion cotton of any definite composition by regulating the three factors. The product obtained when they are carefully attended to is of great uniformity, perfectly soluble, and of great binding power.

It may be just mentioned in passing that a very large quantity of soluble nitro-cellulose, chiefly made from tissue paper, is used in the manufacture of celluloid. Either continuous rolls of this are passed through an acid bath, or it is torn into small pieces by means of teasing machines, and then nitrated in the usual manner.

4. NITRO-HYDRO-CELLULOSE.

Aimé Girard found that cellulose, more especially cotton, when treated with acids forms a new body by taking up water. He called this body hydro-cellulose. It has the formula $C_{12}H_{22}O_{11}$, that of ordinary cotton being $C_{12}H_{20}O_{10}$.

Hydro-cellulose can be made on a large scale by soaking the cotton for twelve hours in sulphuric acid of 1·450 specific gravity at 59° F., or for twenty-four hours in hydrochloric acid of 1·171 specific gravity (21° B.), or for a less period if gaseous hydrochloric acid be used. When heat is applied, the formation of hydro-cellulose takes place in a few minutes, but it is safest to use diluted acids, the prolonged action of which at a high temperature converts the cotton rapidly enough. In this case a mineral acid, dilute sulphuric or hydrochloric acid as a rule, can be used. The usual process is to take 3 parts of sulphuric, or hydrochloric, acid to 97 parts of water, and dip the cotton into it for a few minutes until it completely absorbs the acid, then wring it in a centrifugal machine, dry by spreading it out in the open, and finally heat it to 104° F. for ten hours in an earthenware or enamelled iron vessel placed in a stove. The higher the temperature, the shorter the time of heating required; at 158° F., only three hours are necessary. After this the hydro-cellulose is completely freed from acid by washing.

Hydro-cellulose through the action of the acid forms an easily friable mass, which from the slightest cause falls into a fine powder. It can be nitrated like ordinary cotton, and yields very pure, uniform gun-cotton. Nitro-hydro-cellulose is slightly more sensitive to shocks than ordinary gun-cotton. It is chiefly employed for the manufacture of detonating fuses (*cordeaux détonants*) used in the French army. Trauzl has made primers for blasting gelatine composed of 60 parts of nitro-glycerin and 40 parts of nitro-hydro-cellulose; they have not, however, been in use long.

5. DRYING GUN-COTTON.

For various purposes it is frequently necessary to dry gun-cotton perfectly, such as, for instance, moulded slabs, which have only been sufficiently compressed to enable them to keep their shape without their loose texture being destroyed, and which are used in the manufacture of certain kinds of smokeless powder, finely-pulped gun-cotton for the same purpose, and finely-pulped collodion cotton for the manufacture of smokeless powder and blasting gelatine. In all these cases the gun-cotton contains, either from the pressing or on account of the regulations for transport and storage, a certain quantity of water—as a rule about 30 per cent., but sometimes even 50 per cent.

Like most nitro-compounds, gun-cotton will not stand continuous heating above 158° F. without gradual decomposition, but even at a temperature of 122° F. badly manufactured gun-cotton is liable to decomposition, and even the best gun-cotton is extremely sensitive to mechanical influence (blow, friction, etc.) at this temperature. For these reasons, in actual practice gun-cotton is not dried at a higher temperature than 104° F. (40° C.).

Experiments made by the author have shown that good gun-cotton, which will stand the heat test at the usual temperature for at least twenty minutes, can be exposed to a temperature of between 104° and 122° F. for almost unlimited time without showing the slightest signs of decomposition. In order to dry it completely—that is to say, to a maximum of 0·1 per cent. of moisture, at a temperature of 104° F.—either a very long time is required, or the air must be previously dried, if rapid work be desired. It has been already stated, when dealing with compressed gun-cotton, that a too rapid drying is disadvantageous to the structure of the compressed slabs; on the other hand, the exposing of gun-cotton to a radiant heat of only 104° F. is not practical for drying on a large scale. The natural consequence of this is that a constant circulation of air previously heated is the quickest and most effective method of drying that can be obtained, and most drying arrangements are on this plan. Formerly the slabs were laid on iron plates and dried by blowing warm air over them. At present the presence of metals likely to cause friction or blow in direct contact with the gun-cotton is avoided. The blowing in of heated air has also its disadvantages. Gun-cotton has strong electrical properties, and if a current of hot air passes over it, sufficient friction takes place to develop appreciable quantities of electricity. Moreover, warm gun-cotton is, as already mentioned, highly sensitive to mechanical action, and therefore the unavoidable presence of gun-cotton dust in the drying-house is a source of danger.

The late Col. Cundill once told the author that the hard friction produced by scraping with a rough boot on a wooden floor has been known to set fire to gun-cotton dust. Mr. W. F. Reid, on the other hand, has noticed electric discharges in gun-cotton heated by a current of hot air. There is no doubt that a large number of gun-cotton houses have taken fire without any apparent reason being discoverable, it having been proved that the temperature of the rooms had not exceeded 104° F. As nobody had been in the rooms, and as gun-cotton, as was said before, will stand a temperature of 104° F. for a practically unlimited time, everything tends to prove that in such cases electric discharges must have caused the fire.

There have been many varieties of drying-houses designed, one of the simplest forms being that in which the gun-cotton is placed in a room on trays with a wire gauze bottom, over which a cloth is laid. The heating arrangement, which almost always consists of steam or hot-water stoves, is in a chamber outside below the floor level. The cold air enters from outside, passes over the heating apparatus, enters the drying-room somewhere near its ceiling, and passes out through a high ventilating shaft reaching down to the floor of the room. In this way a continuous slow current of air is produced. Both the air inlet and outlet are covered with wire gauze to prevent substances getting into the room.

Another arrangement, made by Alarik Liedbeck, consists of a hollow wrought-iron column supported at the floor and the ceiling of the drying-room. Round it a number of carriers revolve, on which the drying-frames are placed. The heated air is blown in through the wrought-iron columns and issues into the room through the openings opposite the frames.

The form of drying-house most generally used at the present time is that in which the gun-cotton is laid on wire-gauze-covered trays in a room through which warm air is circulated. The arrangement is somewhat similar to the drying arrangement described in Vol. I. p. 227, for black powder. A steam or water heating apparatus is placed in an annex, and air blown over it by means of a fan. From the heating apparatus the air is carried to the frames by means of zinc tubes, whilst a flue at the top of the room exhausts the hot air.

At Waltham Abbey the heaters are constructed on the lines of a surface condenser—*i.e.* a number of tubes are placed in a sheet-iron jacket, into which steam is admitted, while the air enters an end chamber connecting all the tubes, and passes through them to a similar chamber at the other end. The outer jacket is covered with non-conducting material and provided with a steam-trap.

At Waltham Abbey great stress is laid on the prevention of dust, and therefore, for the manufacture of smokeless powder, only moulded slabs are used, which can be easily rubbed to powder when dried. The whole of the drying building is lined with sheet-zinc, and the floor covered with sheet-lead.

The various arrangements designed by the author for drying gun-cotton have for their chief object rapid drying and a quick removal of the electricity produced in drying. If a number of frames are in a drying-room, and the air enters the building at a few points only and also leaves it at a few, or one only, then the current of air has the tendency of passing to the exit flue along the most direct path, and thus gives unequal heating in various parts of the room. Moreover, the higher frames in the room are subject to the action of the moisture coming off from the lower ones, and the consequence is that the drying is unequal and of longer duration.

In the author's arrangement the cotton is dried in closed cupboards, in which the frames are so arranged that the warm air enters below the trays on one side, is forced to go through the gun-cotton on them, and passes out above. In this way gun-cotton with about 30 per cent. of moisture can be perfectly dried in two days.

In some cases the gun-cotton, especially when containing much moisture, receives a preliminary drying by exposing it in the open air, and then finally drying it in the drying-house. The final drying has also been done by means of unslaked lime, which was spread out on flat trays under those holding the cotton. According to Plach, 1 lb. of caustic lime is required for every $1\frac{1}{2}$ cubic feet of space in the room, and in addition $\frac{1}{2}$ lb. for each pound of gun-cotton.

The removal of the electric charge formed is one of the most important things in gun-cotton drying-houses. To effect this, Mr. Walter F. Reid made the whole framework of brass tubes, and provided brass contacts to earth at the bottoms of the trays, which were covered with wire gauze, so that the electric charge was continually carried off. The author prefers to have as little metal as possible in a drying-room, and therefore instead of using wire gauze and cloth as the bottom of frames, he takes finely-perforated copper plates, the holes of which are made by a special process in such a way that their diameter near the gun-cotton is barely $\frac{1}{100}$ of an inch, and on the lower surface about $\frac{1}{24}$ inch. Even fine gun-cotton dust will not fall through the copper plate, whilst the air enters through the conical holes with a certain pressure. Leather strips are screwed on to the copper plate to prevent friction of metal against metal, and a piece of wire ending in a hook is soldered on to the copper plates. A continuous strip of copper is fixed on the drying cupboard, having soldered on to it opposite to the frames a number of short lengths of chain ending in hooks, which are slipped into those on the tray and thus connect them to earth. Thus no frame can be taken away without the earth contact being first interrupted.

The arrangement at Waltham Abbey, where the drying-house is completely lined with sheet-zinc and the floor with sheet-lead, also ensures complete discharge of electricity from the building if the metallic lining be connected with a suitably large earth-plate; but it is not sufficient to carry away the electricity collected on the trays which stand on wooden frames—on insulating stools, as it were.

The floors of the drying-houses should be made as tight as possible. To prevent undue friction it is well to cover them with sheet-rubber, but rubber has the disagreeable property of becoming hard and brittle on prolonged heating, and, moreover, great friction may occur from the well-known adhesion of bodies to rubber plates. Apart from this, such a covering is an insulator of electricity, which is not at all desirable. Tough smooth-planed wooden floors are sufficiently safe, but it is difficult to keep them permanently tight under heat, and still more difficult to keep them clean. To frequently wash such floors is inconvenient in a drying-room. No doubt covering with sheet-lead makes durable, well-conducting floors, and if, as is usual in gun-cotton drying-houses, the room is entered with felt or cloth shoes only, or the workmen work bare-footed, then it is well adapted for its purpose. The only danger is that the workmen may drop a tray, and it is therefore unwise to provide the frames, as is sometimes done, with brass bindings, since it is much better to renew them from time to time than incur the risk introduced by the brass fittings. The best covering for the floors of gun-cotton drying-houses has been found to be linoleum, which gives a perfectly tight and plain but not rough surface. It can be easily cleaned with a moist rag and thus freed from dust. Should a tray fall or a blow be struck on the floor, the shock will to a certain extent be moderated by the elastic linoleum; at the same time this kind of covering is very cheap.

Special attention has to be given to the air conduits. Tubes of sheet-zinc or other metals should be covered outside, or so placed that any dust collected on them can be easily removed. For although the temperature in the drying-room may not be more than 104° F., it is possible, with unsuitable arrangements for the air running in through the pipes, to have a temperature as high as 140° F., in order to ensure a uniform temperature of 104° F. in the drying-room. Any gun-cotton dust collecting near the air inlets, or on the pipes, will thereby be subjected to an undesirable temperature. It is also quite possible that on a bend, a flange, etc., small particles of gun-cotton may collect and take up a large quantity of heat. Therefore the arrangements ought to be so made that at no place in the drying-room can the temperature exceed 113° F.

Since dry gun-cotton absorbs from $1\frac{1}{2}$ to 2 per cent. of moisture by mere exposure to the air, it is necessary before taking it out of the drying-room to put it into tightly-closed bags, made of india-rubber as a rule, or into air-tight boxes. In general, as little work as possible should be done in the drying-room, otherwise the highly-sensitive warm gun-cotton is exposed to the risk of too much friction. The trays are therefore filled in an annex, as a rule, and handed in to the workmen inside the drying-room through a service window.

The time of drying depends on the thickness of the material on the trays, its density, and the temperature in the room. Finely-pulped loose gun-cotton containing about 30 per cent. of moisture requires between forty-eight and sixty hours, if exposed in layers not thicker than $1\frac{1}{4}$ inches. Slabs of about 2 inches in height and of between 1·1 and 1·3 gravimetric density, require about the same time. If the drying be done too rapidly, the gun-cotton swells up in layers and its density decreases.

The temperature is controlled by thermometers placed in suitable positions. Very often electric alarm-thermometers are used; sometimes two in such a way that the one rings at a minimum temperature, say at 95° F., the other one at a maximum temperature, say at 130° F. Unless such alarm-thermometers are allowed to work within wide limits, they are a source of constant annoyance to the attendants, and it is therefore much better to so dimension the heating arrangement and the fan that the temperature can only vary within certain very small limits, and by introducing a regular system of inspection, to ensure the proper working of the drying-house. In order to avoid entering a drying-room containing compressed slabs oftener than possible, the following indicating arrangement can be used:—A spring, or lever, balance is put against a window in the wall, and on it a slab containing slightly more moisture than the slab to be dried. As soon as the index of the balance shows that the slab has attained constant weight, then the slab is perfectly dry, and, of course, the slabs in the room which contained less moisture must also have become perfectly dry. With regular drying of pulped gun-cotton in large quantities it is best to draw a sample once a day and test it in the laboratory, for moisture, in the usual manner.

6. THE PROPERTIES OF GUN-COTTON.

(*a*) Physical Properties.

The gun-cotton as it leaves the boiling-vats, and before it has been pulped, completely retains the structure of cotton, no difference being observable even under the microscope, but it is no longer so flexible as cotton. It feels rougher to the touch, and is more brittle, crackles slightly on squeezing, and has increased in specific gravity. If rubbed between the fingers it becomes electric and adheres to the dry skin. If dry gun-cotton be rubbed in the dark, it emits a phosphorescent glow; this, however, is only the case with perfectly dry gun-cotton. If examined under the microscope with polarized light, ordinary cotton is colourless, whilst gun-cotton exhibits colours. According to Kindt, gun-cotton may be easily distinguished from ordinary cotton by treating it with a solution of iodine in potassium iodide, and subsequently moistening with dilute sulphuric acid. Cotton when so treated turns blue, whilst gun-cotton becomes yellow. The colour of gun-cotton varies between a pure white and strongly yellowish shade. The longer it has been treated with soda the deeper the yellow coloration becomes. Sometimes there are also brownish-yellow parts in the gun-cotton, which are probably due to the formation of iron salt.

Pure gun-cotton is without odour or taste, and free from acid. The gravimetric density of gun-cotton is 0·1 before pulping, 0·3 when pulped, and from 1·00 to 1·30 after compression. Even under very considerable pressure a higher density than 1·4 cannot be obtained. The absolute specific gravity of gun-cotton is 1·5. To test the density of compressed gun-cotton slabs, Bode's mercury apparatus (Fig. 165), the arrangement and use of which is described in Vol. I. p. 302, is most suitable.

According to the pressure applied, compressed gun-cotton has a larger or smaller cohesion and hardness. In general it resembles compressed wood pulp, and well-compressed moist gun-cotton as it leaves the presses has a surface that can be written on with a black lead-pencil.

Gun-cotton is insoluble both in hot and cold water, and it is through this property that complete elimination of acid from the gun-cotton during manufacture is possible, and, since a percentage of water does not alter the composition of the gun-cotton, that it can be brought to its original efficiency by mere drying. Pure gun-cotton is insoluble both in alcohol and ether. On the other hand, all kinds of collodion cotton are completely soluble in a mixture of two parts of ether to one part of alcohol, and on this property is based the usual designation of soluble and insoluble gun-cotton.

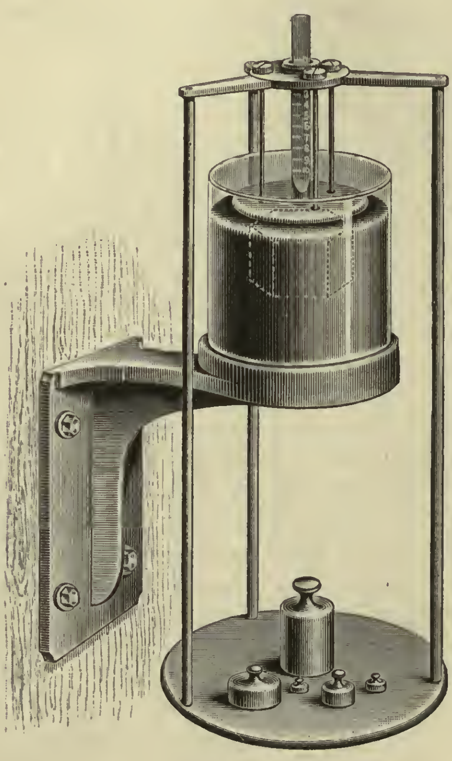

Fig. 165.

Gun-cotton is soluble in all proportions in acetone, acetic ether, sulphuric ether, benzene, nitro-benzene, etc. Hexa-nitro-cellulose (insoluble gun-cotton) is insoluble in nitro-glycerin, but hexa-nitro-cellulose and nitro-glycerin together can be dissolved by acetone. Soluble gun-cotton is partly soluble in nitro-glycerin at ordinary temperature, and completely so at temperatures above 122° F. This property has been made use of by Nobel for making blasting gelatine. By boiling with sodium sulphide gun-cotton can be completely brought into solution, whilst ordinary cotton is not dissolved by this treatment. This is made use of in analyzing gun-cotton. Concentrated solutions of alkalies gradually decompose gun-cotton, especially with heat, but the decomposition frequently takes many days. It shows itself, in the first place, by a black or brown coloration of the gun-cotton, due to the formation of alkaline nitrates and the re-formation of cellulose.

In 1877, Hess and Schwab were the first to notice that a rapid decomposition of nitro-compounds takes place in alcoholic solutions of alkalies; for instance, a 5 per cent. solution of caustic soda mixed with a little alcohol is sufficient to decompose gun-cotton in a few minutes. Caustic potash with alcohol has a very energetic and almost instantaneous action.

The percentage of moisture in air-dried gun-cotton is always between $1\frac{1}{2}$ and 2 per cent., which is less than that of cotton. Even in air saturated with moisture gun-cotton takes up less than 3 per cent., which does not yet materially detract from its inflammability. On the other hand, according to Beckerhinn, the pressure on firing gun-cotton charges decreases rapidly as the percentage of moisture increases.

(*b*) CHEMICAL PROPERTIES.

Pure cellulose has the composition $C_{12}H_{20}O_{10}$. Several other vegetable products of totally different appearance, such as starch, some kinds of gum, dextrin, etc., have also the same composition.

The formation of gun-cotton was formerly considered to be due to the replacement of 3 equivalents of water in the cellulose by 3 equivalents of nitric acid, or to the replacement of 3 atoms of hydrogen by 3 nitro-groups, thus:—

$$C_6H_{10}O_5 + 3HNO_3 = 3H_2O + C_6H_7N_3O_{11}.$$

This product would have the same composition as tri-nitro-cellulose, whilst, by using weak nitric acid, di-nitro-cellulose of the formula $C_5H_8N_2O_9$ would be obtained. Eder found that there are nitro-compounds in which the percentage of nitrogen is between those corresponding to tri- and di-nitro cellulose, and that therefore the formula of the cellulose has to be doubled. He succeeded in producing five kinds of nitro-cellulose :—

			PER CENT.	
$C_{12}H_{14}O_4(O.NO_2)_6$	Hexa-nitro-cellulose	with	14·14	of nitrogen.
$C_{12}H_{15}O_5(O.NO_2)_5$	Penta-nitro-cellulose	„	12·75	„
$C_{12}H_{16}O_6(O.NO_2)_4$	Tetra-nitro-cellulose	„	11·11	„
$C_{12}H_{17}O_7(O.NO_2)_3$	Tri-nitro-cellulose	„	9·15	„
$C_{12}H_{18}O_8(O.NO_2)_2$	Di-nitro-cellulose	„	6·76	„

He did not, however, succeed in making mono-nitro-cellulose. Of the five kinds of nitro-cellulose above-mentioned, the hexa-nitrate alone is insoluble in ether-alcohol and forms gun-cotton properly so called. The others are all soluble in ether-alcohol, and are called collodion cotton.

Vieille made gun-cotton under the most varying conditions with an excess of acid at a temperature of 11° C., so that the water formed should influence the reaction as little as possible. He found in this way eight stages of nitro-cellulose, and assumed therefore that the formula of the cellulose must again be doubled and be $C_{24}H_{40}O_{20}$. He found the following formulæ for the nitrates of the gun-cotton produced by him :—

			PER CENT.	
$C_{24}H_{18}(NO_3H)_{11}O_9 =$	Endeca-nitro-cellulose	with	13·47	of nitrogen.
$C_{24}H_{20}(NO_3H)_{10}O_{10} =$	Deca-nitro-cellulose	„	12·75	„
$C_{24}H_{22}(NO_3H)_9O_{11} =$	Ennea-nitro-cellulose	„	11·96	„
$C_{24}H_{24}(NO_3H)_8O_{12} =$	Octo-nitro-cellulose	„	11·11	„
$C_{24}H_{26}(NO_3H)_7O_{13} =$	Hepta-nitro-cellulose	„	10·18	„
$C_{24}H_{28}(NO_3H)_6O_{14} =$	Hexa-nitro-cellulose	„	9·15	„
$C_{24}H_{30}(NO_3H)_5O_{15} =$	Penta-nitro-cellulose	„	8·02	„
$C_{24}H_{32}(NO_3H)_4O_{16} =$	Tetra-nitro-cellulose	„	6·76	„

Of these nitro-celluloses the endeca- and deca-nitro-cellulose are insoluble in ether-alcohol, whilst the ennea- and octo-nitro-cellulose are soluble. The hepta-nitro-cellulose only becomes gelatinous in acetic ether and ether-alcohol, and the hexa-, penta-, and tetra-nitro-cellulose are only slightly attacked by these solvents. On carrying the nitration of the cotton still further with weak mixtures of acids, products are obtained which are not attacked by the solvents. These contain much unaltered cellulose, and turn a dark colour in a solution of iodine in potassium iodide. The formulæ given by Vieille are not generally accepted, Eder's theory being more favoured. The author has himself made, and on a very large scale, gun-cotton containing

13·65 per cent. of nitrogen, which, according to Vieille's theory, would be impossible.

Gun-cotton was formerly looked upon as a nitro-compound. This view was first contradicted by Béchamp, who considered it to be an ether of nitric acid. That this view is correct is proved by numerous reactions of gun-cotton. Concentrated sulphuric acid decomposes it slowly, even in the cold, liberating nitric acid. Fairly concentrated alkaline solutions (potassium and soda solutions) form alkaline nitrates with the slow re-formation of cellulose at ordinary temperature, and very quickly between 60° and 80° C. Liquid ammonia, according to Béchamp and Guignet, acts in a similar way; whilst, according to Blondeau, intermediary amido-compounds are formed. Chloride of iron (Fe_2Cl_4) and potassium, and ammonium sulph-hydrate (Hadow, von Pettenkofer) also bring about the re-formation of cellulose, whilst the nitric acid liberated oxidizes the iron chloride and transforms the sulph-hydrates into nitrates. All these reactions can only be reconciled on the assumption that gun-cotton is a nitric ether.

(*c*) Chemical Examination.

The analysis of gun-cotton has been done in various ways. Kerkhoff and Reuter weighed the substance in small glass bulbs, which were put through a tap into combustion tubes filled with copper oxide and copper, and there exploded. Peligot conducted the gases formed by the gradual combustion of gun-cotton over incandescent copper oxide and copper. The carbonic acid and water were weighed, the liberated nitrogen measured, and the oxygen, as usual, determined by the loss. Fehling added potassium chlorate to the copper oxide. W. Crum determined the carbonic acid formed by combustion with lead chromate by absorbing it in a eudiometer by caustic potash. The nitrogen was determined by a method of his own. The gun-cotton was brought into contact with twenty times its weight of concentrated sulphuric acid in a graduated tube filled with mercury, thus liberating nitric acid which dissolved the mercury forming nitric oxide. After the reaction was over the level of the mercury was read off, iron sulphate solution added which absorbed the nitric acid, and a reading again taken. Two volumes of nitric acid correspond to one volume of nitrogen.

The safe determination of the composition of gun-cotton is attended with no small difficulty. Many experimenters working with material of different composition and of imperfect manufacture, and therefore with varying properties, obtained results which varied widely. This explains the great discrepancies which will be found on analyzing the following table :—

Components.	Composition of the normal gun-cotton.	Schönbein.	Pelouze.	Fehling.	Kerkhoff and Reuter.	Schmidt and Hecker.	Crum.	Gun-cotton from Hirtenberg.					Redtenbacher, Schrötter, and Schneider.	Beckerhinn.[1]		
														a	*b*	*c*
Carbon ...	24·24	27·43	25·2	25·9	24·6	24·80	24·7	25·6	26·8	26·1	27·7	25·1	23·6 to 25·1	24·47	24·58	24·47
Hydrogen	2·37	3·54	2·9	3·7	2·6	2·75	2·5	2·9	3·1	3·1	2·6	3·0	2·3 ,, 3·0	2·56	2·48	2·46
Nitrogen ...	14·14	14·26	12·6	10·0	14·3	13·50	13·8	12·3	12·5	13·5	11·4	12·4	—	13·72	13·65	13·94
Oxygen ...	59·25	54·77	59·3	60·4	58·5	58·95	59·0	59·2	57·6	57·3	58·3	59·5	—	59·25	59·29	59·13
Increase of weight per cent.	83·33	—	75·0	—	76·2	69·00	77·9	73·3	—	—	—	—	—	—	—	—

[1] From 'Mittheilungen über Gegenstände des Artillerie- und Geniewesens':—
(*a*) Sample of gun-cotton which had been buried in the ground for five years without a wrapper.
(*b*) A sample kept for the same time under water.
(*c*) A sample kept dry.
(*a*, *b*, *c*) Average from two analyses.

From the point of view of its chemical formula, the percentage of nitrogen is the chief thing to be determined in the analysis of gun-cotton, because from this the exact composition can be ascertained. The percentage of nitrogen is also of importance as a measure of the strength of the gun-cotton, as the higher it is the greater will be the energy developed by the gun-cotton. Of course, gun-cotton must be examined for moisture, both for that which is intentionally present and for that which is accidental. The process of analysis that is now generally adopted is as follows:—

(1) *Determination of Moisture.*—In loose gun-cotton an equal-sized sample is drawn from various places in each case, and those from four cases mixed together. With compressed gun-cotton, a slab is taken from about a hundreth part of the whole quantity, and from the centre of each of these a certain quantity is removed. The samples so obtained are rubbed first by hand to a powder, then through a fine wire sieve, in order to mix them as thoroughly as possible, and preserved for the subsequent operations of the analysis. The moisture is determined by drying the gun-cotton in an air-bath at 122° F. to constant weight on small paper trays made by bending up a sheet of paper. The dry weight of the whole sample is then calculated from the result obtained.

(2) *Percentage of Ash.*—Five grammes of dried gun-cotton are burned with a little pure paraffin wax in a platinum or porcelain crucible. The burning may be assisted by an incandescent platinum wire, so that the incineration takes place at as low a temperature as possible. The ashes are mixed with a solution of ammonium carbonate, and repeatedly heated to 200° C. until constant weight is obtained.

(3) *Determination of Calcium Carbonate.*—To 5 grammes of gun-cotton 100 cubic centimetres of water and 20 cubic centimetres of quarter normal hydrochloric acid are added at 15° C. The mixture is boiled for six or seven minutes, filtered, washed out, and the solution run off and cooled down. It is then titrated with quarter normal soda solution, methyl orange or litmus being used as an indicator. The percentage of alkali is calculated from the difference between the number of cubic centimetres of hydrochloric acid and soda solution used. According to another method, 5 grammes of gun-cotton are digested for twenty-four hours with semi-normal hydrochloric acid, and the residue filtered, repeatedly washed, and then dried. The difference in weight is the percentage of calcium carbonate.

(4) *Heat- or Stability Test.*—About 1 gramme of gun-cotton is

submitted to the heat test conducted in the ordinary manner, when the characteristic coloration should not appear for at least fifteen minutes. In Great Britain ten minutes are specified. The Prussian Government requires that the samples should stand the heat test for twenty-five minutes at 80° C., using zinc-iodide paper. The manner of conducting the heat test is described in the Appendix.

(5) *Temperature of Ignition.*—0·05 gramme of gun-cotton is placed in a test-tube which stands in an oil-bath heated to 100° C. The temperature is gradually increased, and that at which the gun-cotton takes fire noted. For good gun-cotton this should not be below 180° C. (356° F.). Ignition takes place at a slightly different temperature, according to whether the heating has been gradual or sudden. If the temperature of ignition when suddenly heated is to be determined, about a dozen test-tubes are put into the cover of the oil-bath, which has a corresponding number of holes in its cover. The heat is increased gradually, and at various temperatures small quantities of gun-cotton are dropped into different test-tubes.

(6) *Determination of Nitrogen.*—There are various methods for this, and that of W. Crum has already been described. Dumas' method is well known, and is described in almost every text-book on chemistry. In France, Schlösing's method is used, and it has been modified by Schultze and Thiemann. According to a description kindly sent to the author, it has been so improved upon by Dr. Schmidt, that, as the author has had the opportunity of satisfying himself, it can be carried out very easily, and gives very exact results. The apparatus used is illustrated in Fig. 166. It consists of a glass cylinder in which a graduated tube is fixed by means of two stoppers. On the top of the tube is a glass cock (a) with funnel (y). It stands in a glass tank (w) with a perforated india-rubber stopper at the bottom, into which a glass T-piece is fixed communicating on one side with a vessel containing sodium solution, and on the other hand with a measuring tube (c). The glass cock (a) and the pinch-cock (b) are opened, and the sodium solution allowed to gradually run into the graduated tube, tube c being closed. As soon as the graduated tube is filled with sodium solution to within about half-an-inch of the filling funnel (y), the taps (a and b) are closed and c opened. Cold water runs into the lower part of the glass vessel through the tube d and out through the tube e. For the estimation, 0·600 to 0·650 gramme of gun-cotton is weighed in a closed weighing-glass, and put into the flask (f). The weighing-glass is then rinsed out with about

25 cubic centimetres of water. Two glass tubes are inserted into the flask through separate holes in the stopper, one of which dips by means of suitable connections into the test-tube (r) and the

Fig. 166.

other into the tank (w), which is filled with soda solution. By alternately opening and closing the pinch-cocks (g and h), whilst the flask is heated to boiling, the air is expelled. As soon as all air is expelled, the pinch-cocks (g and h) are closed, and the gas flame taken away. 25 cubic centimetres of a concentrated solution of iron chloride and 10 to 15 cubic centimetres of concentrated hydrochloric acid are next run into the test-tube (r), and on opening the pinch-cock (g) are sucked into the flask (f), care being taken that no air gets in with them. The test-tube is rinsed with a few cubic centimetres of water, the pinch-cock (g) closed, the tube (i) put under the graduated tube, and nitric oxide gas developed by heating the flask. As soon as the gas pressure in the flask becomes higher than the pressure of the air outside, the connecting tube swells up, whereupon the pinch-cock (h) is opened, and the contents of the flask boiled, with frequent shaking, until bubbles of nitric oxide cease to rise in the sodium solution, and until a rattling noise caused by the hydrochloric acid distilling is heard. The pinch-cock (h) is then closed, and the cock (g) opened. The graduated tube is again brought down tight against the rubber stopper in the tank (w), and the whole apparatus allowed to cool until the water, running out through e, has the same temperature as that entering the cooling-jacket at d. This can be ascertained by thermometers placed in the glass and in the tube (c). If the level of the sodium solution in the measuring tube is then brought, by lifting or lowering this tube, to the same height as that of the sodium solution in the graduated tube, then the volume of nitric oxide gas contained can be read off to one-tenth of a cubic centimetre, and the percentage of nitrogen can be calculated in the usual manner. The sodium solution in the tank is run off after use through the connecting tube (l) into a vessel below the table.

The iron chloride solution is prepared by dissolving iron nails in concentrated hydrochloric acid, care being taken that iron is present in excess. When the development of hydrogen has ceased, the solution is filtered warm through a folded filter, and the filtrate acidulated by adding a few drops of concentrated hydrochloric acid. The sodium solution, which serves as a water seal, has a specific gravity of between 1·210 and 1·263.

The results obtained with the Schultze-Thiemann apparatus are on an average a little (about 0·2 per cent.) higher than those obtained with Lunge's nitrometer.

Of the various other methods the nitrometric method deserves special mention. The forms that have been most generally used are those of Hempel, Lubarsch, and Lunge. Lunge's nitrometer, on account of the convenience and simplicity of its manipulation, and the exact results it gives, is generally or almost exclusively in use. Since there is scarcely a chemist now-a-days who is not conversant with it, a detailed description of its manipulation would be superfluous. Briefly it is as follows:—About 0·6 gramme of gun-cotton is weighed in a small weighing flask, a little concentrated sulphuric acid added, and the whole allowed to stand for from twenty to thirty minutes until all the gun-cotton is completely dissolved. It is then washed into the nitrometer by means of concentrated sulphuric acid, the flask rinsed with about 10 cubic centimetres of dilute sulphuric acid, and the funnel of the nitrometer also washed with a further quantity. After shaking the nitrometer and cooling it in the usual way, the number of cubic centimetres of nitric oxide developed are read off. It is advisable to again note the temperature after about twenty minutes. The author has been informed that by dissolving gun-cotton in sulphuric acid cooled with ice, the quantity of nitrogen found is about 0·2 per cent. greater, but very careful experiments made simultaneously by several chemists have not confirmed this statement.

(7) *Determination of Non-nitrated Cellulose.*—Five grammes of dry gun-cotton are boiled for half-an-hour with a saturated solution of sodium sulphide, allowed to stand for twenty-four hours, and then decanted. After this it is again boiled for half-an-hour with a fresh quantity of sodium sulphide solution. The residue is collected on a dry cloth filter of known weight, and washed with hot distilled water until the addition of lead acetate to the washings no longer gives a coloration. It is then washed with dilute hydrochloric acid, and finally with water, until the filtrate gives no reaction with silver nitrate. The filter is dried on a watch-glass at 50° C. The weight of the dried filter, deducting that of the ash obtained by calcining the filter in a platinum crucible, gives the weight of the non-nitrated cellulose.

(8) *Determination of Soluble Gun-cotton* (*Collodion Cotton*).—The determination of the percentage of soluble gun-cotton in the sample is based on its behaviour in a mixture of ether and alcohol in which it is soluble, whilst gun-cotton proper is insoluble in it. As a rule, 5 grammes of gun-cotton are weighed in an Erlenmeyer's flask, 200 cubic centimetres of a mixture of two parts of ether of 0·735 specific gravity and one part of alcohol of 0·805 specific gravity are added, and allowed to remain for twelve hours, during which time the whole is frequently shaken. The supernatant liquid is then poured off, more ether-alcohol added,

and after standing two hours the solution is filtered through a dried and weighed cloth filter. The residue on the filter is washed repeatedly with ether-alcohol, and the filter then dried at 50° C., the difference giving the percentage of soluble gun-cotton. The disadvantages of this process are that a solution of soluble gun-cotton in ether-alcohol only passes through a filter with difficulty, and that the use of an air-pump does not materially help filtration. Towards the end a gelatinous mass of collodion remains on the filter, which makes perfect filtration impossible. The new English method is therefore much more practical, and also more exact. According to it, 50 grains of gun-cotton are put into a stoppered glass cylinder of 200 cubic centimetres capacity, 150 cubic centimetres of ether-alcohol are poured over it, and the whole digested for six hours with frequent shaking, after which it is allowed to settle. As soon as the solution above the gun-cotton has become clear, 75 cubic centimetres of it are put into a small weighed flask, the major part of the solvent driven out by distilling it on a water-bath, and the rest in a water-bath at a maximum temperature of 120° F. until constant weight is obtained. Multiplying the weight of the soluble gun-cotton extracted by four, gives the percentage of soluble gun-cotton in the sample directly.

The examination of collodion cotton is done in the same way, and with the same apparatus, as gun-cotton. Here the difficulty of determining the soluble gun-cotton exists in a still larger measure, and therefore the English method is again preferable.

According to Lieut. John P. Wisser, gun-cotton is examined in America as follows :—

After the calcium carbonate has been eliminated, the ash is determined by treating a weighed quantity of gun-cotton in a platinum crucible of known weight, with concentrated nitric acid, carefully evaporating it to dryness and burning the residue. The increase of weight gives the quantity of ash.

Mono- and di-nitro-cellulose are estimated by treating 5 grammes of gun-cotton, first with 200 grammes of ether-alcohol in the proportion of 3 to 1 for twelve hours, then with the same quantity in the proportions of 2 to 1 for six hours, and then with the same quantity in the proportion of 1 to 1. Between each operation time for settling is given, and after the third treatment the liquid is poured through a folded filter. After this, it is washed twice with pure alcohol, twice with diluted alcohol, and finally several times with water. The residue is put on to the filter, dried at 60° C., and weighed. The loss of weight shows the proportion of lower nitrated cellulose.

In order to determine the non-nitrated cellulose, a weighed quantity of gun-cotton is boiled for fifteen minutes in a concentrated solution of sodium stannate, whereby nitro-cellulose passes into solution. This is then filtered, and the residue washed, dried at 100° C., and weighed. The sodium stannate is freshly prepared for each determination by melting caustic soda with tin and lixiviating the fused mass.

(*d*) MECHANICAL PROPERTIES.

(1) *Decomposition of Gun-cotton.*

In former times spontaneous decompositions of gun-cotton, seemingly without any exterior cause, have been observed, some of which ended in an explosion. Since special care has been devoted to the manufacture of gun-cotton and to the elimination of all, even the slightest, traces of acidity, such decompositions no longer occur. There is no doubt that the use of impure cotton, bad acids, or imperfect and insufficient washing, will lead to the decomposition of the lower nitro-products formed during nitration, which may also occur if the subdivision of the gun-cotton is not fine enough, as then an efficient washing is impossible. To such causes the widely-differing results obtained by various experimenters must be ascribed. De Luca experimented with some gun-cotton of French manufacture, all samples of which commenced to decompose on exposure to light. The decomposition once started could not be stopped, and was accelerated by slightly heating. According to this authority, the decomposition commences with the evolution of nitrous gases, the mass decreases considerably in volume, and leaves a residue containing small quantities of a nitrogenous substance, and traces of formic, acetic, and oxalic acid, glucose, etc.

Similar results were obtained by A. W. Hofmann from a sample of gun-cotton thirteen years old. Abel, on the other hand, demonstrated by a very thorough investigation of gun-cotton, that when carefully prepared it could be heated up to 194° F., that gun-cotton packed in cases could be heated for six months to a temperature of 122° F. without decomposing, and that sunlight had no action at all on it. Trauzl also obtained similar results.

In general, it may be said that no nitro-compound will stand heating to temperatures above 160° F. for any prolonged period. At 194° F. even the best and purest product is sure to decompose within a few hours, and even pure gun-cotton cannot be exposed to a temperature of 122° F. without injury to its capability of subsequently standing the heat test. It is true that in this case no decomposition takes place, and that the gun-cotton may, under favourable circumstances, be kept for an indefinite time after having been so treated; but whenever it is heated again to 160° F. for the purpose of the heat test, it will at once give a distinct reaction. In general, it would appear that only the most perfect products will stand a temperature of from 113° to 122° F. for some months without their capability of standing the heat test being impaired.

The spontaneous decomposition of gun-cotton made itself apparent very early after the manufacture of gun-cotton on a large scale had been commenced, but instead of trying to find the causes of it, and to eliminate them, preventive means were sought for. Thus Von Lenk had already added sodium silicate and sodium carbonate, while Prentice soaked the gun-cotton fibres in molten paraffin wax, and later on in a solution of india-rubber. The mere addition of sodium carbonate has proved very effective, since gun-cotton so treated has been heated seven hours a day for three weeks to between 203° and 212° F. without decomposing.

Bleekrode even recommended moistening the gun-cotton with carbon disulphide, benzene, etc., which could be evaporated off before using, since he found that on igniting only the liquid employed would burn, and not the gun-cotton; this, however, is always a very risky proceeding.

At present an addition of calcium carbonate is generally made, but on theoretical grounds, and also from the author's experiments, such an addition is quite unnecessary. The only reason for it is to at once neutralize any acid which may be formed by decomposition or be otherwise liberated. Apart from the fact that the small quantity of alkali added (as a rule, about 2 per cent.) is distributed so widely in the mass of gun-cotton that probably any small traces of acid set free would not at once meet with alkaline particles, it has also to be remembered that pure gun-cotton does not contain any trace of acidity that can be detected by the sensitive heat test, and that consequently it is only acid liberated by decomposition which would have to be neutralized. At ordinary temperatures,—that is, those occurring under normal circumstances of storage and carriage—decomposition of gun-cotton, so far as present experience goes, is out of question. If higher temperatures obtain through unfavourable conditions, then the addition of alkali is no remedy. Moreover, in no case is it efficient for a prolonged time, and it only prevents the detection of an incipient decomposition. Alkalies, too, will themselves in course of time decompose gun-cotton and nitro-compounds in general, especially if they are subjected to somewhat high temperatures (86° to 122° F.).

It has been found that dynamite, after a year's storage in tropical climates, no longer contained any trace of alkali. Gelatine dynamite, which was stored in an English magazine for a year, lost in this time 96½ per cent. of the magnesium carbonate it originally contained. Gun-cotton to which alkali had been added, and which was exposed for several weeks to a temperature of 122° F., did not stand the heat test so well as similar gun-cotton without any added alkalies. This is very markedly shown if, instead of the calcium carbonate, sodium carbonate be added, and still more so if ammonium carbonate be used. These substances decompose gun-cotton with the formation of alkaline nitrates. These again have so little stability that finally they constitute the probable cause of the stability of the gun-cotton itself deteriorating. This subject will be more fully dealt with in connection with gelatine dynamite. An addition of aniline makes the gun-cotton permanently alkaline and capable of standing the heat test for a very long time, but the gun-cotton is coloured brown by it, and, as a matter of fact, it is only an adulteration.

In order to make gun-cotton insensitive to mechanical actions, and in order to prevent decomposition as far as possible, Abel recommended that gun-cotton should be stored in a wet state, and this plan is generally adopted at the present time.

Colonel Hess found that gun-cotton stored for a prolonged time in water contaminated with organic matter may undergo some alterations, which show themselves principally by the discoloration of the attacked parts. A sample examined by him, which had been stored under water for two years, had become dark grey in colour, and still remained of a pale grey colour after it had been washed in pure water. Under the microscope it was shown to be contaminated by structureless, partly humous, masses. Hess succeeded in purifying this gun-cotton by boiling it with a solution of one part of potassium permanganate in 200 parts of water. This was then decanted and the gun-cotton boiled in a 1 per cent. solution of hydrochloric acid. After repeated washing, with the addition of about 2 per cent. of sodium carbonate to the last washing water, the gun-cotton so purified was of a brilliant white and in every respect equal to unattacked gun-cotton.

(2) *Ignition of Gun-cotton.*

If ignited in open air gun-cotton burns with a large orange-yellow flame. In consequence of the exceedingly rapid propagation of the ignition (according to Piobert, it is eight times as quick as in ordinary powder), small quantities of gun-cotton can be burned over black powder without igniting it. In vacuo gun-cotton behaves in a similar manner to black powder. It can be exploded by a violent blow between hard bodies, but as a rule only the particles directly struck detonate, excepting where transmission of the explosive wave is readily possible, such as, for instance, along a steel bar. Gun-cotton can be ignited by slow heating. Horsleigh found with one kind of gun-cotton that the temperature of ignition was 163° C., and with another kind 223° C. Good gun-cotton, when rapidly heated, should not ignite below a temperature of 180° to 184° C.

(3) *Products of Combustion.*

Hecker and Schmidt, and also Teschenmacher and Porret, have examined the gaseous products of combustion from gun-cotton deflagrated in vacuo. Károlyi repeated these experiments, examining first the products obtained in vacuo and then those in air. The first set of determinations was made in a eudiometer about one metre long, through the upper part of which a platinum wire was fused. From this he suspended 15 to 20 milligrammes of gun-cotton. In the vacuum produced the decomposition was started by electric incandescence. The gases formed could of course be analyzed in the eudiometer. In order to burn gun-cotton under increased pressure, it was closed in a small air-tight iron cylinder which was placed in the interior of an exhausted 60 lbs. bomb (about 5216 cubic centimetres cubical contents). This could be connected to a eudiometer by a tube fitted with an air-tight tap, and the gases filling the bomb, after the gun-cotton had been exploded and the cylinder burst, could be transferred to an apparatus for gas analysis. Károlyi ascertained from his experiments that 10 grammes of gun-cotton were required to get trustworthy results.

Hecker and Schmidt obtained on an average 588 cubic centimetres of gas from 1 gramme of gun-cotton; Teschenmacher and Porret, 483 cubic centimetres; Károlyi, 755 cubic centimetres (from gun-cotton exploded under a high pressure). All volumes are reduced to 0° C. and 760 millimetres pressure.

The following table shows the composition (in percentage of volume) of the gases as found by the various experimenters :—

	Hecker and Schmidt.	Teschenmacher and Porret.	Károlyi.	
			a	b
	Burned in vacuo.			Exploded under high pressure.
Carbonic oxide	37·91	19·02	28·55	28·95
,, acid	13·32	7·63	19·11	20·82
Marsh gas	—	—	11·17	7·24
Olefiant gas	4·63	—	—	—
Cyanide	—	3·79	—	—
Nitric oxide	15·35	18·08	8·83	—
Nitrogen	4·03	3·82	8·56	12·67
Aqueous vapour ...	24·76	47·66	21·93	25·34
Hydrogen	—	—	—	3·16
	100·00	100·00	98·15	98·18

The most significant conclusion to be drawn from Károlyi's analysis, which agrees also with practical experience, is the fact that the state of the gases depends upon the pressure and temperature of the explosion. Károlyi explains the disappearance of the nitric oxide on combustion under high pressure by the assumption that at the increased temperature marsh gas and hydrogen act as reducers by taking oxygen from the nitric oxide, and that consequently carbonic acid, carbonic oxide, and water are formed with the liberation of free nitrogen. The high percentage of these constituents supports this view. Both in cases a and b the gases are combustible; this circumstance is of practical importance in certain blasting operations.

The products of combustion of gun-cotton, like those of black powder, are poisonous, on account of the presence of carbonic oxide and carbonic acid. The products of combustion of gun-cotton are, with the exception of about 2 per cent. residue, gaseous at the temperature of explosion. The largest share in the propelling force falls upon the carbonic acid, carbonic oxide, nitrogen, marsh gas, and hydrogen. According to Sarrau and Vieille, the products of combustion vary with the density of the charge, since with a higher density the products of decomposition are exposed to increasing pressures which may differ from each other by several thousands of atmospheres. In this case the percentage of carbonic oxide will always be diminished and that of carbonic acid increased.

If gun-cotton is ignited at or near ordinary temperatures, a large quantity of nitrous vapours are developed, together with carbonic oxide.

7. RESULTS OF WORKING.

The results in the manufacture of gun-cotton depend on so many factors that it is hardly possible to give data that will be generally applicable. In certain cases it may be preferable to work so as to get an inferior yield, if, by doing so, compensating advantages are gained, such as using acids of inferior value or different machines and apparatus, since such a course may sometimes effect an economy in the manufacture.

The losses the raw cotton waste is subject to from handling and purifying varies within wide limits. According to the 'Centralblatt für die Textil-Industrie' of 1890, the losses are:—

	PER CENT.
Moisture	3 to 15
By carriage and unpacking	2 to 5
By boiling and washing	5 to 40
By bleaching with chlorine	15 to 20

In addition to these, losses also occur in "teasing" the cotton, but in general it can be assumed that with good gun-cotton waste, which is not contaminated by grease or otherwise soiled, the losses will be within the lower figures given above.

Theoretically, 183½ parts of gun-cotton should be obtained from 100 parts of dry cotton. When working on a large scale, however, this yield cannot be obtained, since the whole of the product obtained is not pure tri-nitro-cellulose, as it also contains non-nitrated and less highly nitrated cotton. According to the results obtained in several factories, the actual yield is between 153 and 156 parts of gun-cotton for each 100 parts of dry cotton nitrated.

The consumption of acids varies within very wide limits, according to the process used for the revivification of acids, to the number of nitrations which are made with one and the same original quantity of acids, and also according to the percentage of nitrogen which the gun-cotton made contains. When making the highest nitrated gun-cotton with a maximum of 2 per cent. of soluble cotton, the consumption of 4·25 parts of nitric acid and 9 parts of sulphuric acid for each part of raw cotton treated is good work; but the author knows of factories where the consumption is as much as 9 parts of nitric acid and 18 parts of sulphuric acid. The yield of collodion cotton is only slightly less than that of gun-cotton.

The quantity of waste acid can, as a rule, be calculated from its percentage of nitric acid by taking the total quantity of sulphuric acid used and adding to this the quantity of nitric acid and water contained in the waste acids. The amount of water originally contained in the sulphuric acid has, of course, to be included. A loss of about 1 per cent. occurs in pulping gun-cotton. With proper arrangements for pressing, the process should not give rise to any loss.

8. USE OF GUN-COTTON.

The much more powerful effect of gun-cotton as compared with that of black powder, and its property of burning without leaving any residue or giving off smoke, have led to many attempts to utilize it as a propellant and as a blasting material. Those referring to its use as a propellant in fire-arms will be dealt with in a subsequent chapter dealing with smokeless powders.

The possibility of using gun-cotton for blasting operations was, properly speaking, made possible by the discovery of Mr. E. O. Brown, one of Sir Frederick Abel's assistants, that gun-cotton could be detonated by small charges of fulminate of mercury. Since that time gun-cotton has been used, especially in Great Britain and Germany, as an explosive in military engineering. On account of its loose structure, its use was at first attended by many difficulties, but when the practicability of compressing it into cartridges had been demonstrated, all objections to its use were removed, and its suitability for industrial blasting operations was of course immediately obvious.

In 1847 successful attempts in this direction had already been repeatedly made. Messrs. Hall and Sons had used gun-cotton for making some "cuttings" in clay and sand-stone, and had found that 1 part of it gave as great an effect as 6 parts of black powder. A French commission, under the chairmanship of the Duke of Montpensier, also conducted some experimental blasts in rock with gun-cotton. For fuller information on the action of gun-cotton in blasting operations in general, the author must refer readers to his hand-book on 'Blasting,' as the subject is too large a one to treat adequately in the present work, and is, moreover, hardly within its scope.

From the composition formula of gun-cotton, it is evident that it does not contain sufficient oxygen to effect perfect combustion of the carbon and hydrogen. The addition of various materials to supply this deficiency has consequently been recommended. In 1851 Coxthupe and Caldwell proposed dipping gun-cotton for fifteen minutes into a concentrated solution of potassium chlorate and subsequently drying it.

Combes proposed mixing 5 parts of gun-cotton with 4 parts of potassium chlorate, or the same quantity of potassium nitrate, or with 3½ parts of sodium nitrate. This mixture gave effects equal to 9 parts of gun-cotton.

Sir Frederick Abel suggested the manufacture of cartridges by compression from a mixture of gun-cotton and potassium nitrate. This process is still used in this country for making the explosives known as "Potentite" and "Tonite," and in Germany for the manufacture of the so-called "Miners' cartridges." In the two latter, however, barium nitrate replaces the potassium nitrate, their composition being gun-cotton 52½ parts and barium nitrate 45½ parts. The process, as a rule, consists in thoroughly mixing finely-pulped gun-cotton in suitable machines or incorporating-mills with saltpetre and water, a little sugar being sometimes added. The mass is then formed into cartridges in hydraulic presses. The cartridges are of course dried afterwards.

The other uses of gun-cotton only require brief mention. Collodion cotton is used for making celluloid, and transparent roofing in connection with solutions of resin in surgery, for making collodion; and as a filtering material for strong acids, alkalies or liquids that are easily decomposed, such as solution of potassium permanganate, etc. Abel obtained very pretty pyrotechnic effects by saturating gun-cotton with solutions of salts capable of imparting colour to the flames. Finally, an important application of gun-cotton is in the manufacture of detonating fuses (*cordeaux détonants*), of which fuller details are to be found in a subsequent chapter.

9. GENERAL REMARKS ON THE ERECTION OF GUN-COTTON FACTORIES.

Much of what has been said in the chapter dealing with the construction of black powder factories is equally applicable to gun-cotton factories. They are not considered from the same point of view in every country, however. Although, speaking generally, the manufacture of gun-cotton up to the process of pressing is not considered dangerous, because it is always mixed with large quantities of acid or water, yet in some countries the erection of such factories is regulated by special laws. In Great Britain the manufacture of gun-cotton, as long as it is in a wet state, is unrestricted, although it still comes under the Explosives Act.

On account of the large quantities of acid vapours developed in nitrating, it is necessary to keep the building in which this process is carried on some distance apart from the other buildings of the factory. It is also essential that those buildings, in which the gun-cotton undergoes purification by pulping and washing to render it perfectly stable, should, if possible, be about 100 yards from the nitration building, and so placed that the prevailing winds shall not carry acid vapours from it towards them. This is important, since traces of acid vapour are carried to great distances by the wind, and might render all efforts to make the gun-cotton pass the heat test futile. Ample ventilation and light are important in all buildings in a gun-cotton factory. In large quantities the acid vapours are not only opaque, but also of a dark colour, and they would render work very difficult unless ample lighting were provided. In the nitrating-room, care must be taken to ensure a good natural ventilation, or the nitration should be carried out in open sheds.

With the modern nitrating apparatus it is customary and very convenient to have a hood over a number of the nitrating vessels connected with a chimney with a good draught, or with a tower made of earthenware pipes, in which the draught is sometimes created by an exhaustor, such, for instance, as that illustrated in Vol. I. p. 148. A Lunge-Rohrmann plate-tower may be used with advantage for condensing the acid vapours.

A further important desideratum for a gun-cotton factory is a supply of pure water free from mechanical impurities, and neither acid nor strongly alkaline. The use of river-water without ample means for filtering is precluded, since gun-cotton itself acts like a filter, and any impurities in the water would be caught by it and their subsequent removal could only be effected with great difficulty. The use of mine-water, or water running over minerals, or spring-water is not, as a rule, to be recommended. Where the only supply available is not sufficiently pure, the water must be purified before use by suitable means, of which a good filtering arrangement is especially essential. Either sand, wood-wool, gravel, or similar filtering media may be used. In some cases it may also be necessary to treat the water chemically; this will of course depend upon the amount and quantity of impurities present.

The water running away from the gun-cotton houses should pass through suitable settling apparatus in order to remove all finely-suspended particles of gun-cotton. It may also be necessary to neutralize the water if it be strongly acid before it is allowed to run outside the factory ground.

Those parts of the factory in which the gun-cotton is pressed and dried must be arranged and worked according to the regulations for explosives factories. Drying-houses have been referred to in detail above.

The buildings for pressing and shaping gun-cotton should be constructed to be as light and strong as possible. It is advisable, therefore, to make them with main walls about 18 inches thick, and to provide them with large windows, and further to have either one wall made of wood and glass, or to provide a very light roof, so as to permit of an easy and rapid dispersal of the gases in case of an explosion. The conditions governing the production and transmission of the power required, as well as of the transmission of steam, air, and acid, are identical with those dealt with in connection with black powder factories, Vol. I. p 277.

The amount of power required to drive the various machines used in the manufacture of gun-cotton is, according to the author's experience, as follows:—

	HORSE-POWER
For an acid centrifugal with a 40-inch basket	3
,, a washing machine, 13 feet long, 6 feet wide	2
,, a water centrifugal machine, with a 40-inch basket ...	2 to 3
,, a pulper 13 feet 6 inches long and 6 feet 6 inches wide	6 to 8
,, an air-pump (according to size)	4 to 6
,, a hydraulic pump, with two 3-inch and two 2-inch pistons	16

The consumption of water may be taken to be from 15 to 20 gallons for each pound of gun-cotton made. In addition to that required for generating the necessary driving power, about 200 to 300 square feet of boiler heating surface have to be allowed in a factory producing daily 500 to 750 lbs. of gun-cotton, in order to provide the steam necessary for boiling, heating, etc.

The consumption of gun-cotton has considerably decreased within the last few years, as picric acid and bodies allied to it have been found to be very suitable explosives for charging shells, and for the purpose of military engineers in general. A further cause is that most governments have for a long time been provided with all the stock of gun-cotton they require for torpedoes and submarine mines, and the likelihood of the renewal becoming necessary in the immediate future is highly improbable. On the other hand, within the last few years considerable quantities of gun-cotton and collodion cotton have been required for the manufacture of smokeless powders, although the demand for this purpose rarely affects private factories.

CHAPTER IX

OTHER KINDS OF NITRO-CELLULOSE

WITH gun-cotton almost pure cellulose in the shape of cotton fibre is available, and this has only to be purified from fatty substances to render it fit for conversion. Nevertheless attempts have also been made to convert less pure cellulose into explosive bodies by the action of nitric acid.

1. PYRO-PAPER.

As was only natural, the idea occurred of obtaining a suitable pyroxiline substance from paper, and a "vegetable" paper was, in fact, used for this purpose. This was converted into the so-called pyro-paper by dipping it for two minutes in a mixture of nitric and sulphuric acids. After this it was first washed with water, with the addition of some ammonium hydrate at the end, then with pure water, and finally dried.

2. SCHULTZE POWDER.

The rifle and blasting powder invented by E. Schultze of Potsdam, a captain of the German artillery, has nitrated wood substance as its base; it is at present manufactured in Great Britain. In preparing it, a thorough purification of the wood, especially the removal of sap, has first to be effected. Hard wood is cut into thin sheets, and these, by means of a punch, into regular grains. These are first boiled several times with dilute solution of soda. After being washed they are steamed, then again washed with water, and finally dried. The grains after this treatment are bleached with bleaching-powder, and again washed and dried. Six parts by weight of the purified and bleached wood grains are then thoroughly soaked for from two to three hours in

100 parts of a mixture of 28·5 parts of nitric acid (specific gravity 1·48 to 1·50) and 71·5 parts of sulphuric acid (specific gravity 1·84) in order to nitrate them, the acid mixture being kept cool by a suitable arrangement. When nitration is finished, the greater part of the acid is extracted by means of a centrifugal machine, the nitrated grains washed and boiled with a dilute solution of soda, again washed, and then dried. In order to increase its effect, the pyroxiline so obtained is impregnated with a solution of saltpetre, to which barium nitrate or potassium sulphocyanide is also sometimes added. The impregnated grains are then again dried at a temperature between 90° and 111° F. For impregnating each 100 parts of pyroxiline, 26 parts of saltpetre (or 22½ parts of saltpetre and 7½ parts of barium nitrate) dissolved in 220 parts of water are used.

To increase its safety, Schultze recommended that his powder should be stored wet, and dried when required for use. According to him, the advantages of this powder are that it gives a double or triple effect, no smoke, and almost no residue. It is also asserted that the gases have no injurious action on the respiratory organs.

In practice these good results were not always obtained. Shooting experiments made with the powder in Switzerland gave unfavourable results: 49·5 grammes of it carried the ball of an experimental mortar just as far as 92 grammes of ordinary black powder, but the variations in range were very considerable. Since the volume of the Schultze powder is about 2½ or 3½ times that of black powder, a larger volume was required to obtain the same effect. Attempts at using the powder in carbines were not attended with favourable results. It is true that the residue was only small and little smoke was observed, but it was difficult to clean out the residue, if ungreased projectiles were used.

Blasting experiments made in the districts of Saarbrücken and Mansfeld with Schultze powder gave better results, although hardly any advantage over black powder was observed. It was pointed out, however, that on account of the small amount of smoke, it was only necessary to suspend work in the mines for a short time. The cost was about the same as in the case of black powder.

On the other hand, Schultze powder has been used successfully, especially in Great Britain, as a sporting powder, and is very much liked. It is said to give but a slight recoil, no smoke, and good results in shooting. It is also said to stand moisture and climatic changes well.

In 1886 Schultze patented a sporting and target powder, which he makes by adding various proportions, up to one-twelfth, of a mono-nitrate made from resinous products, such as turpentine, oil of turpentine, resin, etc., to a powder consisting of wood nitro-cellulose and saltpetre. Those resins which are solid and friable are finely pulverized. About one and a half parts by weight of nitric acid of 1·420 to 1·460 specific gravity are poured over them and heated on a water-bath with constant stirring, whereby a constantly increasing frothing takes place, which brings the nitrated resin into a state of fine subdivision. In a similar way the soft or liquid resinous materials are treated, but the nitric acid is only added gradually. The resulting nitro-products are subsequently washed.

Schultze recommends as a powder giving a strong penetration, and yet free from disruptive effects, a mixture of 1 part of nitro-resin, 5 parts of nitro-cellulose, and 6 parts of saltpetre, which are granulated by the addition of a binding material and glazed by paraffin wax, collodion, and similar materials.

The wood-pulp commonly made in wood-pulping mills, consisting of fine fibres of wood, and which is usually worked up into paper, has been nitrated by Darapsky, and recommended under the name of "yellow shooting powder."

Lannoy and Co. made a mixture of 65 parts of sodium nitrate, 13 parts of sulphur, and 22 parts of nitrated wood, sawdust, or bran as a blasting powder under the name of "white powder," (sometimes called "lithofracteur"), but it has not found favour in practical hands.

F. Krantz and Co., of Bautzen, sell a powder under the name of Bautzen blasting powder, which consists of equal parts of nitrated wood and saltpetre. It is sometimes also compressed into cartridges, and is said to give very good results.

3. NITRO-STARCH.

Starch has the same composition as cellulose ($C_{12}H_{20}O_{10}$), and Braconnot and Pelouze have shown that, like it, it is transformed into an explosive substance by concentrated nitric acid. Von Uchatius first used sulphuric acid in conjunction with nitric acid, but since starch clogs much in the mixture of these two acids, its use impedes the reaction. Uchatius' process was to dissolve 1 part by weight of potato starch in 8 parts of fuming nitric acid, the temperature being prevented from rising. The thick solution was then poured in a fine stream into 16 parts of concentrated sulphuric acid, being meanwhile well stirred. The resulting paste was allowed to stand for twelve hours, and the mass was then washed with an excess of water, soda being added towards the end. After repeated washing, the white powder was dried at a temperature of from 122° to 140° F.

Volney (American patent, No. 366281) stirs ground and dried starch into a mixture of concentrated nitric and sulphuric acids, which are kept cool during the operation. The nitro-starch separated in a granulated form is then washed, dried, and pulverized.

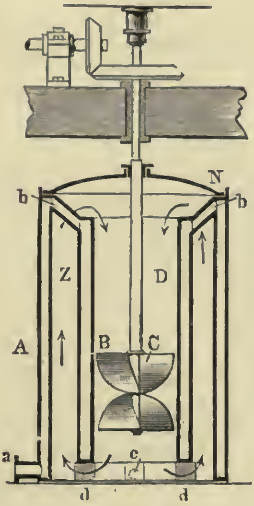

Fig. 167.

So far nitro-starch has not been used on a large scale, because by itself it is very hygroscopic, and on account of insufficient purification it is liable to spontaneous decomposition. Only quite recently the Actien-Gesellschaft Dynamit Nobel has taken up nitro-starch as an ingredient for smokeless powder, and has patented a process for its rational manufacture.

The nitrating apparatus is illustrated in Fig. 167. It consists of two lead vessels (A and B) put one within the other, each of which is surrounded by a cooling-jacket, and connected with the other by the pipe (b). The inner vessel rests on earthenware tiles (d), and in it moves a paddle stirrer (C), which is revolved by a pair of cog-wheels. The cooling-water enters the outer jacket by the pipe (a), passes through the tube (b) into the inner cooling-jacket, and leaves this through the outlet pipe (c). The vessel is filled with nitric acid of 1·501 specific gravity, and through an opening in the cover (N), ground potato starch, which has been dried at 212° F., is introduced into the inner vessel (D), and distributed in the acid by means of the stirrer (C). The movement of the stirrer forces the acid downwards through the tiles into the interstice (Z), and from there back into the inner space (D), thus causing a constant circulation of the acid to take place, which brings the starch into solution. The proportion of starch to nitric acid is as 1 : 10, and the temperature is kept between 68° and 77° F.

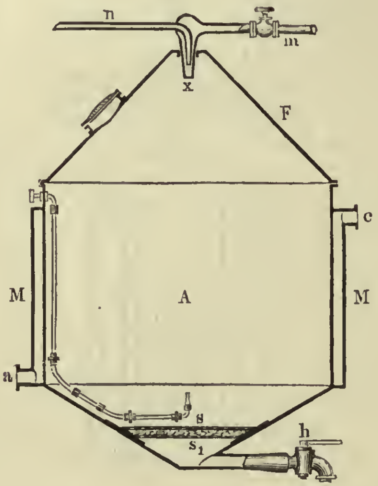

FIG. 168.

When all the starch is dissolved, the solution is transferred to a precipitating apparatus, which is illustrated in Fig. 168. It consists of a leaden tank (A), the lower part of which is funnel-shaped, surrounded by a cooling-jacket (M); a and c are the inlet and outlet, respectively, for the cooling-water. At the bottom of the apparatus there are two sieves (S and S_1), with a layer of gun-cotton between them; their object is to filter the acid before it is run through the tap (h). The funnel-shaped cover (F) is provided with observation windows, and an inlet pipe (x), through which the solution of nitric acid and starch is introduced by means of an "atomizer" in which m is the air-pipe and n the admission pipe for the solution. There is also an air-pipe passing down to the bottom of the vessel.

A mixture of nitric and sulphuric acids, as a rule waste acids from nitro-glycerin manufacture, containing about 70 per cent. sulphuric monohydrate, 10 per cent. of nitric monohydrate, and 20 per cent. of water, is introduced into the apparatus. For each 100 lbs. of starch solution 500 lbs. of waste acids are employed. The starch solution is introduced in the form of an extremely fine spray by means of the atomizer, and the nitro-starch is precipitated from it in the form of a fine flour by the waste acid. The precipitate is retained on the gun-cotton filter, whilst the acids are drawn off at the end of each operation.

The nitro-starch retained in the apparatus is freed from adhering acid in pots provided with sieve bottoms, by subjecting it to the action of a hydraulic press. The cake thus formed is strongly agitated in water, and washed until neutral. After this the nitro-starch is treated for twenty-four hours with a 5 per cent. solution of soda, made into a milky paste in incorporating-mills, centrifugaled, and finally soaked in a solution of aniline to give it stability.

The nitro-starch so made is said to be distinguished by a very high degree of solubility in nitro-glycerin and kindred liquids, to have a high specific gravity when compressed, and to mix readily with other forms of nitro-cellulose, when they are treated together in a pulping machine. It dissolves in nitro-glycerin even when cold, and forms, according to the proportions used, a mass which varies in hardness from that of glue to that of bees-wax.

Nitro-starch is very hygroscopic, insoluble in water and alcohol, but soluble in ether and similar solvents. Three varieties of nitro-starch exist, namely:—

Mono-nitro-starch	$C_{12}H_{19}(NO_2)O_{10}$
Di-nitro-starch	$C_{12}H_{18}(NO_2)_2O_{10}$
Tetra-nitro-starch	$C_{12}H_{16}(NO_2)_4O_{10}$

They are all formed simultaneously when starch is nitrated, in proportions depending on the strength of the acid used. The ignition point of nitro-starch is near to 175° C. (347° F.).

CHAPTER X

EXPLOSIVES DERIVED FROM SUGARS

THE various kinds of sugar known have almost the same chemical composition as cellulose, yet they differ widely from it in their physical properties. As with cellulose, nitric acid acts also upon sugar, and the products are no less explosive than the corresponding pyroxiline substance, but their practical importance is not sufficient to call for more than the following brief description:—

1. FULMINATING SUGAR. (*Nitro-cane-sugar, Nitro-saccharose.*)

The action of a mixture of nitric and sulphuric acids on cane-sugar ($C_{12}H_{22}O_{11}$) was first studied by Schönbein. He used a mixture of 2 parts of sulphuric acid and 1 part of strong nitric acid, which, when allowed to act upon cane-sugar at a temperature of 54° F., transformed it into a sticky substance. After washing with water and drying, this substance became solid and brittle. When slightly heated it softened and could be kneaded, whilst strong heating caused it to explode violently without leaving any residue. Since the product—the so-called nitro-cane-sugar (no doubt a nitric-ether)—dissolves in alcohol and also in ether, it was at one time suggested that ordinary black powder might be coated with such a solution and thus be made waterproof, and at the same time have its explosiveness increased. L. Thompson suggested the use of fulminating sugar for firework mixtures.

2. NITRO-LACTOSE.

Lactose, which has the same composition as cane-sugar, is also transformed by the action of nitric and sulphuric acids into a highly explosive body which crystallizes from a solution of alcohol or ether. Nothing definite is known of the composition of these two fulminating agents.

3. NITRO-MANNITE. (*Fulminating Mannite.*)

Another variety of sugar, mannite, which occurs very frequently in plants (and largely in the sap of the manna-ash), is similarly transformed by a mixture of nitric and sulphuric acids, and is only distinguished from the other kinds of sugar by its more simple composition. Stenhouse dissolved mannite in 4 parts of nitric acid without heating, and then added sulphuric acid until a precipitate ceased to form. Strecker took 4½ parts of nitric acid and 10½ parts of sulphuric acid for each part of mannite, the acids being added alternately in small quantities. Reinsch added mannite to a mixture of 1½ parts of sulphuric acid to 1 part of fuming nitric acid which was kept constantly agitated. In all cases, the product precipitated was well washed and then dried at 104° F.

The fulminating mannite so obtained is soluble in hot alcohol, or ether, and can be obtained in snow-white needle-shaped crystals. From its composition it may be considered as a nitric ether. Mannite is regarded as a hexatomic alcohol, and has therefore the formula $C_6H_8(OH)_6$. From all the reactions of its explosive substitution products, it is to be assumed that in it six hydroxyl radicles are replaced by the same number of nitro-radicles, so that fulminating mannite should have the formula $C_6H_8(NO_3)_6$.

Nitro-mannite is distinguished by its highly explosive properties. It can be detonated by a moderate blow, like fulminating mercury, but it is less sensitive to friction and heat than the latter. The use of fulminating mannite has, therefore, been suggested for charging detonators in place of fulminate of mercury, on account of its lower degree of sensitiveness. The reason why it has not been adopted is probably that when impure, and exposed to light, it easily decomposes.

CHAPTER XI

NITRO-GLYCERIN

Nitro-glycerin is a nitric ether of glycerin. Since glycerin is a tri-atomic alcohol, it is formed by the replacement of three atoms of hydrogen by three nitro-groups:—

$$C_3H_8O_3 + 3HNO_3 = C_3H_5(NO_2)_3O_3 + 3H_2O.$$

The name nitro-glycerin, or rather tri-nitro-glycerin, is not well chosen, since what is termed nitro-glycerin is not, strictly speaking, a nitro-compound. If it were, it would be transformed into an amido-compound, just as nitro-benzene, picric acid, etc., are, by reducing agents such as nascent hydrogen, ammonium sulphide, etc. But with these reducing agents glycerin is re-formed; and, further, since by the action of caustic alkalies the corresponding nitrates and glycerin are formed, nitro-glycerin must without doubt be considered as an ether of nitric acid. Its rational formula is therefore—

$$C_3H_5(O.NO_2)_3.$$

The proper name should consequently be—nitric glyceride. The use of the name nitro-glycerin has, however, become so firmly established by use, that it cannot now be discontinued in a technological work.

With nitro-glycerin, as with gun-cotton, a mono- and a di-nitro product can be conjectured, and it is probable that they are constantly formed in the waste acids, and at once dissolved in the large quantity of sulphuric acid present. They are also soluble in water, so that they cannot exist free either in the manufacture of nitro-glycerin or if formed from the waste acids. Besides these reasons for their non-occurrence, it is probable that they at once decompose in contact with moist air.

Liecke ('Dingler's Polytechnisches Journal,' 1869) has asserted that by using nitric acid of 1·3 specific gravity, mono-nitro-glycerin is formed, di-nitro-glycerin with an acid of 1·4 specific gravity, and tri-nitro-glycerin with an acid of 1·5 specific gravity, but his experiments have not so far been verified. Henriot ('Annales de Chimie et Physique,' cinquième série, 17,118) claims to have made mono-nitro-glycerin by mixing glycerin with dilute nitric acid (1 part of HNO_3 to 3 parts of H_2O). He states that it is easily soluble in water and alcohol, with difficulty in ether, and that it does not explode by shock.

The manufacture of nitro-glycerin is guided by the same principles as that of gun-cotton, which have been explained in a previous chapter. With nitro-glycerin, as with gun-cotton, sulphuric acid is necessary as an intermediate agent to constantly keep the concentration of the nitric acid up to the required degree—that is to say, to take up the water formed during the reaction. On the other hand, sulphuric acid also helps to precipitate the nitro-glycerin, since the latter is soluble both in concentrated sulphuric acid and in concentrated nitric acid, but not in a mixture of both. If too great an excess of one or the other be taken, part of the nitro-glycerin is dissolved and the yield diminished.

It is not possible to at once bring the whole of the quantity of glycerin to be nitrated into contact with the mixture of acids, as is done to a certain extent with gun-cotton, since the heat liberated by the reaction would cause too great an elevation of temperature. The addition of the glycerin must therefore be made gradually. Neither is it possible to run the mixture of acids into the glycerin, since the latter is too viscous to allow an intimate mixing to take place with sufficient rapidity. It is therefore preferable to put the whole of the acid mixture to be used into a vessel, and then let the glycerin slowly run into it. It is true that by these means the available nitric acid at the end of the process is too much dispersed through the mixture, and that the glycerin then running in is not at once attacked by the nitric acid, which is a disadvantage, but on account of the many other advantages this process possesses it is the best one to adopt.

(a) PREPARATION OF THE ACID MIXTURE.

The discoverer of nitro-glycerin, Ascanio Sobrero, and also Praeger and Bertram, and Liebe, allowed the glycerin to run drop by drop into the acid mixture, which was continually stirred. Sobrero used a cooled mixture of 2 parts of sulphuric acid (specific gravity 1·831) and 1 part of nitric acid (specific gravity 1·525), into which he allowed half a part, by volume, of glycerin to run, the mixture being stirred and prevented from heating.

Praeger and Bertram, and also Liebe, used the same acid mixture. De Vry at first used strong nitric acid alone, but subsequently he also used a mixture of nitric and sulphuric acids.

E. Kopp prepared the acid mixture by passing nitric acid vapours into sulphuric acid of 1·834 specific gravity contained in receivers.

G. M. Mowbray used the following method of preparing the acid mixture in his nitro-glycerin factory at North Adams in Massachusetts, U. S. A.:—300 lbs. of sodium nitrate and 375 lbs. of sulphuric acid were put into stills and the nitric acid vapours formed absorbed by sulphuric acid in earthenware receivers. In each of the first two receivers 150 lbs., in the third 100 lbs., in the fourth no sulphuric acid was contained. He thereby obtained about 600 lbs. of acid mixture direct, which was drawn off in carboys and poured into a trough made of soap-stone. In order to eliminate the nitrous acid and to thoroughly mix the acids, air was blown through the mixture from an iron tube for five minutes.

Later, when manufacturing on a larger scale, a mixture of 1 part of nitric acid and 2 parts of sulphuric acid was as a rule used. In course of time it was found that a saving could be effected in the quantity of nitric acid used, and at present the acid mixture generally consists of 37½ parts by weight of nitric acid containing from 93 to 95 per cent. monohydrate, and 62·5 parts by weight of sulphuric acid with 96 per cent. monohydrate—that is to say, 3 parts of nitric acid and 5 parts of sulphuric acid.

Both the nitric acid and the sulphuric acid must be of the highest possible concentration. If weak nitric acid be used, only a small quantity of tri-nitro-glycerin will be formed—or, at any rate, its formation will only take place very slowly and the other nitro products, which will nevertheless form, are soluble in sulphuric acid. Glycerin is similarly soluble, and the consequence of using weak nitric acid will be a violent disengagement of heat and rapid decomposition. The sulphuric acid must therefore be of the highest concentration, so that the water formed may be taken up quickly and with certainty. If it were of inferior strength, the formation of tri-nitro-glycerin would soon cease.

All that has been said of the mixture of nitric and sulphuric acids for making gun-cotton (page 8) also applies to that for nitro-glycerin. The vessels and apparatus there used, and the means of transporting the mixed acids are the same; but whilst a certain percentage of lead sulphate in the sulphuric acid has but little influence on the manufacture of gun-cotton, it is not desirable in the manufacture of nitro-glycerin. On mixing nitric acid and sulphuric acid the lead sulphate soon precipitates, and it also separates when the mixed acids are stored in lead vessels. If mixed acid containing lead sulphate be used, the separation of nitro-glycerin from the waste acid takes place much more slowly. It is advisable, therefore, to draw off the mixed acid, not from the bottom of the storage vessels, but from a point some distance above it.

(*b*) PROPORTIONS OF MIXTURE.

According to the chemical equation expressing the formation, 227 parts of nitro-glycerin should be obtained from 92 parts of glycerin and 189 parts of nitric acid. Therefore, to produce 246·74 parts of nitro-glycerin, 100 parts of glycerin and 205·43 parts of nitric acid are required, or, for 100 parts of nitro-glycerin, 40·53 parts of glycerin and 83·26 parts of nitric acid.

In practice, it is necessary to take a higher proportion of nitric acid, since it is not possible to bring the glycerin immediately in contact with it in the large volume of acid mixture that is used, and the less so in proportion as more nitro-glycerin has been formed. Sobrero used half a volume of glycerin to one volume of nitric acid and two volumes of sulphuric acid. Praeger and Bertram took 1 part of glycerin to 8 parts of an acid mixture of the same proportions. Liebe used only 6 times the quantity of acid; De Vry, 6 to 7 times the quantity; Kopp, 8 times; Mowbray, 8½ times; and Champion and Pellet, and also Von Förster at Opladen, 6 times the quantity of acid mixture.

At present a mixture of 3 parts of nitric and 5 parts of sulphuric acid is almost universally used, and the quantity of glycerin is varied according to the strength of the acids and the quality of the glycerin. In America, and in some continental factories, 1 part of glycerin is used to 8 parts of the above acid mixture, but up to 1·15 parts is frequently taken. The following table gives a comparison of the quantities of acid mixture used for each 100 parts of glycerin :—

	NITRIC ACID.	SULPHURIC ACID.
Sobrero	238	584
Praeger and Bertram	266	533
Liebe	238	584
De Vry	300	370
Kopp	266	533
Mowbray	383	567
Champion and Pellet	260	520
In the Saverne quarries	220	440
St. Lambrecht dynamite factory, formerly	250	420
Modern dynamite works, from	270 to 300	450 to 500

Now-a-days, not only the question of the yield, but also the question of general economy determine the relative quantities of glycerin and acids. In some cases it may be convenient to obtain a smaller yield of nitro-glycerin from unit weight of glycerin, and in others the cost of manufacture may be cheaper, if larger quantities of nitric acid be used and the yield of nitro-glycerin thereby increased. The best practice to adopt is discussed in a subsequent part of this work; it will chiefly depend upon the prices of glycerin and nitric acid, for which reasons the proportion between glycerin and nitric acid is frequently varied.

(*c*) NITRATION.

The early apparatus for nitrating glycerin were arranged for the production of only small quantities, and were therefore very primitive.

Kopp devised his process with a view to simplicity, so that nitro-glycerin could be prepared in the quarries themselves and used either immediately or soon afterwards. Nitro-glycerin was made by his process for many years in the sandstone quarries near Saverne in Alsace. The apparatus used by him are illustrated in Fig. 169. They were a large earthenware vessel (a) for cooling and washing the nitro-glycerin, a cast-iron vessel (b) for mixing, with an index mark on the outside and the inside, a measuring vessel (c) of sheet-iron or china for glycerin, a glass funnel (d) with india-rubber tube, pinch-cock, and outlet-pipe, and finally an iron rod with a mark on it, for stirring. Five to six litres of ice-cold water were put into the vessel (a) in which the vessel (b) containing 2·8 kilogrammes of mixed acid stood. Into this 350 grammes of glycerin were run from c, the contents of b being stirred the meanwhile. After all the glycerin had been run in, the stirring was continued for five minutes with the iron rod, and the crude nitroglycerin was then washed in earthenware vessels (a). It was then decanted and all traces of water removed by aid of the separating funnel (d).

By Kopp's process, three to four operations per hour are said to have been made, and the yield to have varied between 143 and 200 per cent. Of course the nitro-glycerin was not suitable for prolonged storage, since it could not of necessity be pure.

FIG. 169.

Nitro-glycerin was manufactured in a similar manner in continental factories down to as late as 1880. China pots, and also enamelled or ordinary cast-iron vessels or lead vessels with burnt joints, were used. As a rule, a number of such pots stood in a trough filled with water, over which a corresponding number of small glycerin vessels fitted with taps were placed. The workman held a thermometer in one hand and in the other one a glass rod, with which he rapidly agitated the mixture. If, as frequently happened, the temperature rose too high, and a decomposition took place, he simply tipped the contents of the pot quickly into the water. By and by the workman became very expert in this method of regulating the process, and with such simple apparatus yields of up to 200 lbs. of nitro-glycerin for each 100 lbs. of glycerin were obtained. Later, the stirring with the glass rod by hand, which required a large number of workmen, was replaced by a mechanical stirring arrangement. Such an apparatus, as it existed in the Rheinische Dynamitfabrik of Opladen, is illustrated in Fig. 170. In it also a number of cast-iron pots (a) stood in a trough (b) filled with water, and the stirring was done by one single arrangement for a whole series of pots. The stirring arrangement consisted of a long wooden bar (c) extending over all the pots and carrying over each a disc (e), to which were fixed six wrought-iron rods (d). Each end of c was attached to a crank-plate (f), consisting simply of a round wrought-iron disc with two iron pins one above and the other below. The lower pins turned in holes in the end of the trough frame, the upper ones in holes in the wooden bar. As the crank-plates rotated, it is clear that the bar gave a circular motion to the stirring bars, which dipped into the pots, and a thorough agitation of the mixture was consequently effected.

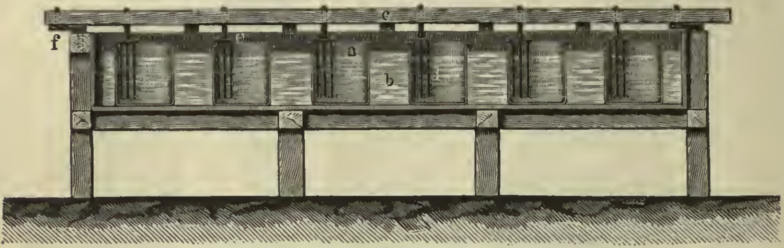

Fig. 170.—One-40th full size.

A further advance in mechanical stirring is to be found in the apparatus illustrated in Figs. 171 and 172, which was used at the dynamite factory of St. Lambrecht. Here also a number of cast-iron or leaden pots (a) were placed in a long trough (b) filled with cold water, and into each pot a stirrer (c) dipped, consisting of two sheet-iron paddles fixed on to a common axis and bent in the shape of a screw. The stirring-paddles were carried in bearings on a wooden frame-work, and geared at their upper end with the main shaft (e) by cog-wheels (d). The glycerin vessels were placed along the side of each apparatus. From the bottom of the pots a discharge-pipe (g) passed through the trough to a main gutter (h), which carried the mixture either to the washing apparatus or into a safety-tank. This apparatus also had the disadvantage of requiring constant watching of the thermometers in a large number of vessels, and consequently several persons to attend to it. Direct separation had not at this time been introduced.

In America, where, on account of the great consumption, considerable quantities had to be produced per day, larger nitrating apparatus were very soon used, in which the stirring was done either by large paddle-wheels or by agitators moved to and fro on a bar.

A nitrating apparatus, erected by Fred. Nicholas Engels in Australia, Kalk, and Matagne la Grande, which was used for a long time, is illustrated in Fig. 173 in longitudinal section, and in Fig. 174 in cross section. It consisted of a cast-iron tank (A) in which a cast-iron trough (B) was so placed that its rim rested tightly on the tank (A). The discharge branch (C) was carried through the latter, the joint being made tight with putty, and as a rule closed with an india-rubber plug. The trough rested on two brick pillars (D) with a recess for reaching the discharge branch (E). The whole was surrounded by a large wooden flue (F) 33 feet high, which carried away the gases. On the outside of the casing were two wooden guide-blocks (F_1) carrying a wrought-iron bar (G), which could be pushed to and fro by means of a handle on its end. On this bar two slotted cast-iron plates (H) were fixed, which, as the bar was moved to and fro, stirred the

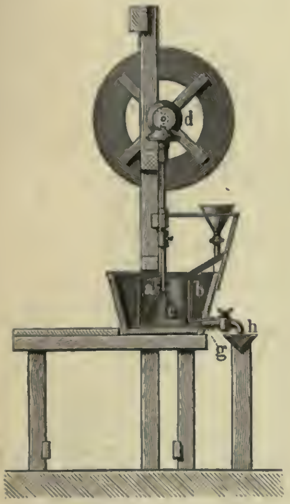

Fig. 171.—One-60th full size.

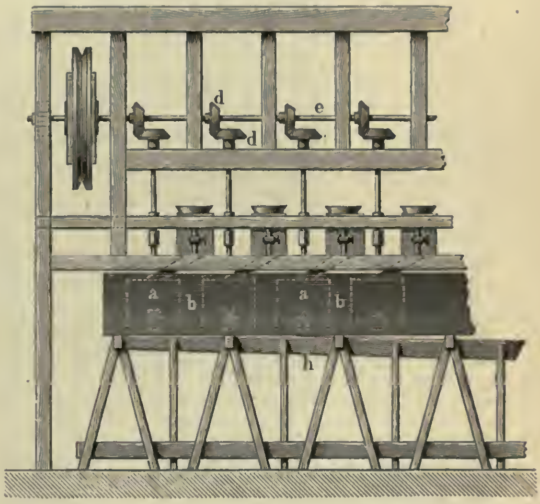

Fig. 172.—One-60th full size.

mixture. Cooling-water entered into the tank (A) through the tube I, and flowed off through the tube J. The glycerin was in a vessel (K) placed outside the flue, whence it ran in a thin stream into the vessel (B) by means of two pipes (L) provided with taps.

The apparatus was used for a charge of 10 kilogrammes of glycerin which could be run in and converted within an hour. For this quantity, 28 kilogrammes of nitric acid and 56 kilogrammes of sulphuric acid were used. A workman put the stirrer in motion and gave it 25 double strokes per minute. When the nitration was finished, the india-rubber plug was withdrawn, and the mixture run into an oval tub (L) made of oak, with an inclined bottom and filled to three-quarters of its height with water. The water was discharged and changed by means of the decanting taps (M), and the nitro-glycerin drawn off by an india-rubber tube fixed on the socket (N).

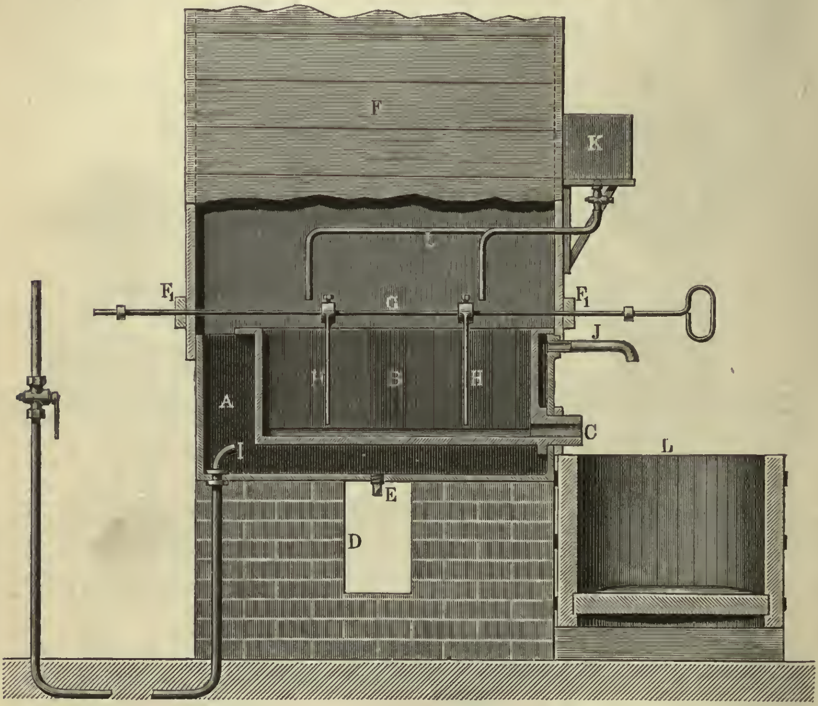

FIG. 173.—One-20th full size.

Engels made very considerable quantities of nitro-glycerin by means of this apparatus, the yield being on an average 19·5 kilogrammes from each 10 kilogrammes of glycerin.

Ferdinand Capitaine described in 'Dingler's Polytechnisches Journal' of 1872 a continental factory which turned out as much as 5000 lbs. of dynamite daily. The nitrating apparatus used in this factory consisted of a wooden tub 6½ feet high and 4 feet diameter, lined with ¼ inch lead. In it were placed two worms of stout lead pipe of 1¼ inch bore which extended from the bottom to the upper edge, and each of which was fed separately with cold water. The apparatus was fitted with a stirring arrangement consisting of two round perforated, lead-lined iron plates, of 2 feet diameter, fixed horizontally on a vertical iron bar, at a small

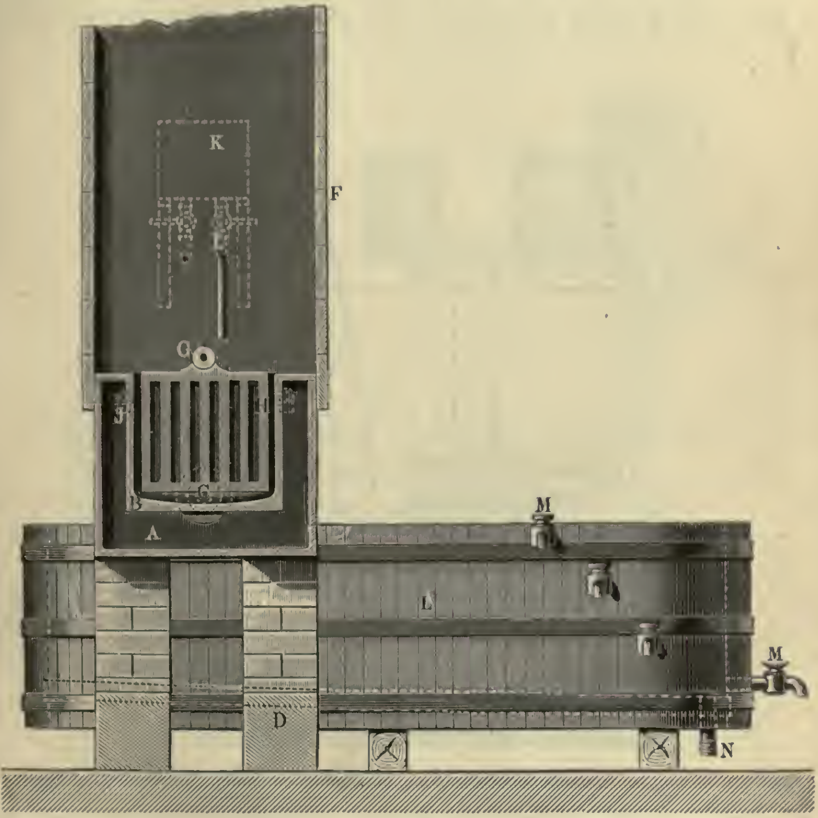

FIG. 174.—One-20th full size.

distance from each other. The iron bar was connected on its upper end with a rope passing over pulleys at a distance of about 30 or 40 feet from the apparatus, where, behind a strong mound, a workman, by pulling the rope up and down, actuated the stirrer. The apparatus was closed by a lead cover, through which a thermometer passed to the interior of the tub. The temperature was maintained at between 14° and 16° C. On the cover there was also a small sheet-iron box, into which the glycerin was fed from a reservoir on the roof of the building. The bottom of this iron box was perforated with a number of holes closed by means of corks fixed on little bars. In this way the glycerin was made to run in fine streams. The time of nitration depended on the temperature of the cooling-water, and varied from one-half to 2½ hours.

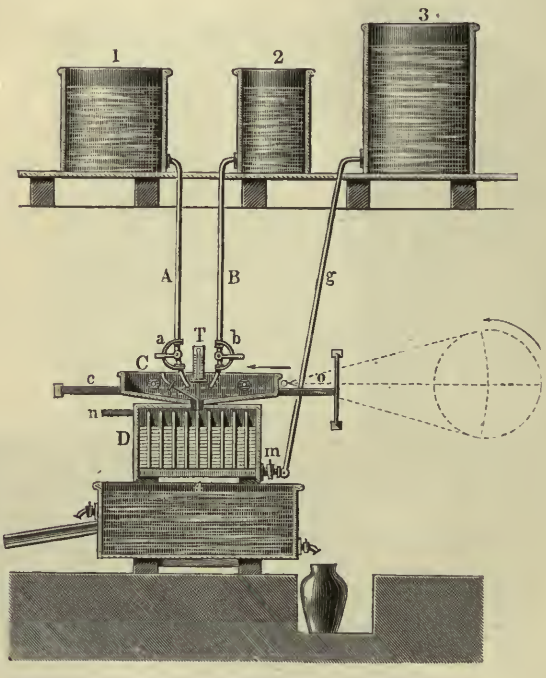

Fig. 175.

Rudberg of Stockholm used the apparatus illustrated in cross section in Fig. 175, and in longitudinal section in Fig. 176. The acid mixture, which was prepared in a lead vessel, was drawn off when quite cool into the vessel 1. Vessel 2 held the weighed glycerin, and vessel 3 was filled with cold water. All these vessels had discharge-pipes (A, B, g), to which taps (a, b, m) were fitted. The vessels 1 and 2 communicated with a box (C), which was hung over the channel (D) either like a pendulum, or resting on wheels, so that a continuous to-and-fro movement could be given to it by means of a handle and crank, as indicated by the dotted lines in Fig. 175. The lead-lined channel (D) was divided into stepped compartments, and the whole surrounded by a wooden trough. In this box (C) a thermometer (T) was placed, from which the temperature set up by the reaction could be read off. The water issuing from the vessel 3 filled the interstices between the channel (D) and the wooden trough, and ran away through the tube (n).

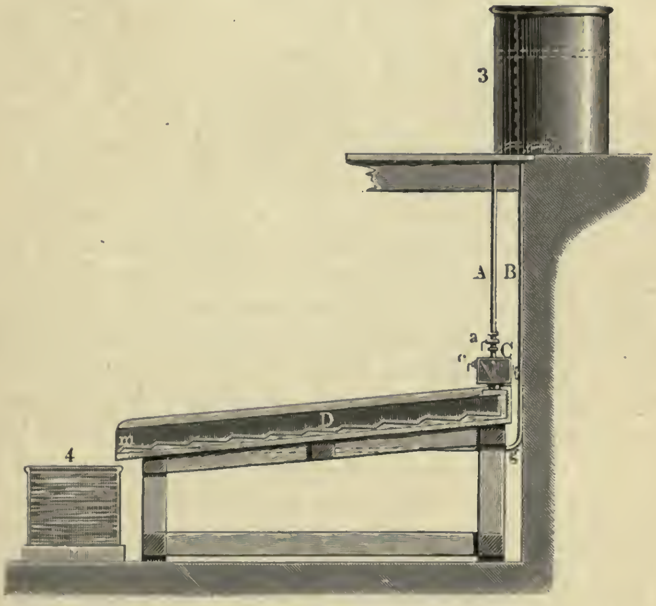

Fig. 176.

On making nitro-glycerin the cooling-water was first allowed to surround the channel (D), the acid mixture was then run into the funnel of the vessel (C), which was oscillating, so that the mixture ran into the various compartments of the channel (D). As soon as the acid began to run out from all these compartments, the glycerin was allowed to run out through the tap (b). The reaction was finished when the liquid had passed the channel (D). The mixture obtained was then allowed to run out into the vessel 4, which was filled with water, and the nitro-glycerin which settled down was then carefully washed. The temperature was always kept below 30° C.

G. M. Mowbray, at North Adams, Massachusetts, in 1868, seems to have been the first to use compressed air for stirring the nitrating mixture. In his nitrating-room there were 9 wooden troughs, in which 116 earthenware pots, each holding 17 lbs. of acid mixture, were placed, either in ice-cold water or in a mixture of ice and salt. The glycerin was allowed to run in drop by drop from a glass vessel through a syphon and rubber pipe. Whilst the glycerin was running in, cold compressed air was blown in through glass tubes 16 inches long and ¼ inch diameter, which were connected with an iron air main.

At this time Professor Barker had already observed that a current of air thoroughly mixed the materials, partly eliminated the nitrous vapours, and cooled the mixture itself, and also that it transformed any nitrous acid, which was either already present, or which had formed during the reaction, into nitric acid, and thus allowed the production of chemically pure nitro-glycerin. The time of nitration with Mowbray's process was 1½ hours.

The next step towards the apparatus now exclusively used with compressed air was the employment of those nitrating apparatus formerly used in several factories, which had both a mechanical stirring arrangement and a compressed air-pipe. In these apparatus the cooling-water was ingeniously utilized to drive the motor for the stirring arrangement as well as to cool the mixture. They consisted of large lead vessels standing in a wooden vat, and were closed with a conical cover. Inside these vessels, according to their sizes, were two or three independent lead cooling-worms. In the centre an agitating helix, actuated by a pair of cog-wheels, was hung. The helix was made of cast-iron, carefully covered with lead by soldering. On the shaft of the agitating helix, just inside the apparatus, a disc was fixed, on to which the glycerin ran, so that as the shaft revolved the glycerin was thrown in fine drops on to the surface of the mixture by centrifugal force. Thermometers passing through the cover served to control the temperature of the acid, and a fume-pipe on the cover to carry away the gases formed. One or more pipes for the admission of compressed air were provided, passing down to the bottom of the apparatus. The apparatus was placed at a sufficiently high level to permit the contents to be run off into the next apparatus in the process. As a rule, the plant was arranged on the side of a hill in such a way that the inlet of the glycerin was at one level, the discharge of the nitrating mixture at a second lower one, while the safety-tank was placed at a third still lower level. On the lowest level was placed a water-wheel of considerable diameter (about 17 feet), having thin wrought-iron spokes and narrow sheet-zinc buckets. The power was taken off from a pulley on the water-wheel axle, and transmitted to a second pulley on the shaft actuating the stirring helix in the nitrating apparatus. In case of necessity the agitating apparatus could be worked by means of a crank handle and fly-wheel by two workmen.

The water entered the cooling-worms under high pressure, and left them with only a slightly lower one. It was then carried through an open channel, or a pipe, direct to the water-wheel, so that the agitating helix revolved without interruption so long as the cooling-water flowed through the apparatus—that is to say, during the whole time of the operation. The agitating screw revolved in the direction necessary for the glycerin dropping on to the surface of the acid mixture to be at once drawn downwards, whilst, on the other hand, the compressed air forced it upwards. The combined effect of the two was a very violent mixing in the apparatus, which was very advantageous for nitration.

The apparatus almost exclusively used at the present time for the manufacture of nitro-glycerin is similar to that just described, except that there is no mechanical agitator. It is illustrated in Fig. 177, and consists of a lead vessel (A), standing in a wooden vat (B); with a number of air inlet-pipes (C), cooling-worms (D), thermometers (E), fume-pipe (F), and inlet-pipe for the acids (G) and for the glycerin (H), as described above. The cover (I) can be taken off, the joint being as a rule made with cement or glazier's putty. The connection of the cooling-worms, and generally of all the pipes entering the apparatus, is made so that they can be detached at will, in order that the apparatus may be readily taken to pieces should repairs be necessary. A sight glass (J) is fitted between the fume-pipe and the cover to enable the colour of the gases given off to be observed. The cooling-water runs constantly through both the cooling-worms and the space between the inner lead vessel and the outer wooden one. The bottom of the apparatus is inclined, and at its lowest point there is a socket, in which is fixed an earthenware tap (K) of about 2 inches bore.

On to the cover of the apparatus a number of glass panes (L) are inserted, so that the reaction going on in the interior of the apparatus can be watched. The discharge-tap can as a rule be put in connection with at least two gutters at will. As a rule, it is in connection with the one leading to a safety-tank, so that the

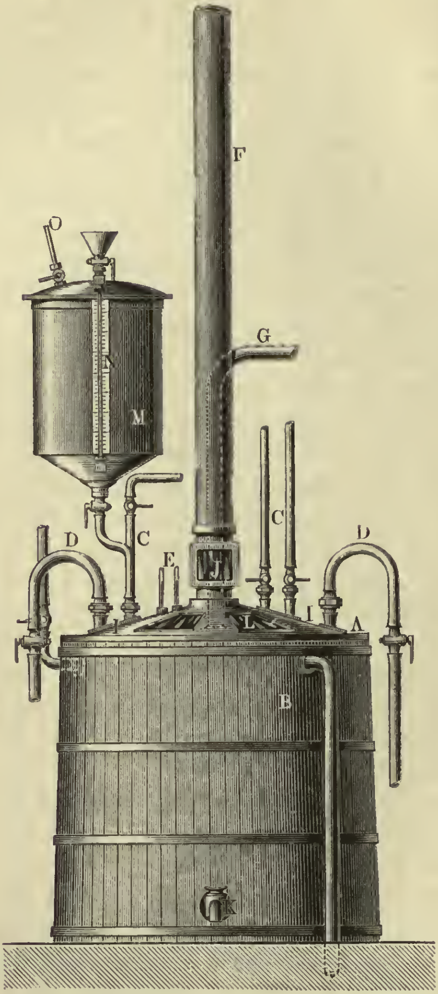

Fig. 177.—One-30th full size.

whole contents of the apparatus can in case of danger be immediately discharged into the safety-tank. When the nitration is finished, the discharge-tap is connected with a gutter leading to the separating apparatus. Frequently a third gutter is also provided, which is used for cleaning the apparatus, the washing-water being run down it to a special separating vessel, where it is freed from any traces of nitro-glycerin it may contain.

The acid mixture is run into the apparatus from the storage vessels before referred to, either in weighed or measured quantities. If the acid be freshly prepared, its temperature will sometimes be between 86° and 104° F., and it is therefore necessary to cool it before the nitration is started. The simplest way of doing this is to admit compressed air.

The introduction of glycerin is effected in various ways. The method adopted in apparatus with screw agitators has already been described. Glycerin can be introduced in a simple manner by a tube on the cover bent to a circle and perforated with a number of fine holes, so that the glycerin drops on to the surface of the mixture in a number of thin streams, the inlet being regulated by a tap. With larger apparatus an injector worked by compressed air may be used with advantage. It is fixed near to the bottom of the vessel, and the inlet-pipes for the glycerin and air are carried through the cover of the apparatus. The injector can be made of china or earthenware, or of metal if carefully lined with lead; such a lining, however, is always liable to be eaten away, and the burnt joints where the pipes enter the apparatus are also a source of trouble. Injectors have the further disadvantage that the expansion of the compressed air, as it is doing work, produces cold, which makes the glycerin viscous, and therefore brings it into the apparatus in a less finely-divided state than is desirable. On the other hand, the injector has the great advantages of introducing the glycerin into the acid mixture from below, thereby materially assisting its rapid transformation into nitro-glycerin and preventing decomposition by local heating, such as might be the case if it were merely dropped on to the surface of the acid mixture.

The most practical way of introducing glycerin is certainly by pressure vessels, as is the case with the apparatus shown in Fig. 177. This consists of a wrought-iron cylinder (M) sufficiently strong to withstand the air pressure used, and fitted with a gauge-glass and scale (N). Compressed air is admitted by the pipe (O) and acts on the upper surface of the glycerin, forcing it out of the vessel by a pipe passing through the cover of the nitrating apparatus. This pipe terminates in a single-turn flat coil which rests on the bottom of the nitrating apparatus, and has in it a number of fine perforations.

The glycerin should not enter the apparatus at too low a temperature, as it would then be too thick to divide up well. On the other hand, it must not be too warm, because the temperature of the mixture would then rise rapidly. A temperature of from 68° to 77° F. has been found to be best. In order to ensure this, it is necessary to store the glycerin in warm rooms in winter, since, as has been stated in the chapter on glycerin, it freezes easily in winter, and is only thawed with difficulty. Moreover, when pouring the glycerin into drums, for transport, it is impossible to avoid impurities, such as pieces of wood and similar foreign matter, which find their way into the drums, and it is therefore advisable to strain the glycerin as it is run out of the drums. The pipes conveying the glycerin to the nitrating-house should be protected from cold, if the room in which the glycerin is warmed is away from the nitrating-house.

If the temperature of the glycerin is kept fairly the same all the year round, it is not necessary to weigh it for each charge, as sufficient accuracy is obtained in running it into the nitrating apparatus of known contents, or from one filled with a gauge-glass and index. In the latter case, the smaller the diameter and the greater the height of the measuring vessel, the greater will be the accuracy of the measuring.

Nitration is started when the acid mixture has dropped to a temperature of about 60° F., the admission of the glycerin being regulated so as to prevent the temperature from rising above 77° F. In nearly all factories the rule is that the temperature shall not under any circumstances rise above 86° F. This is necessary because at 122° F., decomposition may readily take place, and if incipient decomposition should occur at such a high temperature, it is hardly possible to stop it. In the large quantity of acid a local heating up to 122° F., or still higher, would not be immediately indicated by the thermometers dipping into the liquid, yet it is possible that local heating centres may occur which will spread in the liquid. The occurrence of a general temperature of above 86° F. will therefore probably mean that there is at some spot a local one of over 86° F., and it is therefore imperative that steps be taken in time to prevent a dangerous rise of the temperature of the whole mass. To do so, the inflow of glycerin is either diminished or shut off altogether. If the temperature, nevertheless, continue to rise, all air-taps must be opened, when the cold produced by the expansion of the compressed air, together with the violent agitation it gives rise to, is as a rule sufficient to distribute the local heat throughout the whole mass of liquid and thus cool it down. If, in spite of the effect of all means of cooling, the decomposition still continue, the discharge-tap at the bottom of the nitrating apparatus leading to the safety-tank must be at once fully opened, and the worker must immediately after leave the building.

Since at the present time only perfectly pure glycerin is used, and the process of nitration is always carefully watched, explosions in nitrating apparatus are extremely rare, and can only occur through flagrant carelessness or from the use of bad materials. The cooling-water supplied to the nitrating apparatus should be of such temperature that, together with the air agitation, it will keep the contents of the apparatus within the limits of temperature mentioned above. It is only rarely that the cooling-water has a temperature of over 68° F.; and where this or a lower temperature is unattainable, water of a higher temperature can still be used by working more slowly, or by cooling the water artificially with ice, or the nitration may be done in the early hours of the morning when the air temperature is low. A sufficient store of cooling-water has to be provided, and it is therefore not advisable to connect the cooling-worms directly with the water main, but to have a special water-tank for the nitrating apparatus placed near them. The compressed air used is, as a rule, at about 60 lbs. pressure, since at least this pressure is generally required for lifting up the acids to the store-tanks by means of acid elevators. It is not necessary to admit the air at such a high pressure into the nitrating apparatus. Mr. E. J. Barbier, in a private communication to the author, has even observed that a high air pressure influences the yield of nitroglycerin. Although this cannot be easily accounted for, yet it may be that part of the nitric acid is driven off as vapour by the air rushing in with such great force, assisted by the slightly higher temperature set up during the reaction, and the yield thus reduced. On the other hand, a certain pressure is required to overcome the head of acid and to violently agitate it. It may, therefore, be assumed that with a depth of acid of 4 feet an air pressure of about 7 lbs. per square inch will be required to just bring the acids into ebullition. If a pressure-reducing valve is used, it is not advisable to reduce the pressure below 30 lbs., but in most cases it will be sufficient to have a sufficiently large air main and to only partially open the tap, when, due to the air expanding into the apparatus after it has passed the partially opened tap, its pressure will be reduced. This method has the advantage that in case more air or a more violent agitation be required, it can easily be attained by fully opening the tap.

It must be noted that in constructing a nitrating apparatus care has to be taken to prevent the possibility of foreign matter, especially any of an organic nature, from falling into it. The cooling-worms and the inner lead vessel must be examined every day to see that no holes, not even the finest, through which water could enter into the acid mixture, have been eaten into them by the acids. Organic matter or water, especially the latter, grease, and similar substances may originate violent decomposition of the whole mixture, and most of the explosions which occurred when large nitrating apparatus began to be used were caused by these means, or by the use of bad glycerin.

The material from which the nitrating apparatus, and in general all apparatus used in the manufacture of nitro-glycerin, are made, is exclusively lead, because the sulphate of lead formed by the action of the acids does not injuriously affect the process of nitration. In time, lead sulphate will be deposited on the bottom and the sides of the apparatus, on the cooling-worms, and especially on the cover in large flakes, which will gradually crack off and get into the nitro-glycerin. Although the lead sulphate, as long as it adheres to the cooling-worms, etc., protects them to a certain extent against the destructive influence of the acids, yet by its continual cracking off the pipes are gradually worn away, and it is therefore necessary to carefully examine the apparatus at intervals. It is even a good plan to replace them altogether by new ones at stated periods, whether the pipes appear to be still serviceable for some further time or not. The expense of this is more than compensated for by the increase of safety it ensures.

A special form of nitrating apparatus used in the Boutmy-Faucher process will be described further on, when that process is dealt with. The time of nitration depends upon the quantity of glycerin used for each charge, on the temperature of the cooling water, and on that of the atmosphere. With well-constructed apparatus an operation can be finished in from twenty-five to thirty-five minutes, and the whole duration of an operation, including introduction of the acids, injection of glycerin, and discharging the apparatus, is about one hour.

During the process of nitration copious acid fumes are developed. They are produced partly by the heating of the mixture and partly by the action of the compressed air, which drives off part of the nitric acid, and finally by the local decomposition of small particles which always occurs during the reaction, and which results in the development of hyponitric acid.

These fumes consist chiefly of hyponitric acid, and are as a rule carried into the open air through a wide vertical tube passing up some distance above the roof. In order to prevent foreign matters from falling down it into the apparatus, the tube is provided with a rain-proof hood. As these gases are heavier than air, they have on cloudy days a tendency to sink to the ground, and in general they will be driven to the mounds surrounding the building according to the direction of the wind, and will damage the vegetation and the roofing of the building. It is nevertheless an interesting fact, that in some cases it has been shown that these acid vapours, on moving through the air, are transformed by the moisture of the air into nitric acid again, and on acting upon a calcareous soil have produced a natural manure, favourably influencing the vegetation. This has been conclusively confirmed by the author's own experience; for at a factory where the prevailing wind was from the west all the mounds facing east, inside the factory, had a much thicker crop of grass than the others. This influence even extended as far as the neighbouring fields. Nevertheless, in many cases it will be necessary to prevent the escape of these fumes into the air, and the best means is probably a Lunge-Rohrmann plate tower, which, while its absorbing power is good, does not obstruct the draught, as would be done by a coke-tower or a box filled with coke, thus possibly hindering the nitrating operation. No saving must be expected from the absorption of the gas, because in order to render it harmless a considerable quantity of water is required, and the resulting acid will therefore be extremely dilute.

(*d*) SEPARATION.

After the glycerin is nitrated, it is necessary to separate the nitro-glycerin formed from the waste acids mixed with it. There are two ways of doing this—namely, the indirect and the direct separation. The former has now practically gone out of use.

(1) *The Indirect Separation.*

The simplest way of separating nitro-glycerin from the acids which have produced it is to run the whole lot into a large quantity of water. The acids become so dilute that the nitro-glycerin, which is insoluble in water, is at once precipitated on account of its higher specific gravity, and can be separated by simply decanting off the water. This method was used in all the earlier manufactories. As a rule, large lead-lined wooden vats, as illustrated in Fig. 174, which were sometimes as much as 14 feet in diameter and 8 feet in height, were used. They were about half filled with water, and the mixture of acid and nitro-glycerin was run in in a slow stream, the contents of the vat being at the same time agitated either by hand with wooden paddles or by a number of perforated wooden plates fixed vertically on a bar which was rocked to and fro. The bottom of the tank was inclined, and at its lowest point it had an earthenware tap for drawing off the precipitated nitro-glycerin. On running the mixture into the water a large amount of heat was developed—and the rate at which the acid and nitro-glycerin were run in had to be controlled according to the reading of thermometers, in order to prevent decomposition taking place in this tank. Sometimes special vats were not used, but the safety-tank was constructed so that it could be used as a separating tank.

Of course, with the indirect separation, the whole of the waste acids is lost, as they are diluted with so large a quantity of water. Besides this, a large quantity of nitrous acid vapours, which are a nuisance to the workmen and the neighbourhood, are formed on drowning such large quantities of acid in water. To avoid this, and also to allow the nitric and sulphuric acids contained in the waste acids to be utilized, the indirect method of separation has been abandoned.

(2) *The Direct Separation.*

In the early days of manufacturing nitro-glycerin on a large scale, attempts were made to effect separation by skimming off the nitro-glycerin floating on the top of the acids with wooden scoops, but it was soon abandoned, as several accidents were caused by it. Much later, about the year 1877, when the process of nitration had become better understood, the direct separation was again taken up.

The mixture of acids and nitro-glycerin coming from the nitrating apparatus soon separates. According to whether the nitration has been more or less perfect, the waste acids contain a less or greater quantity of nitric acid monohydrate, and the waste acids have consequently a less or greater specific gravity. With waste acids of normal composition the specific gravity is 1·70, that of pure nitro-glycerin being 1·60, so that a very long time should not be required to effect a complete separation of the two. Separation is partly prevented by impurities, such as fatty acids, slimy and other particles in the glycerin, by lead sulphate and other materials in the acid mixture, and also by various impurities which, if suspended in the nitrating mixture, adhere to the oily nitro-glycerin and by surface tension prevent separation taking place properly, and thus sometimes cause sensible delay.

In one case that came under the author's notice, the sulphuric acid had been stored in a wrought-iron tank which had originally served as an air-vessel, and had been tarred inside. Through a layer of rust forming over the tar the presence of the latter had been overlooked. The sulphuric acid became coloured black by dissolving off fine particles of tar, and the nitro-glycerin made with it was not completely separated even after two days' treatment in the separator. It will be seen later, in connection with the Boutmy-Faucher process, how strong an influence such finely-divided particles of foreign matter have in that process. As has been already mentioned, the quality of the glycerin delays separation, and therefore care has to be taken when it is selected to see if it be capable of separating within a certain time.

The simplest form of separating apparatus was probably the separating funnel, and lead vessels with a conical or inclined bottom and an earthenware tap at the lowest point were originally used. In these the mixture was allowed to stand for a certain time, and the waste acids were then drawn off and the nitro-glycerin after them.

At present a separating apparatus similar to the one illustrated in Fig. 178 is almost exclusively used. It consists of a square vessel made of stout sheet-lead with a pyramidal pointed bottom supported by a strong wooden frame (A). The apparatus is closed either by a cover of glass panes fixed in a cast-iron or lead framework, or with one of sheet-lead supported by lead straps (C), from cross-bars (B), and fitted with one or two panes of glass let into the cover, in order that the process going on in the interior may be watched. A tube (D) leading into the open air from the cover serving for carrying off the gases developed, has proved to be of advantage; of course it must be covered by a hood at its upper end. An inspection window (E) between the cover and the pipe (D) is provided, so that if any nitrous vapours are formed they can be seen. A thermometer passes through the cover into the liquid, and also a tube or a channel for introducing the mixture coming from the nitrating apparatus. At the lowest point of the bottom another inspection window (F) is fixed. This is either a round glass tube or a lantern-shaped prolongation, made of lead, fitted with two panes of glass. This terminates in a wide tube (G), on which two or three taps (H) are fixed. One of them is connected with the gutter leading to the second separation, a second one stands free in the separating-house, but can also be connected with a gutter leading to the waste-water-house, and a third one is in connection with the gutter leading to the safety-tank. Instead of having three separate taps, one only, arranged so that it can be connected by means

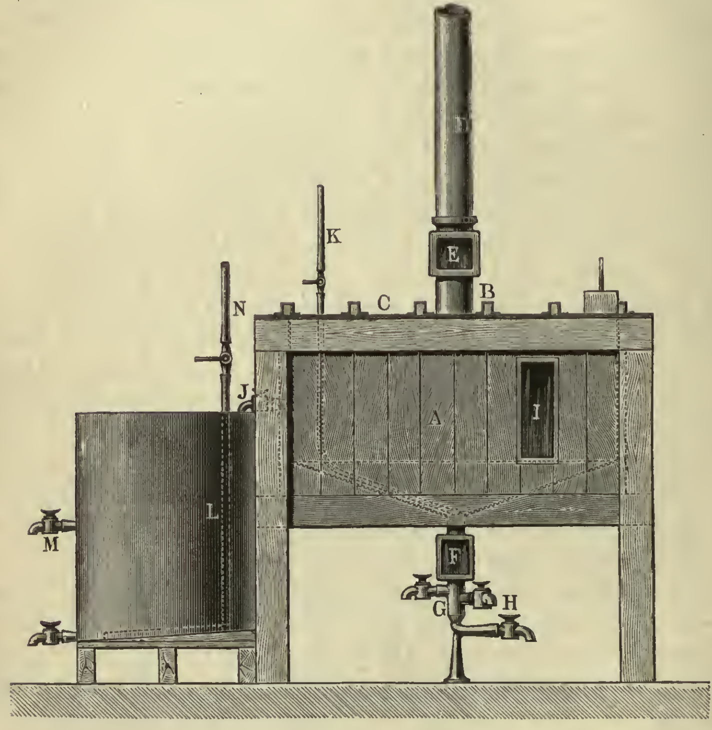

FIG. 178.—One-30th full size.

of a movable channel with any of the various gutters at will, may be used; but on account of the large quantity of acid vapours which even small traces of waste acids give off, it is preferable to have the taps permanently connected with their respective gutters, as this will allow of their being fitted perfectly tight. On one side of the apparatus an oblong hole (I) is cut out, and a lead frame soldered round it, into which a pane of glass is fixed with putty. This allows the upper layers of the liquid to be observed so as to ascertain when the separation is finished. Since the charges treated in these apparatus are always of the same volume, the height of the line of separation of the nitro-glycerin and waste acids is known beforehand, when the nitration has been normal, and a small tap (J) is fixed on the front of the apparatus at about this height, through which the nitro-glycerin can be drawn off immediately, when the separation is about finished. The advantage of this is that if excessive heating of the mixture occur, a minimum of nitro-glycerin is present, and that any remaining particles of nitro-glycerin can rise more easily to the surface. A saving of time is also effected by working thus, since whilst the bulk of the nitro-glycerin is being washed, any such small particles of nitro-glycerin have sufficient time to separate out. Sometimes a perforated lead pipe (K) is fitted, which just reaches to the lowest point of the separating apparatus. This is for blowing in compressed air to agitate the contents of the separator in case excessive heating occur.

When good glycerin and highly-concentrated acids have been used, the separation should be completed in about half-an-hour. At first the nitro-glycerin rises in large masses, then tree-shaped formations appear on the line of separation. These with glycerin of an inferior quality sometimes remain for a long time extending downward from the nitro-glycerin into the layer of acid; but with a good glycerin they should disappear quickly and leave a perfectly sharp line of separation between the milky turbid acid mixture and the more clear nitro-glycerin.

As soon as the separation appears finished, the nitro-glycerin is run into washing vessels (L) standing in front of the separating apparatus. When nearly all the nitro-glycerin has been drawn off in this manner, the waste acid is allowed to run out through one of the taps at the bottom of the "lantern" to the after-separation house, until a dirty, turbid mixture of lower nitro-compounds, impurities, etc., appears in the lantern. At this moment the tap is closed, and the remaining portion drawn off through a lower tap into suitable buckets and put into the washing vessel with the rest.

In the separating apparatus, not only are the nitro-glycerin and the waste acids separated, but also the mechanical and chemical impurities. The latter consist chiefly of lower nitro-compounds which have been formed either from impurities or by a too prolonged nitration. These lower nitro-compounds have an inferior specific gravity to nitro-glycerin, and float on the top in the form of a dirty scum. In some factories this scum is taken off with flat spoons of hard rubber or porcelain as it forms in the separator, and is at once put into the washing vessel. These lower nitro-compounds are those which in contact with the air are especially liable to gradually decompose, and they may produce violent heating in the contents of the separator; but if the latter be carefully covered and water or other foreign matter prevented from falling in, no decomposition need be feared, and the cumbersome work of scumming can be saved.

(*e*) PRELIMINARY WASHING.

The nitro-glycerin in coming from the direct separation is at once put under water. For this purpose round tubs, lined with sheet-lead (L, see Fig. 178), are used. They have an inclined bottom and two taps (M). One of these taps is at the lowest point and discharges into the gutter leading to the filter-house, whilst the other is placed slightly above the normal level of the nitro-glycerin in the tub, so that only washing-water and no nitro-glycerin can run out through it. It is connected with the "deposit of washings," which is described further on. A suitable quantity of water, which in winter has to be previously heated with steam in a vat standing outside the building, is run into the washing vessel. The nitro-glycerin is then allowed to run in in a thin stream from the separator. Compressed air is blown in through a perforated lead pipe (N) lying on the bottom of the vessel, and this keeps the nitro-glycerin constantly agitated.

At the commencement the washing-water should have a temperature of about 60° F., and this should not rise above 86° F. during the running in of the nitro-glycerin. On the other hand, it must not be too cold, or the nitro-glycerin would at once freeze on to the sides and bottom of the vessel, and since it still contains considerable quantities of acids, such freezing might set up dangerous decomposition. Should it, nevertheless, freeze on to the sides, it must be at once carefully rinsed off with water.

In order to rapidly neutralize the large quantities of acid that are always carried in with the nitro-glycerin, some calcined soda may be put direct into the washing-water, or better still, a solution of soda may be added. This causes the mass to froth up on account of the copious development of carbonic acid it originates. The amount of soda required is considerable, and it also decomposes part of the nitro-glycerin. It is therefore preferable to wash repeatedly with water before the soda solution is added. Since the difference between the specific gravities of water and nitro-glycerin is very considerable (*i. e.* 1·6 to 1), the two liquids separate completely in the washing-tub within a few minutes after the air-tap is shut off, and a very short rest is sufficient to allow the nitro-glycerin to settle in order that the water may be run off and a fresh quantity run in. Small particles of nitro-glycerin are nevertheless carried by this water into the gutter leading to the deposit of washings, where it is sent.

As is the case with the gun-cotton, nitro-glycerin, in spite of repeated washing, will still contain minute traces of acid, and accordingly a small quantity of soda solution has to be added to it. When this has been done, the whole is run down a gutter to the filter-house, where the final washing is effected.

(*f*) THE BOUTMY-FAUCHER PROCESS FOR THE MANUFACTURE OF NITRO-GLYCERIN.

Entirely different to all the above described methods for the manufacture of nitro-glycerin, is that invented by Boutmy and Faucher, who deposited descriptions of their inventions in a sealed letter with the French Academy of Science in the years 1872 and 1875. Boutmy and Faucher started from the principle established by Berthelot, which was dealt with in Vol. i. p. 172. This principle may be again stated as follows:—

"If a system of simple or compound bodies under constant volume and pressure undergo a chemical or physical change without any exterior mechanical effect being produced, the heat developed depends solely on the initial and final state of the system, and is the same, whatever the nature or the sequence of the intermediate changes may be." With the usual process of nitration, heat is developed by mixing the sulphuric acid and nitric acid, and also by the reaction of the acid mixture on the nitro-glycerin, and the quantity of heat developed in the process is therefore the sum total of the heat developed in the two operations. Since the acid mixture is cooled down before using it, only that heat is developed in the ordinary process of manufacture which is formed by the nitrating process itself. Boutmy and Faucher divided the process into three stages:—First, they made a mixture of nitric acid and sulphuric acid; secondly, a mixture of sulphuric acid and glycerin; and the above two were mixed together to effect the nitration. The sum of the heat developed is the same as in the usual process, but the quantity of heat developed in the single stages is slight, and the heat developed with the actual nitrating operation—that is to say, when the nitro-sulphuric and the sulpho-glyceric mixtures are brought together—will be smaller than that developed in the ordinary process of nitration. The temperature with the ordinary process may, according to Dr. Dupré, rise to 41·1° C.; whilst with the Boutmy-Faucher process the heat developed is only sufficient to raise it to 21·9° C. This smaller development of heat would therefore be sufficient to give the Boutmy-Faucher process a considerable advantage over the usual one, if it did not introduce disadvantages which more than counterbalance the gain. This, however, as will be seen later, is the case.

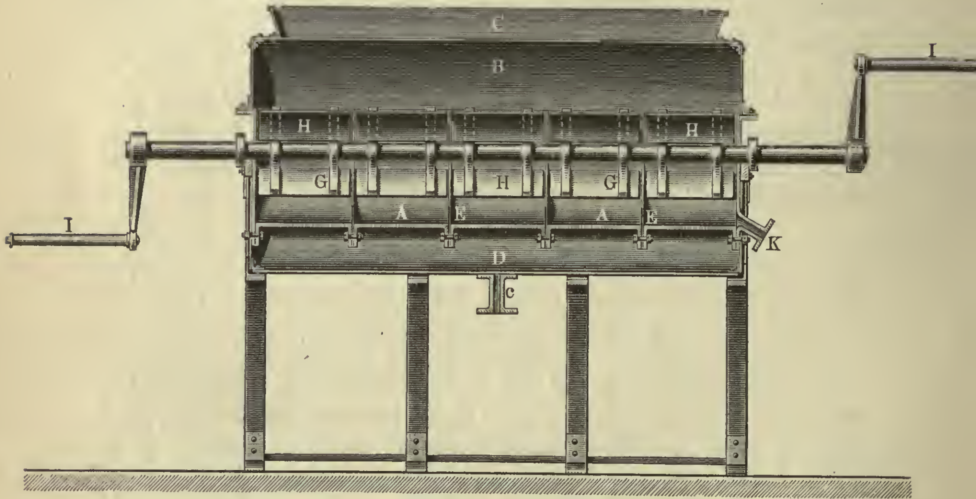

Fig. 179.—One-24th full size.

The Boutmy-Faucher process was introduced in the French gunpowder factory of Vonges in 1872, but was only carried out on a small scale, and although explosions occurred there, as well as during experiments at St. Médard, it was nevertheless used until recently, special precautions being taken.

This process was also used on a large scale in the factory of the Explosives Company, Limited, at Pembrey in Wales, but it was the cause of a very disastrous explosion there, which led to the closing of the factory. The process of manufacture at Vonges, as far as the author is acquainted with it, is as follows:—

(1) *PREPARATION OF THE NITRO-SULPHURIC MIXTURE.*

This consists of equal parts of nitric and sulphuric acids. The acids are brought into the factory in earthenware vessels. A carboy of nitric acid and one of sulphuric acid are put on to a scale, and into each of them is put a contrivance similar to the cork and the glass tube of a washing bottle, but made of lead, the joint being made tight by means of wet clay. The shorter tube is connected with an air compressor, and the other or outlet pipe of both carboys delivers into a third carboy, in which the mixture takes place. By blowing air into the earthenware vessel the acid is forced out. As soon as the scales, the weights on which have been properly adjusted, are again balanced, the inlet of air is stopped. The carboys containing the nitro-sulphuric acid are then placed in a pit filled with water for cooling, where they remain until the next day.

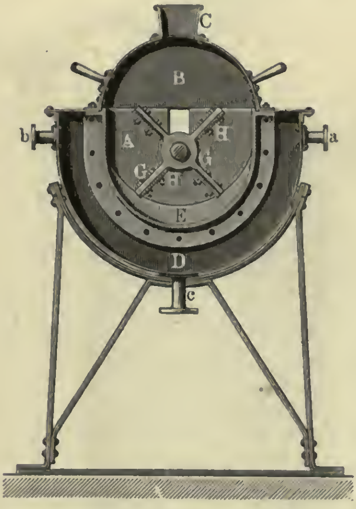

Fig. 180.—One-24th full size.

(2) *PREPARATION OF THE SULPHO-GLYCERIC MIXTURE.*

For each 100 parts of glycerin, 320 parts of sulphuric acid are used. In order to prevent heating of the mixture of glycerin and sulphuric acid, a means of cooling has to be provided, as otherwise considerable quantities of acrolein and charred substances would be formed, which would prevent the subsequent separation of the nitro-glycerin.

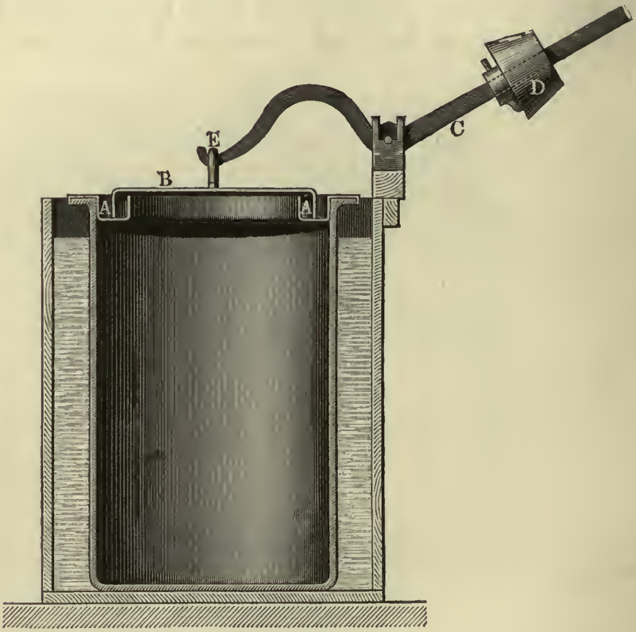

FIG. 181.—One-12th full size.

The apparatus used for the preparation of the sulpho-glyceric mixture is illustrated in longitudinal section in Fig. 179, and in cross section in Fig. 180. It consists of a semi-cylindrical cast-iron vessel (A), which is closed by a semi-cylindrical cover (B). On the top of the latter an opening is left, into which a funnel (C) for introducing the mixture is fixed along the whole length of the apparatus. The lower semi-cylindrical vessel is surrounded by a wrought iron-jacket (D), through which cold water circulates, and is fitted with an outlet pipe (a), an overflow pipe (b), and a discharge branch (c). The vessel is composed of several parts connected together by flanges and bolts. Between each part is a lead partition (E), which serves both for making the joint and for dividing the vessel into compartments.

The apparatus is traversed by a wrought-iron shaft (F), to which are fixed a number of cross-arms (G) corresponding to the compartments. The two cross-arms in each compartment are connected by lead plates (H) forming flat paddles. These paddles are arranged spirally along the shaft. The whole apparatus is carried on a wrought-iron frame, and the shaft is revolved by hand by means of two crank handles (I). The branch pipe (K) enables the apparatus to be quite emptied.

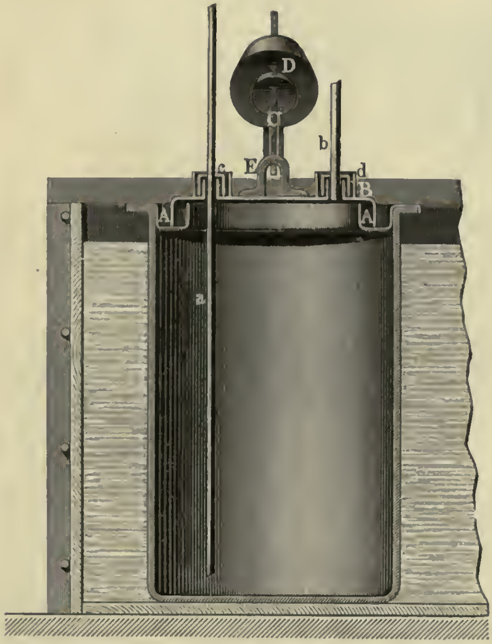

Fig. 182.—One-12th full size.

The apparatus is charged with 41·6 kilogrammes of sulphuric acid and 13 kilogrammes of glycerin, run in a thin layer along the sides of the funnel on to the acids. The lead paddles effect a very perfect mixture on account of their shape and spiral arrangement. The handle is turned for from 1 to 1½ hours, according to the season. As soon as the mixture is sufficiently cooled stirring is stopped, and the sulpho-glyceric mixture run into earthenware vessels, which are put into a pit filled with cold water and allowed to remain there until the next day.

(3) *PRODUCTION OF THE NITRO-GLYCERIN.*

Ten earthenware pots at a distance of 0·80 metre from centre to centre are placed in a long trough filled with water. The pots are 0·65 metre high and 0·40 metre diameter. Originally cast-iron pots were used, but after the explosions that happened at Vonges and St. Médard, their use was discontinued. The mixture used consisted of

		KILOGRAMMES.
Sulpho-glyceric acid	Glycerin	10
	Sulphuric acid ...	32
Nitro-sulphuric acid	Sulphuric acid ...	28
	Nitric acid	28
		98

The earthenware vessel is first charged with sulpho-glyceric acid, care being taken that no straw from the carboys falls in. The nitro-sulphuric acid is then slowly run in by a pumping arrangement similar to that previously used for mixing it.

The earthenware vessels are illustrated in Figs. 181 and 182. On their upper edge they have a lead gutter (A), filled with sulphuric acid. Into this gutter a lead cover (B) is put, through which two pipes pass. One of them (a) goes to the bottom, and serves for agitating by compressed air, and the other (b) serves as an outlet pipe for the fumes. The two pipes can be taken out. They are connected with a small cap (c), which stands in a groove (d) on the cover, also filled with sulphuric acid. A lever (C) is fixed on an arbour on the trough; on one side is a weight (D), and on the other side a hook engaging with a ring (E) on the cover. The weight is so placed that if through the development of gas in the interior of the vessel the cover be lifted in the slightest degree, the lever falls down at once and entirely lifts off the cover. In spite of all these precautions, the difference between the original temperature of the mixture and the temperature of the reaction amounts to over 20° C. The mixture is allowed to stand over-night. At first nitro-glycerin is gradually formed, but by and by more rises, until finally a layer of nitro-glycerin collects on the top. In the morning the acids are first drawn by means of the taps at the bottom, and then the nitro-glycerin.

In Pembrey, where much larger quantities were treated at the same time, the apparatus illustrated in Fig. 183, which is taken from Colonel Majendie's report on the explosions which occurred at this factory on the 11th and 17th November 1882, was used. A large wooden vat (A) was placed over a safety-tank (A_1) made of concrete. In it were placed the nitrating vessel, with its cover (C) and gas-pipe (D). A second iron vessel (E) was connected with the nitrating apparatus at the top by means of a wide pipe (e_1), connected by flanges with the upper part of the nitrating apparatus, and at the bottom by means of a lead pipe (e_2), similarly connected with the lower part of the apparatus. At the bottom a discharge valve (F), 12 inches diameter, was fitted. This valve was faced

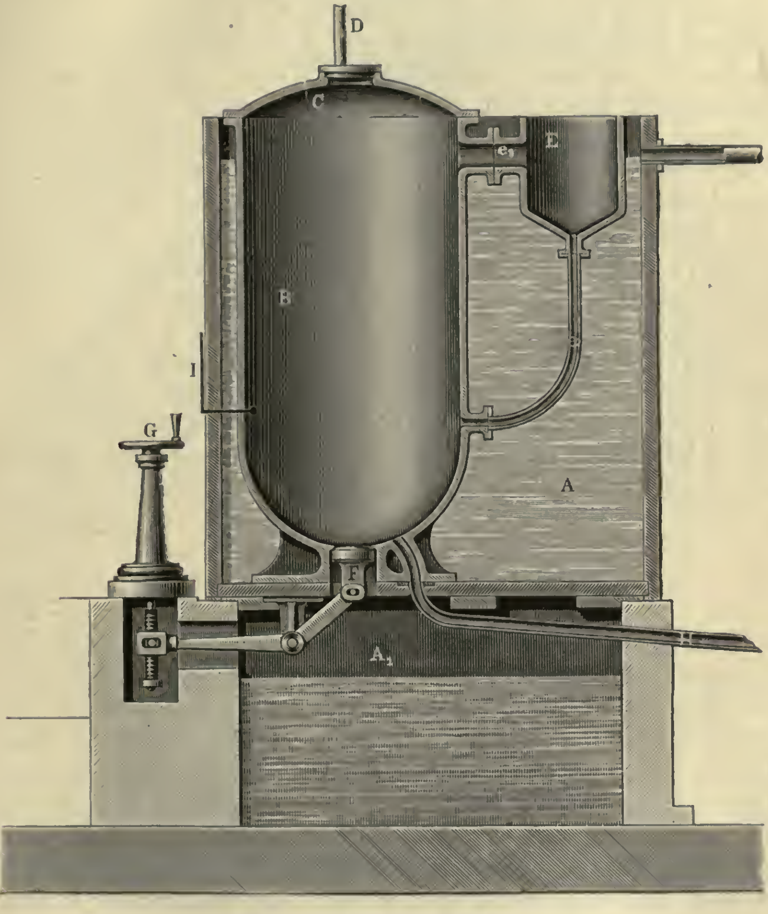

Fig. 183.—One-50th full size.

with asbestos and actuated by means of a lever, screw, and hand-wheel (G), so that in case of a decomposition the charge could run off into the safety-tank within 1½ minutes. There was also a discharge pipe (H) provided for running off the acids, and a thermometer (I), which passed through the wooden vat and could be observed from outside. In this apparatus 1500 lbs. of nitro-glycerin were produced in one operation. The nitro-sulphuric mixture finally attained such a height in the apparatus that it reached to the bottom of the secondary vessel (E). The sulpho-glyceric acid was run in through the secondary vessel, it being supposed that it would run through the nitro-sulphuric mixture from below. Any nitro-glycerin carried over ran into the secondary vessel through the upper connecting tube. After the operation was finished, the nitro-glycerin was drawn off by means of a syphon, and the waste acids passed into the secondary separator. In this nitrating apparatus no means for agitating the mixture were provided.

In Vonges the nitro-glycerin, as it left the apparatus, was put into an enamelled cast-iron vessel, in which it was washed with water. On the bottom of this vessel there was a discharge pipe, closed by a cork stopper, through which the nitro-glycerin was drawn off into small earthenware vessels.

(*g*) Final Washing and Purification.

The nitro-glycerin coming from the preliminary washing still contains many impurities. There are traces of acids, slimy impurities from the nitration, and soda mud from the soda solution. In order to give the nitro-glycerin the stability required to stand storage, sometimes for years, under varying climatic conditions, and also to make it more suitable for the manufacture of dynamite mixtures which will not exude, it is necessary to thoroughly purify it. This final purification is effected by thoroughly washing it with lukewarm water, as a rule in wooden lead-lined vats. There are people who remember the washing being performed at Nobel's factories in a petroleum barrel sunk into the ground, into which the nitro-glycerin and water were put and churned by a workman with a wooden plate fixed to a rod. Later on, the nitro-glycerin was first washed in wooden vats by agitating it with wooden stirrers, and then in a second vat fitted with agitators, such as are used in churns, which were worked by means of gearing. Fig. 184 shows a churn-washer such as was used for treating very large quantities of nitro-glycerin. No description of it is required, as the action of the apparatus is fully shown by the illustration.

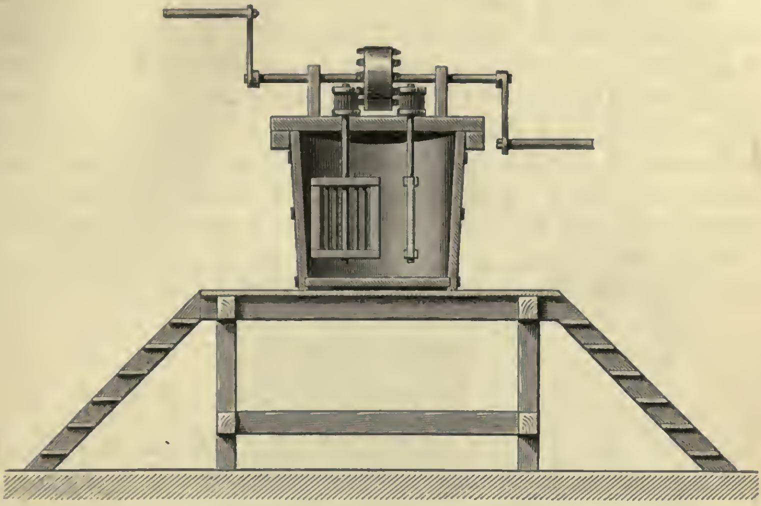

FIG. 184.—One-40th full size.

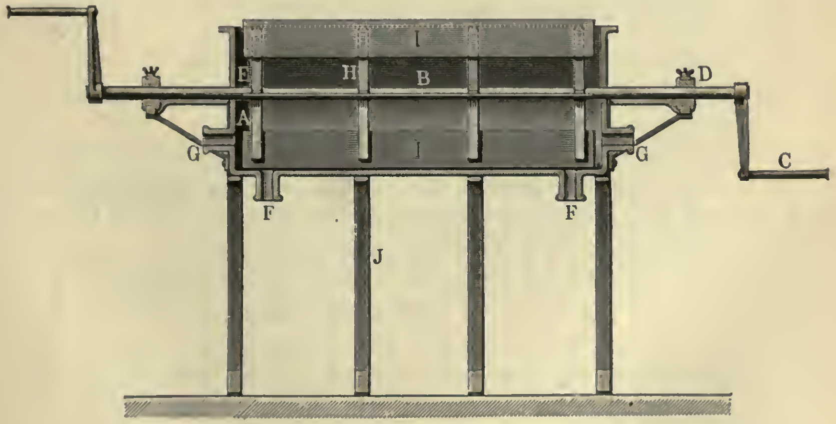

FIG. 185.—One-24th full size.

With the Boutmy-Faucher process at Vonges, the washing apparatus illustrated in longitudinal section in Fig. 185, and in front elevation in Fig. 186, was used. It consisted of a semicircular cast-iron vessel (A), in which a cast-iron shaft (B) revolved in copper plummer blocks (D) by means of the handles (C). Above the semi-cylindrical part there was a box (E) to give additional depth to the vessel. The two branches (F) on the bottom were for discharging the washing-water. The shaft carried four sheet-iron plates (I) attached to four sets of cross-arms (H). The whole was supported by a wrought-iron frame (J). Such an apparatus took about 55 kilogrammes (121 lbs.) of nitro-glycerin, which was washed in it from 10 to 20 times, until the nitro-glycerin was perfectly free from acid. The washing-water was at a temperature of 30° C., and for each of the first two washings 100 grammes of sodium bi-carbonate were added to the water.

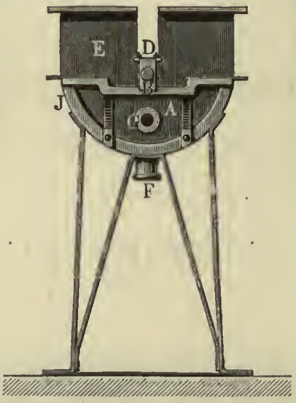

Fig. 186.—One-24th full size.

At present agitation by air is exclusively used, and the washing can consequently be done in a very simple apparatus. Wooden lead-lined vats, which are fitted with two taps, one at the lowest point for discharging the nitro-glycerin, and the other one just above the highest level of the nitro-glycerin for drawing off the washing-water, are used. A little soda solution is added to the nitro-glycerin before it leaves the first washing-house, and is sent down to the final washing-house with it. In the latter house, the nitro-glycerin is run into the above-mentioned vats and thoroughly agitated in them by compressed air blown in through a perforated pipe reaching down to the bottom, the water being frequently changed.

In some factories the nitro-glycerin is washed with water at

50° C., because it is considered that the last traces of acidity can be eliminated more quickly with hot water than with cold. This is true to a certain extent, because the traces of acidity are generally due to lower nitrogen acids, which volatilize at about 50° C., but on the other hand the nitro-glycerin itself is volatile, in quantities that are by no means negligible, at a temperature of 50° C. The consequence, therefore, is a loss of nitro-glycerin and considerable inconvenience to the workmen, who are forced to remain in an atmosphere filled with nitro-glycerin vapour; and even if they no longer suffer from nitro-glycerin headache, they nevertheless suffer from heavy pressure in the head, heavy

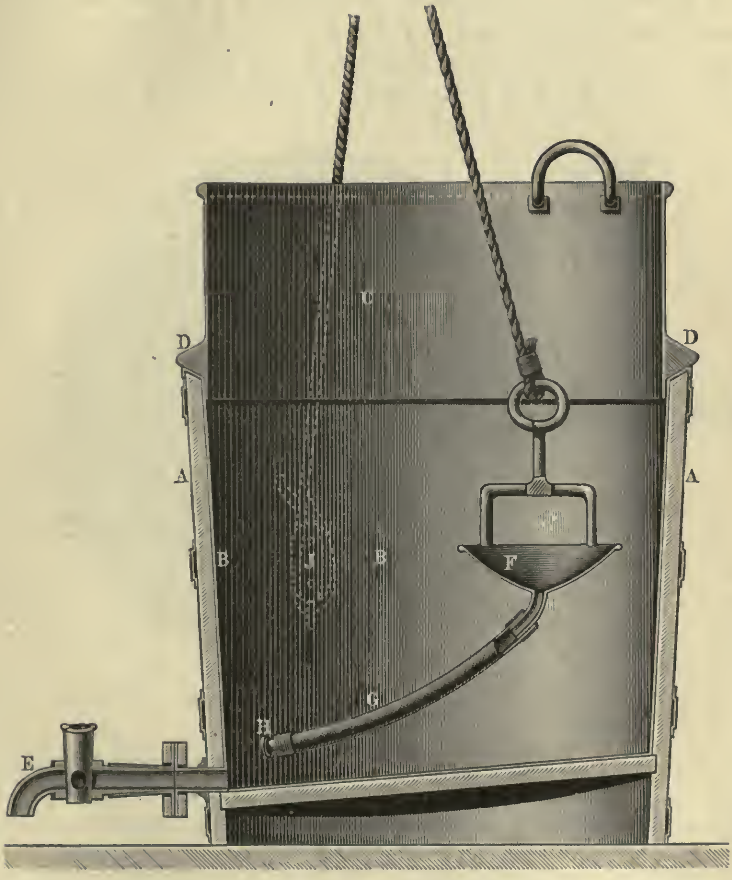

Fig. 187.—One-16th full size.

breathing, and other complaints. Moreover, the saving of time is effected just as well by more frequently changing the washing water.

An apparatus which was used at Nobel's dynamite factory at Ardeer in Scotland, and at the Royal Gunpowder Factory, Waltham Abbey, for washing nitro-glycerin, is illustrated in section in Fig. 187 and in plan in Fig. 188. It consists of a wooden vat (A) with a cylindrical upper part (C) for preventing splashing, which is burnt on to the rim (D) of the lead lining (B) of the vat. At the bottom of the vat is a tap (E) for drawing off the nitro-glycerin.

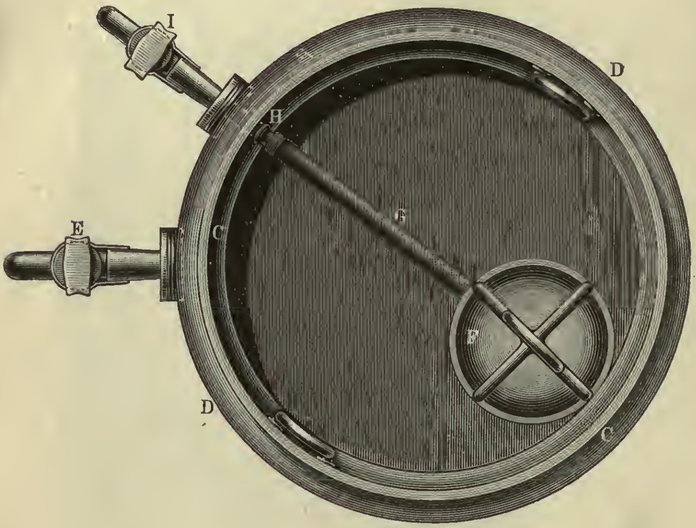

FIG. 188.—One-16th full size.

The washing in this apparatus is done at a temperature of 50° C., which is obtained by admitting water at 70° C. into the vat simultaneously with the nitro-glycerin in such proportions that a mixture of the two gives a maximum temperature of 50° C.

In order to eliminate the last traces of acidity, the final washing of the nitro-glycerin at Waltham Abbey is done with soda solution. First, 4 inches depth of soda solution are run in, then a charge of 750 lbs. of nitro-glycerin, then again 4 inches of soda solution, and then the vat is filled up with water. In this way the washing solution contains 2·68 per cent. of soda. After 15 minutes' washing the solution is run off, then 2 inches of the soda solution and sufficient water to dilute it to 1·91 per cent. of soda are added, and the washing continued for 15 minutes. The washing solution is again drawn off, and a fresh quantity of soda solution and water, again of 1·91 per cent. strength, added, and the washing continued for 45 minutes. The washing solution is then again run off, and the nitro-glycerin finally washed for 15 minutes with a 0·38 per cent. soda solution.

The soda solution is drawn off after each washing by a skimmer (F), which consists of a funnel with a cross frame and ring soldered to it. A piece of india-rubber pipe (G) is attached at one end to the outlet of the funnel, and at the other to the socket (H), passing through the wall of the vat. Into this socket a tap (I) is fixed. The skimmer could formerly be raised or lowered by a rope, which passed over a pulley on the ceiling, and could be secured at any height by twisting the rope round the staple (J). In order to draw off the soda solution, the skimmer is let down into the liquid until its upper edge is almost level with the surface of the nitro-glycerin and the tap opened. The washing liquor is thus run off and goes to the deposit of washings.

An explosion occurred in the washing-house at Waltham Abbey, and at the request of the War Office Committee appointed to inquire into the matter, the author carefully examined the case. The result of his examination was that he had no doubt that the cause was the sudden dropping of the skimmer on to the nitro-glycerin on the sides, which was of course in a very sensitive state on account of its being heated to 50° C. The weight of the lead skimmer which was in use at the time of the accident was 36 lbs.

The apparatus at present in use at Waltham Abbey has been modified. The vat has been made higher and the separate top piece omitted. The skimmer is made of thin sheet-iron, and is normally held up by a counter-weight outside the building. When the solution has to be run off, the skimmer is pulled down and held in the required position until the running off is finished. This apparatus was introduced at Ardeer with the special object of being able to draw off the washing-water without carrying away any nitro-glycerin, since they have to treat varying quantities of nitro-glycerin in the apparatus. At Waltham Abbey there was no necessity for such an apparatus, as the quantity treated was always the same. The running off of the soda solution can be done without having recourse to such a complicated appliance by placing taps at various levels on the vat, which does away with the danger inevitably attending the use of any moving parts like this skimmer. Moreover, as has been mentioned, washing with a warm soda solution or warm water is superfluous under ordinary circumstances.

Washing is an operation which requires great care. It is practically impossible to give a definite opinion on the quality of a glycerin and the acids used before the nitro-glycerin has been washed the usual number of times, and even when this has been done one is sometimes surprised to find that the nitro-glycerin will not stand the regulation heat test at all or only very badly. If this be the case, a further addition of soda is of very little use, as some of the soda solution always causes partial decomposition of the nitro-glycerin, and nitrous gases may be formed. The continual blowing through of compressed and possibly slightly heated air is efficacious, but repeated washing is still better. Which of these means is to be preferred depends considerably on the special circumstances of the case, and it is often very difficult to hit upon the right thing, as so very little is known of the causes of such bad properties in nitro-glycerin.

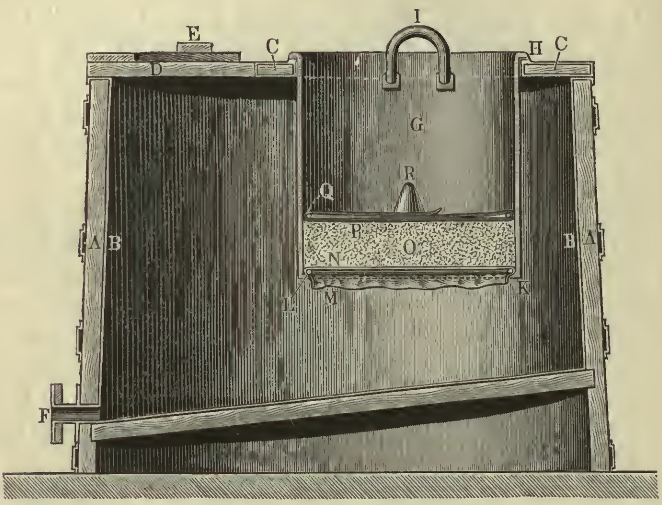

Fig. 189.—One-16th full size.

After the nitro-glycerin is thoroughly washed, it is filtered to remove all slimy particles, soda mud, and also any foreign particles that may have got in. The simplest and most usual method is to filter through flannel or felt stretched on a frame, which is put over a vessel into which the nitro-glycerin gradually runs. It is advantageous to use two such frames one over the other. The whole of the water can never be completely drawn off in the washing vessels, so there will always be a thin layer of it floating on the nitro-glycerin, and the nitro-glycerin itself will contain fine drops of water suspended in it through the violent agitation with air. It is, therefore, the custom in many factories to put a layer of dried salt on the filter, the action of which is to absorb the water, and at the same time promote more perfect filtration. After it has been used several times, the layer of salt is removed and dissolved in water, when any nitro-glycerin absorbed by it will sink to the bottom and can be recovered. The slimy particles easily clog the filters, and the latter must therefore be washed from time to time in warm water. In order to separate these slimy particles before they can clog the filters, some factories have tried putting broken glass on to the filters with fairly good result.

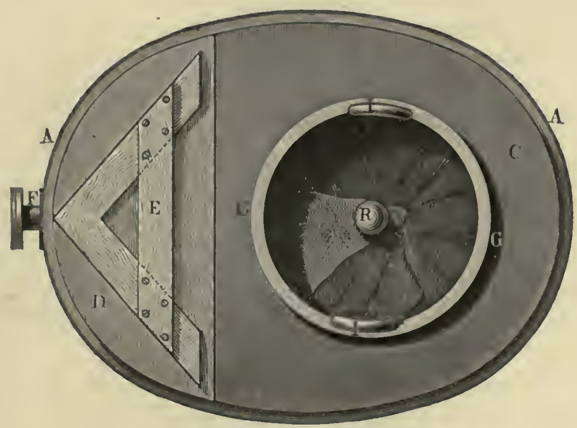

Fig. 190.—One-16th full size.

At Ardeer and Waltham Abbey, the apparatus illustrated in section in Fig. 189, and in plan in Fig. 190, is used for filtering the nitro-glycerin. It consists of a wooden vat (A) with inclined bottom, lined throughout with lead (B). About two-thirds of the upper part is permanently covered by a lead-lined wooden cover (C), the rest is closed with a movable wooden cover (D), which can be lifted by the handle (E). The nitro-glycerin is drawn off through the tap (F). In the loose cover there is a round opening, in which a lead cylinder (G) can be placed. In the top of G is a rim (H), which rests on the cover, and two handles (I), by which it can be lifted off. Round its lower edge there is an internal flange, on which the filters rest. The filter consists of a brass ring (L), over which wire gauze (M) is fixed, a filter cloth (N) being laid on the top of this. The whole is covered with a layer of common salt (O), and a second filter cloth (P) held down by a lead ring (Q) placed above it. The upper filter cloth is folded round and kept in its place by a conical lead weight (R).

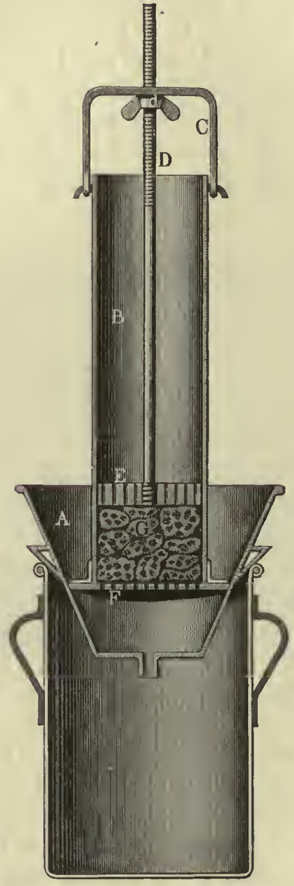

Fig. 191.—One-10th full size.

In the author's opinion the use of this filtering apparatus is also a source of danger, since the heavy filtering cylinder suspended over the nitro-glycerin, together with the stretching rings of the filter cloth, has to be lifted in and out from time to time for cleaning, and it is quite possible that an accident might be caused by its being dropped. The loose lead weight for holding down the filtering cloth also seems objectionable, whilst the arrangement does not give any results which could not be easily obtained by simply putting a layer of salt on wooden cloth-covered filter frames.

At Vonges a sponge filter was employed, the use of which was suggested by Hagron. He found that sponges had a peculiar action on nitro-glycerin containing water, retaining one liquid and letting the other run through. The Vonges filtering apparatus is illustrated in Fig. 191, and may be used with advantage for operations on a small scale. It consists of a sheet-iron funnel (A), with two handles at the sides and a discharge pipe at the bottom. A wrought-iron cylinder (B) is placed into the funnel which has two hooks riveted on, on to which a bridle (C) is fixed. An iron bar (D) passes through the bridle, its upper end being screwed so that the bar can be raised or lowered by means of a wing-nut (d). A perforated wooden disc (E) is fitted to the iron bar, and on the bottom of the cylinder is placed a perforated iron plate (F). Between F and E a number of pieces of sponge are put, which can be compressed by turning the nut (d). The nitro-glycerin coming from the washing apparatus is allowed to run through this filtering apparatus one or several times, according to circumstances.

In some factories the nitro-glycerin is not filtered over salt or similar absorbing materials, but it is merely allowed to stand in a suitable vessel for some time, when the bulk of the water will rise to the surface and the nitro-glycerin can be drawn off fairly free from water. For the manufacture of blasting gelatine, smokeless powder, etc., it is essential that the nitro-glycerin should as far as possible be free from water, and it is therefore convenient to let it stand in a warm room for a day or two, when all the water will separate completely. The warmer the nitro-glycerin, the more rapid is this separation; and, in fact, formerly on making blasting gelatine, the quite milky, water-bearing nitro-glycerin was brought to a temperature of between 50° and 60° C. in mixing pans, when the water was perfectly separated within half-an-hour, and the nitro-glycerin itself became quite clear.

(*h*) SECONDARY, OR AFTER SEPARATION.

The acids coming as waste from the first separation, always still contain a small quantity of nitro-glycerin. Besides this, nitro-glycerin and also lower nitro-compounds are formed in them after prolonged rest. It will be seen later on, when dealing with working results, that a large part of the glycerin is not converted into nitro-glycerin during the nitration, but that it is simply dissolved in the sulphuric acid and remains with it in the waste acid. When these waste acids are allowed to rest for a prolonged time, the small quantity of nitric acid contained in them gradually acts on the glycerin, and traces of some nitro-compounds are formed even after undisturbed rest for a fortnight. Since they are only of inferior stability, and are liable to decomposition in contact with strong acids or moist air, care must be taken to separate them from the waste acids before the latter are further utilized.

Originally ordinary vats were used, in which the waste acid was allowed to rest and the nitro-glycerin drawn off after a few hours. In some places the waste acid was run into glass carboys, and these put in the open air. After a few days the nitro-glycerin floating on the top was drawn off by syphons. This method of proceeding, however, was the cause of a violent explosion at Matagne-la-Grande, from which the manager of the company lost his life. At present special separators are exclusively used. They are something like the nitrating apparatus—namely, large cylindrical lead vessels, generally standing in a wooden vat with cold water between the two. The vessels are closed with a conical lid, at the apex of which is a glass tube for observing the level of the liquid, terminating in a fume-pipe to carry off any gases developed. Inside the vessel there are, as a rule, one or several leaden cooling-worms, and on the lowest part of the inclined bottom a large outlet tap, which is connected with a safety-tank, but which can also be connected with a gutter leading to the waste acid storage tanks when required. A perforated lead pipe to admit compressed air passes down to the bottom of the apparatus. Below the glass tube an earthenware tap is fixed, or the tube itself is fitted with a ground-in glass stopper on one side. The apparatus is filled with the waste acids (as a rule, about two tons are required) to about half-way up the glass tube, and the acid is then left to rest. After a few hours, nitro-glycerin and black or dark-coloured impurities will be found in the glass tube. These must be drawn off through the small tap or stopper into a small vessel; a little acid is then introduced from a small storage vessel, and the apparatus is again allowed to rest. In this manner any nitro-glycerin separating out can be drawn off several times during the day, until after about a week no further nitro-glycerin is formed. The nitro-glycerin which is drawn off is at once put into a vessel filled with water, where it is washed, and then taken to the filter-house.

If during the separation the temperature should from any cause rise unduly, which will be indicated by thermometers provided for the purpose, water is allowed to run through the jacket and cooling-worm, and, in case of need, air is also blown in. Decomposition in such an apparatus only takes place gradually, and it can be kept under control, sometimes for many hours, by cooling and stirring with air. Nevertheless, when there has been a want of proper precautions, and when the nitro-glycerin has been allowed to rest for a long time over the acids, explosions have happened; but on account of the small quantity of nitro-glycerin present, they cause but little damage. In Great Britain the regulations demand that a workman shall constantly be in the secondary separating-house day and night, and that there shall be efficient control to ensure that any nitro-glycerin separating out shall be drawn off at not too long intervals.

When the second separation is finished the waste acid is stored in lead or cast-iron vessels. Its average composition is—

	PER CENT
Nitric monohydrate	10
Sulphuric monohydrate	70
Water	20
	100

and it thus corresponds to a nitrous vitriol containing much nitric acid. It is advantageous to let the waste acid rest in the storage vessels for a prolonged time before it is used, because, as has been already said, lower nitro-compounds always form, and these rise to the surface and are slowly decomposed by contact with air. The storage vessels may be conveniently placed in the open air, but must be well covered and protected from rain; nevertheless, when the air is very moist, or on very warm days, or during violent showers, the acid in the storage vessels sometimes undergoes decomposition, which is accompanied by the development of thick brown red vapours until all nitric acid is driven out. When it is inadmissible to let such a decomposition take its own course, safety-tanks are provided, into which the whole contents of the storage vessels can be run.

(*i*) TREATMENT OF WASHINGS.

The large quantities of washing-water which come from the various apparatus used in the manufacture of nitro-glycerin sometimes carry with them very considerable quantities of nitro-glycerin. The waste water from the separation process is, as a rule, acid; that from the final washing alkaline, but the quantity of alkali is not sufficient to neutralize the whole of the acid in the other waste water. The waste washing-water also carries with it all soda mud. It is necessary to completely free these washings from nitro-glycerin. For this purpose there is either a special catcher provided in each building, or, which is a better plan, the whole of the waste washing-water is treated in a special building.

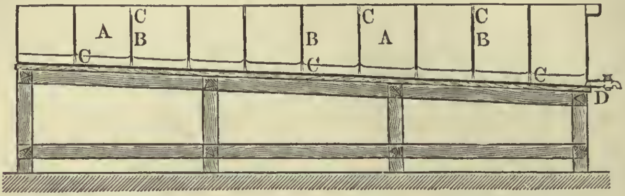

FIG. 192.—One-48th full size.

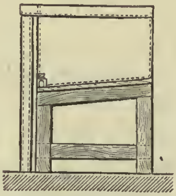

FIG. 193.—One-48th full size.

The apparatus for separating the nitro-glycerin from the waste washing-water are generally overflow apparatus (labyrinths, trop-pleins) consisting of oblong rectangular lead vessels (A, Figs. 192 and 193) divided by partitions (B) into a number of compartments. The partitions have holes (C) alternately at the top and bottom, by which the water is caused to alternately rise and fall and pass through the apparatus in a zigzag path, dropping on its way any suspended nitro-glycerin particles. The deposited nitro-glycerin is drawn off from time to time through a tap (D) or a waste-water channel. It is advisable to give such overflow vessels an inclination in the direction of their length and breadth, so that the whole of the nitro-glycerin will collect in the lowest part of the apparatus, and can be more easily drawn off. The collected nitro-glycerin is washed, and then taken to the filter-house. Since, as a rule, the washings are acid, and the apparatus of lead, considerable wear takes place, which necessitates frequent repairs.

(*k*) SAFETY-TANKS.

In order to quickly render the contents of nitrating or separating apparatus harmless in case of decomposition occurring, safety-tanks are provided. They are either pits with cement or stone walls, or leaden tanks. The latter are made in the same manner as sulphuric acid chambers, on strong wooden frames, round which lead supporting strips are soldered. As a rule, they have an inclined bottom in order to enable any nitro-glycerin which may be carried into them to be conveniently drawn off. The safety-tanks must be sufficiently large to contain, when half filled with water, at least ten times the quantity of the charge to be drowned. As a rule, an air agitating pipe is also provided in these tanks, in order to keep the contents in constant agitation whilst a charge is being drowned. The drowning of a charge is accompanied by the violent development of nitrous fumes, and the water is consequently considerably heated. The water kept in the tanks should be renewed from time to time, to prevent the formation of organic growths. It is best to place the safety-tanks outside the buildings in which the operation is done. Formerly, they were put immediately below the apparatus for the sake of convenience, but there is no object in doing so, because the charge runs out in a thin layer in a gutter to the safety-tank, and is from this alone rendered fairly harmless, and if the tank is in the house itself the large quantity of nitrous fumes developed when it is used only damage the building.

(*l*) WORKING RESULTS.

It has already been mentioned that according to the chemical equation 227 parts of nitro-glycerin should be formed from 92 parts of glycerin and 189 parts of nitric acid, or 246·74 parts of nitro-glycerin from 100 parts of glycerin and 205·43 parts of nitric acid. Such results, however, are never obtained in practice, the chief reason being that as the nitrating operation progresses the quantity of nitric acid in the mixture becomes less and less, whilst the sulphuric acid is not only not used up during the process, but increases in bulk as it becomes more and more diluted by the water formed during the reaction. In this way the glycerin as it continually runs in finds less and less nitric acid to combine with, and finally the nitric acid will be so widely disseminated through the mixture that the greater part of the glycerin will be dissolved by the sulphuric acid and thus escape nitration. It is true that less glycerin could be used and thus all be completely transformed into nitro-glycerin; but in manufacturing on a large scale it is simply a question of economy, and a calculation will at once show that in order to effect complete nitration of all the glycerin, a large excess of nitric acid will be required, and thus the sulphuric acid will not be completely utilized, and it will be found that the extra cost of so working will be much larger than that of the glycerin lost. Therefore an average, found by practice, is struck, the quantity of glycerin used being such that will leave after the nitration about 10 per cent. of nitric monohydrate in the waste acid.

With Kopp's very primitive process a yield of from 143 to 200 per cent. was obtained, but the glycerin used was not pure and on continued washing some of the nitro-glycerin would still have been lost. It is said that by Mowbray's process 224 parts of nitro-glycerin for every 100 parts of glycerin were obtained, but that such a high result was got regularly is a little questionable. In the factory described by Capitaine, 151 to 190 per cent. were obtained, which is small. In well-managed factories, since 1874, about 200 per cent. of nitro-glycerin have been obtained when a normal acid mixture was used. Cases where the yield is over 210 per cent. are very rare, and it can only be obtained by using the highest concentrated nitric and sulphuric acid, in the cold of a hard winter. An average yield during the summer months of 200 per cent. and of 205 per cent. in the winter ones may be considered as good. Of course, this only refers to nitro-glycerin perfectly free from water.

As mentioned previously, the yield varies with the temperature of the air and that of the cooling-water, and it would seem that more nitro-glycerin is obtained at moderately low temperatures than at higher ones; the cause of this, however, has not yet been satisfactorily explained. The difference of specific gravity of the acids at the lower temperature is not sufficient to account for the sometimes considerable difference in the yield. It is more probable that the larger quantity of hyponitric acid that is formed at the higher temperatures, partly decomposes the nitro-glycerin after it has been formed. This much, however, is certain, that the difference in the yield with cooling-water at a temperature of 20° C., and with that of 8° C., is nearly 5 per cent.—that is to say, that in one case 200, in the other 210, parts of nitro-glycerin are obtained from 100 parts of glycerin.

(*m*) GENERAL REMARKS ON THE ERECTION OF NITRO-GLYCERIN FACTORIES.

The conditions which govern the erection of buildings for the manufacture of nitro-glycerin are to a large extent those that must be observed in designing buildings for the manufacture of black powder, except that on account of the greater distance to which *débris* may be thrown in the case of explosion, the buildings must be kept further apart or the quantities allowed in the buildings must be reduced.

The method of constructing the buildings and the various steam-, air-, and acid-conduits will be dealt with in detail under the erection of dynamite factories. It is only necessary here to point out one peculiarity of nitro-glycerin factories—namely, the means of conveying the nitro-glycerin and the waste acids from one part of the factory to another.

Originally leaden pipes or open gutters inside the buildings and pipes outside them were used for connecting the nitrating apparatus and the separating-house, and also the separating-house and filter-house. When such pipes and channels were kept clean, and were frequently washed out, they answered their purpose, although they frequently froze in the winter, and explosions happened in spite of all precautions, especially in those conduits through which nitro-glycerin and acid were conveyed together. There were various causes for this; sometimes the pipe or the gutter was too much exposed to the direct rays of the sun, and the acid nitro-glycerin decomposed. Sometimes the conduit was not laid perfectly level, and depressions caused in it, in which of course nitro-glycerin collected. Since this was in a state of permanent impurity, decompositions easily happened.

At present the use of pipes is avoided as much as possible. Frequently long galleries are made between the buildings along which a man can pass, although by stooping sometimes, and in these an open lead gutter is placed. Such galleries can of course be heated by steam pipes, but since they are costly, it is preferable to carry the gutters on supports between the various buildings. They are conveniently made of strong sheet-lead (about $\frac{3}{8}$ inch for acid gutters, and about $\frac{3}{16}$ of an inch thick for nitro-glycerin gutters). They are either V-shaped or semi-circular in section. In order to ensure their being carried in a perfectly uniform slope, they must be supported by boards, or better still, they may be laid in boxes filled with insulating material and carried on proper supports. The acid gutters are provided with lead covers, and the other gutters with wooden ones, in order to prevent foreign matter falling in. Sometimes they are provided with a second cover as well.

The chief thing is to ensure the conduits being quite rigid and easily accessible and interchangeable. They must be cleaned with the greatest care at short intervals, and should be carefully inspected at frequent periods by a lead-burner. Since there are always differences of temperature between the air and the lead gutter or the mixture running through them, the lead conduits expand, and consequently the gutters buckle; or if they are too stiff, fine cracks are formed at the soldered joints, through which nitro-glycerin gradually oozes. To prevent freezing of such conduits in the winter, means of continuously heating them by warm water must be provided.

The buildings for nitro-glycerin factories are best arranged on the slope of a hillside, the acid store and the glycerin heater being placed at the highest point, and the buildings for each successive part of the process following in such a way that each one is lower down the hill than the previous one. The total amount of drop in the ground level required for this varies according to the number of operations, and is sometimes very considerable. Of course it increases with the distance apart that the buildings have to be kept, as there must be sufficient fall for the gutters leading from one to the other—namely, at least 1 per cent.

On the Continent, it is usual to put the apparatus for nitration and separation, and the vats for the first washing into the same building, and sometimes also the safety-tank and an apparatus for preparing the washing-water. The secondary washing and the filtration, and also the serving out of the nitro-glycerin, are done in a second one, and the after separation in a third. In Great Britain, where the quantities allowed in a single building are limited by law, and also in the best continental factories, it is customary to perform each operation in a building by itself, and rather to have several apparatus of one kind in the same building. By these means the working capacity of a factory is considerably increased, and the danger diminished, because only operations of one kind are going on in any building, and so do not interfere with each other, one workman does not get in the way of the others, and there is less likelihood of casualties happening. Of course the difference of levels required is then much greater, and sometimes buildings have to be erected on artificial mounds or on scaffolds.

The greatest care must be given to keep the various apparatus used in the manufacture of nitro-glycerin quite tight. Any trace of leakage of a tap or a joint must be at once seen to and stopped, because it might cause great inconvenience at a moment when least expected. Spilling of nitro-glycerin should not happen at all. If drops are spurted about or fall on to the floor, they must be at once wiped up with a sponge. All sponges used for cleaning apparatus, and especially acid vessels, must after use be at once put into a vessel containing water. Fires and explosions have repeatedly taken place through this precaution being omitted.

Formerly covering the floor with sand or kieselguhr was prescribed for nitro-glycerin buildings. In very rare cases this is still done to-day, but it cannot be too strongly deprecated, since it is impossible to keep such a building clean. It is true that if any nitro-glycerin is spilled, it is taken up by the sand, but at the same time, being out of sight, it is liable to be overlooked and may freeze in the winter. At Ardeer, where the entire factory is built on ground covered naturally with fine beach sand, no other floors at all are provided, but the upper layer is taken off from time to time and fresh sand added. It is much better and more usual to cover the floor round the apparatus where acids or nitro-glycerin are treated with sheet-lead with a rim about $\frac{3}{8}$ of an inch high, and where no liquid mixtures are worked, to make the floor of closely-laid smooth dressed boards. The inlet and outlet openings of the gutters leading into the buildings should be fitted with covers and stoppers in order to prevent propagation of fire or explosion along the gutters. An explosion which occurred at San Francisco attained considerable dimensions on account of first fire, and later, explosion being transmitted in this manner.

2. PROPERTIES OF NITRO-GLYCERIN.

(*a*) Physical Properties.

Pure nitro-glycerin—namely, that made from perfectly bleached nitric acid, chemically pure sulphuric acid, and water-white glycerin, and without any increase of temperature having occurred during its making—is a water-white oily liquid without smell at ordinary temperature. Nitro-glycerin made on a large scale has a colour varying between that of hock and a dirty yellow. If carefully washed and freed from water, nitro-glycerin is transparent. It has a slightly sweet burning taste, and is very poisonous. Schuchard described the symptoms arising from its use from observations on himself. After swallowing a drop, he was attacked with headache, giddiness, and faintness. The effects showed themselves in a much more marked manner when he incautiously swallowed more nitro-glycerin, this giving rise to unconsciousness accompanied by ague (shaking fever).

In the manufacture of nitro-glycerin the cases which have to be chiefly considered are those where it comes in contact with the skin, and especially with the glands of the moist membranes of the nose and mouth, especially when the nitro-glycerin is heated over 86° F. In the latter case it gives off a peculiar smell through evaporation, and the action as a vapour is just as poisonous, although in a lesser degree. Highly nervous and also very robust people seem to be more sensitive to the action of nitro-glycerin than people of normal constitution. Some persons never get over the effects, but in most cases the workers get so completely inured within a day or two to the effects produced by handling nitro-glycerin, that they no longer experience any inconvenience. As a rule, the chief effect is a violent headache, especially at the back of the head. As antidotes, cold compresses applied to the head and neck, fresh air, drinking black coffee, and (under the advice of a doctor only) particularly morphine acetate, may be recommended. Schuchard is convinced that nitro-glycerin penetrates the skin and is decomposed in contact with the blood, giving rise perhaps to the formation of nitric oxide.

According to Werber, the smallest trace of nitro-glycerin can be detected by its behaviour with aniline and concentrated sulphuric acid. On adding the two a purple red coloration is obtained, which is transformed into green by the addition of water.

The specific gravity of nitro-glycerin is 1·600, or more exactly, according to Beckerhinn, 1·599; that of frozen nitro-glycerin is 1·735, which shows that it contracts $\frac{10}{121}$ of its volume on freezing.

Nitro-glycerin dissolves in alcohol, ether, methylic alcohol, benzene, etc. In water it is only soluble to an extremely small degree, about 0·003 per cent. Berthelot says that in presence of a sufficient quantity of water it is completely soluble, and that it therefore should not be allowed to stand long in running water; but this does not seem to coincide with practical experience unless constant renewal of water takes place, and with anything like a large quantity, constant stirring for many hours would be required to dissolve appreciable quantities.

A. H. Elliot ('School of Mines Quarterly,' vol. iv. p. 15) examined the solubility of nitro-glycerin in various solvents both cold (at ordinary temperature) and warm (by putting a test-tube into boiling water). His results are contained in the following table:—

Solvent.	Cold.	Warm.
Water	insoluble	a little soluble
Absolute alcohol	soluble	soluble
Alcohol of 93 per cent.	,,	,,
,, 80 ,,	slowly soluble	,,
,, 50 ,,	insoluble	a little soluble
Ether	soluble	soluble
Chloroform	,,	,,
Benzene	,,	,,
Carbon disulphide	insoluble	partly affected
Concentrated sulphuric acid	soluble	soluble
Glacial acetic acid	,,	,,
Turpentine	insoluble	,,
Methylic alcohol	soluble	,,
Amyl alcohol	,,	,,
Carbolic acid	,,	,,
Nitro-benzene	,,	,,
Toluene	,,	,,
Petroleum naphtha (71 to 76° B.)	insoluble	insoluble
Astral oil	,,	,,
Mineral jelly	,,	,,
Olive oil	soluble	soluble
Stearine oil	,,	,,
Caustic soda solution (1 : 10)	insoluble	insoluble
Ammonium hydrate 0·98 specific gravity	,,	insoluble, a little affected
Nitric acid 1·4 specific gravity	slowly soluble	soluble
Hydrochloric acid 1·2 specific gravity	insoluble, slowly decomposed	slowly soluble
Tin chloride solution	a little affected	affected
Iron sulphate solution	,, ,,	,,
Ammonium sulph-hydrate	insoluble, sulphur gradually separated	decomposed
Iron chloride, 1·4 grammes Fe in 10 cubic centimetres	slowly affected	,,
Borax, 5 per cent. solution	insoluble	insoluble
Acetic ether	soluble	soluble
Acetone	,,	,,
Glycerin	insoluble	insoluble

At + 8° C. (46·4° F.) nitro-glycerin freezes into long whitish crystals, and in so doing undergoes a material change in its properties. It only melts, however, at about 11° C. (51·8° F.). The temperatures given are only correct if the nitro-glycerin be exposed for a prolonged time; and some experiments made by the author showed that pure nitro-glycerin, if suddenly exposed to a temperature of 25° C. (13° F.), produced by a freezing mixture, was not frozen even after some hours. If, on the other hand, a nitro-glycerin crystal, however small, was thrown into the nitro-glycerin thus cooled, the whole mass froze at once. In the same way the thawing of the nitro-glycerin lasted a longer time, but it should not be done at a higher temperature than 50° C. (120° F.).

In a frozen state nitro-glycerin is less sensitive to concussion than in the liquid state, but it seems as if on breaking frozen crystals of nitro-glycerin a peculiar molecular change takes place—at least it is supposed that some explosions are due to this.

According to Hess, nitro-glycerin can be completely evaporated by continuous exposure to a temperature of 70° C. (158° F.). The author's experience has shown him that dynamite exposed for several days to a temperature of 40° C. (104° F.) loses 10 per cent. of its nitro-glycerin, and also that by washing for two hours with water at 50° C. (122° F.) with constant agitation by compressed air a loss of 0·15 per cent. of nitro-glycerin occurs.

(*b*) CHEMICAL PROPERTIES.

The formation and constitution of nitro-glycerin has been dealt with in previous pages. Beckerhinn compared the actual composition of two samples of nitro-glycerin with the theoretical one, and found—

	FOUND.		THEORETICAL COMPOSITION.
	(*a*)	(*b*)	
Carbon	15·42	15·62	15·85
Hydrogen	2·27	2·40	2·20
Nitrogen	—	17·90	18·50

Beckerhinn's experiments were made about twenty years ago, when pure nitro-glycerin was frequently unobtainable.

Hess found that nitro-glycerin extracted from kieselguhr dynamite contained between 18·34 and 18·45 per cent. of nitrogen as determined in Lunge's nitrometer and between 18·42 and 18·55 per cent. where Champion and Pellet's method was used. The author, and many others, using Lunge's nitrometer according to the instruction given by Hess (*i. e.* introducing dilute sulphuric acid in the second instance), have nearly always found the theoretical percentage of nitrogen in pure anhydrous nitro-glycerin.

Nitro-glycerin is reduced by alkalies with the formation of glycerin. According to Hess and Schwab ('Proceedings of the Vienna Imperial Royal Academy of Science,' 1877) this is much promoted by using alcoholic potash or soda solution, in which case, besides glycerin, potassium and sodium nitrate are formed. Hydriodic acid of 1·5 specific gravity decomposes nitro-glycerin into glycerin and nitric oxide.

The chemical examination of nitro-glycerin is conducted in the following manner:—

(1) *Percentage of Water.*—A weighed quantity of nitro-glycerin is exposed in a desiccator over calcium chloride, until constant weight is obtained.

(2) *Percentage of Nitrogen.*—This is determined in the usual way with Lunge's nitrometer, but the determination is only of theoretical interest, since pure anhydrous nitro-glycerin has practically always the same composition.

(3) *Acidity and Alkalinity.*—On superintending the working of a factory, it is desirable to have the nitro-glycerin always perfectly neutral. It is a matter of course that it should not be acid, since acid nitro-glycerin should never be used, and if acid it should be still further washed. Alkalinity, on the other hand, is also undesirable, since it gives rise to spurious results, as nitro-glycerin can be made alkaline by a considerable addition of soda solution. If the soda solution be washed out, the nitro-glycerin may still show some traces of acidity which have escaped its action, or which come from the decomposition of the nitro-glycerin by the soda. Distilled water, therefore, is poured over the sample of nitro-glycerin, well shaken up with it, and the water separated in a separating funnel. If into this water be poured a little methyl orange, or better still, congo-red solution, the coloration will at once show whether the water be alkaline or not. One or two drops of quarter-normal hydrochloric acid solution should at once convert the colour of the congo red into a deep blue. If a large quantity of soda solution be suspected in the nitro-glycerin, it can be determined by any of the well-known titration methods.

(4) *Heat Test.*—This is carried out according to the regulation given in the Appendix. Good nitro-glycerin should stand it at least ten minutes, but cases of thirty minutes or more are not at all rare.

(c) MECHANICAL PROPERTIES.

(1) INFLAMMATION AND SPONTANEOUS DECOMPOSITION.

Burning bodies ignite nitro-glycerin with difficulty only. It extinguishes a match and cools an incandescent platinum wire plunged into it, the nitro-glycerin only volatilizing. If nitro-glycerin be ignited, it burns away in layers, as is general with burning liquids, but by and by its temperature will increase, and if the whole mass be brought to too high a temperature, it may explode.

Formerly nitro-glycerin was believed to gradually undergo spontaneous decomposition, but since methods of perfectly purifying it have been devised, it has been found that under normal conditions and normal temperature no decomposition can take place; but, like gun-cotton, even perfectly pure nitro-glycerin will not stand heating to a temperature of 212° F. for more than a few hours without decomposition. At 70° C. (158° F.) traces of acidity begin to appear within ten to fifty minutes on the potassium iodide paper in the heat test, and they will continue to appear, although the sample may be taken out and cooled down and exposed to the same temperature again after some time. With larger quantities, decomposition of that kind might become dangerous. It appears that the temperature between 45° C. (113° F.) and 50° C. (122° F.) is the critical one for nitro-glycerin. At temperatures up to 45° C. it can be kept for months without its capability of standing the heat test being impaired. Between 45° C. and 50° C. this will be lowered considerably after a few weeks.

According to Miller and Warren de la Rue, decomposition is promoted by small traces of hyponitric acid, and in consequence of the oxidation glyceric and oxalic acids are formed. Liebe and List also observed the presence of oxalic acid in nitro-glycerin that had been kept for a long time. Beckerhinn, who studied the action of a mixture of ozone and oxygen on a sample of nitro-glycerin, the purity of which he had previously tested, found that oxidation took place at ordinary temperature with the formation of glyceric and nitric acids. The attacked part was coloured green, nitrous fumes and carbonic acid being at the same time developed.

On working on a large scale decomposition sometimes takes place. In most cases this happens in apparatus giving comparatively small outlets for gases, whereby so much heat is retained that an explosion may take place. If such decomposition happen in an open vessel, and in not too large quantities of nitro-glycerin, or when it is being stirred by an agitator or compressed air, the heat being gradually formed will be carried off and decomposition will take place with a copious development of nitrous fumes, but without explosion. The author has examined the residue after such decomposition, but could not find any oxalic acid in it. It consisted chiefly of sulphuric acid.

(2) *EXPLOSION.*

If a drop of nitro-glycerin be absorbed by filter paper and struck with a hammer on an anvil, it explodes, but as a rule only the part actually struck by the hammer. The explosion of a long thread of liquid is sure to take place, if the mass on which it lies be of small volume; thus, for instance, it may happen on a thin steel bar, or if a strip of tinfoil, sometimes even a strip of paper only be laid over it, because in this case the propagation of the explosive waves takes place in sufficient volume. Frozen nitro-glycerin is, in general, less sensitive to shock than the liquid. The work required to detonate liquid nitro-glycerin is 0·5 foot-pound (0·75 kilogramme-metre). If fired at with a rifle, it explodes if the distance be not greater than 165 yards (150 metres).

The safest way of detonating nitro-glycerin is to put a fulminate of mercury detonator on the top of it. Nitro-glycerin explodes when exposed to a temperature of 180° C. (356° F.).

Leygue and Champion found by gradually heating nitro-glycerin that at

185° C. it boils with the evolution of vapours,
194° C. it volatilizes slowly,
200° C. it evaporates quickly,
217° C. it detonates violently,
241° C. it explodes,
257° C. it explodes violently,
267° C. it explodes less violently,
287° C. there is a slight explosion,
dark cherry red, there is no explosion (spheroidal state).

The last result was confirmed by Kopp, who allowed a drop of nitro-glycerin to fall on a red-hot metal plate, and only obtained a slight report.

These results only obtain with quite small quantities. With larger quantities, any heating over 180° C. will, without fail, be followed by an explosion.

(3) GASES OF COMBUSTION.

The manner in which nitro-glycerin decomposes can be expressed by the equation,

$$2C_3H_5(NO_2)_3O_3 = 6CO_2 + 5H_2O + 6N + O,$$

from which the gases of combustion should contain

PER CENT.	
58·20	CO_2.
19·80	H_2O.
18·50	N.
3·50	O.
100·00	

and 1 kilogramme of nitro-glycerin should give 1135 litres of gaseous products.

According to Vuich, the temperature of combustion of nitro-glycerin is 3005° C., and the quantity of energy given off by 1 kilogramme, 6050·48 kilogramme-metres.

(4) LOWERING THE FREEZING-POINT OF NITRO-GLYCERIN.

The peculiarity possessed by nitro-glycerin of freezing at comparatively high temperatures is a great drawback to its practical use, and want of care in handling frozen nitro-glycerin has caused a great number of accidents. Many attempts have been made to get over, or at least reduce, this liability to freeze.

In 1885 Alfred Nobel and the author almost simultaneously observed that an addition of nitro-benzene reduces the freezing-point of nitro-glycerin. Continued experiments by the author showed that many other bodies of the benzene and phenol series, including the pyridine bases and pyridine salts, have the same action.

Johann Ritter von Dahmen in Vienna, and Abraham Strauss-Collin in London, patented a process in which the glycerin was mixed with three or more per cent. of nitro-benzene and then nitrated whilst nitrogen was constantly blown through it. They affirmed that from this a dynamite could be made that would stand temperatures of −40° C. without freezing. This, however, has been since shown to be incorrect.

Such additions to nitro-glycerin are of but little use. The author, for instance, found that though a mixture of nitro-glycerin with 2 per cent. of nitro-benzene would not congeal in a freezing mixture within an hour, yet when exposed to the air temperature in a hard winter in the open, it froze over-night. If, after such mixtures have been exposed to a freezing mixture, a crystal of ordinary frozen nitro-glycerin be thrown in, they rapidly solidify, and, once frozen, they can be repeatedly thawed and easily frozen again. Moreover, such additions to nitro-glycerin have proved to be impracticable, chiefly for the reason that the freezing-point is not much lowered by their use, whilst only ½ per cent. of such an addition already diminishes their explosive effect very considerably, and an addition of say 5 per cent. of nitro-benzene makes the exploding of the mixture very difficult, whilst it reduces the effect by one quarter.

3. THE APPLICATIONS OF NITRO-GLYCERIN.

About 1864 Nobel began to use nitro-glycerin on a large scale for blasting purposes. The numerous accidents which it led to during carriage and in the mines forcibly impressed both him and others of the necessity of finding some means for obviating the danger attending the manipulation of liquid nitro-glycerin.

Mowbray in North Adams, Massachusetts, first on the construction of the Hoosac Tunnel and later for general purposes, carried nitro-glycerin in a frozen state in tin cans and thawed it up before use. Since Mowbray took special care to manufacture only pure nitro-glycerin, there was not much fear of decomposition during carriage; and since frozen nitro-glycerin is in general less sensitive to concussion, and it is improbable that if a can were battered about anything could leak out, or be lost, this manner of carriage had some advantages. It is only quite recently that the use of nitro-glycerin in mines has been repressed in America.

Nobel tried to obtain the same result in a different way. He dissolved nitro-glycerin in 15 to 20 per cent. of anhydrous methylic alcohol, in which state it was fairly free from danger. Before use it had to be washed with 6 to 8 times its volume of water.

Wurz recommended mixing nitro-glycerin and a solution of zinc or calcium nitrate into an emulsion from which the nitro-glycerin could be washed out with water before it was used.

In spite of all these precautions, liquid glycerin still remained a dangerous explosive. First of all it was difficult or nearly impossible to charge bore-holes which slanted upwards, and there was always the danger of some of it being spilled. Then the nitro-glycerin found its way into all crevices and fissures in the rock and escaped explosion. The non-exploded parts continued either to burn and fouled the air, or they were hit by the pick-axe when clearing away the rock and gave rise to frequent accidents.

In consequence of the many accidents from the use of nitro-glycerin, a committee was appointed in Great Britain in 1874 to decide the question finally, which it did by entirely prohibiting the use of nitro-glycerin. Since that date it has been no longer used in Europe, and, as has been already said, it was only in America that its use continued to find favour for a longer time.

At present the chief use of nitro-glycerin is for the manufacture of dynamites on the one hand, and of smokeless powders on the other.

To a slight extent it is used medicinally, as a rule in 1 per cent. alcoholic solutions, as for instance in Switzerland and Great Britain, or in minute doses worked up into pills, as in Germany. It is especially recommended by Professor Rossbach (Berlin, 'Klinische Wochenschrift,' 1885, No. 3) as a remedy for nervous headaches, asthma, angina pectoris, neuralgia, Bright's disease, etc. He prepares for this purpose tablets containing nitro-glycerin. American physicians now recommend a dose of ½ to 1 milligramme injected into the blood as antidote in case of poisoning by carbonic oxide, or illuminating gas.

CHAPTER XII

EXPLOSIVES FROM AROMATIC HYDRO-CARBONS

By distillation a series of hydro-carbons are obtained from coal-tar, which are distinguished by an aromatic flavour, and which form valuable prime materials for manufacturing purposes. Most of these hydro-carbons can, in the same way as those of the fatty series, have one or more atoms of hydrogen replaced by a corresponding number of nitro-groups (NO_2).

In connection with explosives, it is chiefly benzene, toluene, naphthalene, phenol, and cresol that have to be considered, and the manufacture and general properties of these have been treated at length in the chapter on prime materials.

1. NITRO-BENZENE [$C_6H_5(NO_2)$].

Nitro-benzene was discovered by Mitscherlich in 1834. It is formed by treating as pure benzene as possible with a mixture of nitric and sulphuric acids. According to the strength of the acids, mono-, di-, or tri-nitro-benzene is obtained.

As a rule, an acid mixture of 40 parts of nitric acid of 1·400 specific gravity and 60 parts of sulphuric acid of 1·840 specific gravity (95 to 96 per cent. of monohydrate) is used. Sometimes the proportion of sulphuric acid is reduced, and a mixture of 41⅔ per cent. of nitric to 58⅓ per cent. of sulphuric acid, or 100 parts of nitric acid to 140 parts of sulphuric acid used. As a rule, three parts of mixed acid are taken for each part of benzene treated. On the Continent the nitrating apparatus are smaller than those used here, being generally capable of working 2 cwt. of benzene. In England the work is done on a much larger scale, and between 8 and 16 cwt. of benzene are nitrated in one apparatus.

The reaction taking place in the formation of nitro-benzene is rather violent, and on account of the volatility of benzene and also of nitro-benzene it is advisable to reduce the temperature as much as possible. For this reason the nitration is not effected by adding the hydro-carbon to the acid mixture as in making nitro-glycerin, but inversely by running the acid into the benzene. The mixed acid is made in the usual way and pumped into the storage tanks by acid elevators. Thence it is run in weighed or measured quantities into a tank or lead vessel fitted with a tap standing over the nitrating apparatus.

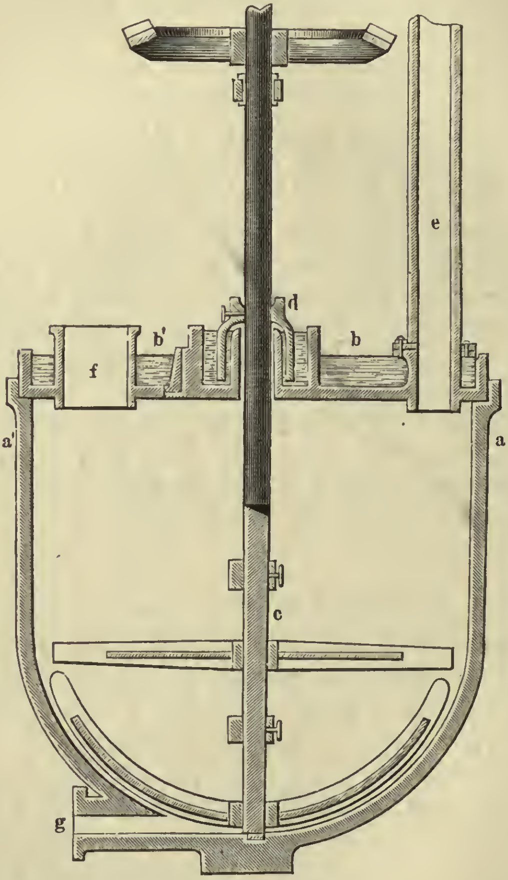

FIG. 194.

A nitrating apparatus used in continental factories is illustrated in Fig. 194. It is made of cast-iron, and has an opening (f) in the cover, through which the acid mixture is introduced. A second opening is fitted with a large pipe (e) to carry away the gases developed in the reaction. A cast-iron shaft (c), which is square at its lower end and can be revolved by a pair of cog-wheels, passes through the centre of the apparatus. It has four stirrers, the lowest of which is bent to a semi-circle corresponding to the bottom. In order to prevent the acid vapours from escaping, the shaft is fitted with a cap (d), which dips into a seal filled with nitro-benzene. The discharge branch (g) is for running off the contents of the apparatus. The acid, as a rule, is run in in a thin stream through a syphon-shaped tube, and it is advisable to have a thermometer fitted to the apparatus, so that the temperature may be observed during the reaction. The accumulation of heat during nitration is prevented by causing water to run in a continuous stream in the hollow spaces (b and b′) in the cover, whence it passes in a fine layer over the sides of the apparatus, thus effecting cooling both directly and through evaporation. The temperature during nitration is not allowed to rise above 25° C. (77° F.). The process is so regulated that the whole of the acid is run in within from eight to ten hours. As soon as this is done, the flow of cooling-water is stopped and the temperature allowed to rise to 122° F. Some factories go so far as to raise it to 212° F. The gases escaping from the tube (e) pass into a suitable absorbing apparatus.

Other nitrating apparatus have a more cylindrical shape, and are surrounded by a jacket through which water can be circulated and into which steam can, if necessary, be introduced. In England large iron cylinders of about 4 feet in diameter and about 4 feet high are used. To increase the effect of the stirring arrangement vertical ribs are fixed on the sides of the cylinder. The stirring arrangement can be set in motion or stopped at will by means of a clutch.

After the nitration is finished, the mixture is allowed to cool either in the apparatus itself, or in a tank into which it is run off and allowed to remain over-night and to separate.

The nitro-benzene floats on the top of the waste acid, the latter is first drawn off, and then the nitro-benzene. This is freed from all traces of acidity by washing with water to which a little soda has been added. If only mono-nitro-benzene be desired, and the formation of di-nitro-benzene is to be avoided, an excess of benzene is used, and this remains in the nitro-benzene. When impure benzene is used or sulphuric acid containing much lead sulphate, the mixture coming from the apparatus is viscous and dark-coloured, and some scum is separated in the waste acids; this scum, on account of its percentage of nitro-benzene, can be still utilized. The washed nitro-benzene is freed from all traces of unconverted benzene by putting it into an iron pressure vessel, and introducing a current of steam after the vessel has been tightly closed. Since nitro-benzene boils at a much higher temperature than benzene, the latter is almost instantaneously carried off by the steam and condensed in an iron cooler. The benzene which distils over is kept for a future nitration. The waste acids from the nitration of benzene contain only very small quantities of nitric acid and a little nitro-benzene, as a rule not more than 1 per cent. It is therefore not worth while to denitrate them. They may, however, be used either directly or after concentration for the manufacture of nitric acid.

When pure benzene is used, the yield is about 152 parts of nitro-benzene for each 100 parts of benzene, instead of the theoretical 157 parts. In trade several qualities of nitro-benzene are recognized, and are known under the names of "light" and "heavy" nitro-benzene. The light boils at from 205° to 210° C., and is distinguished by its purity. It is also known by the name of "Mirban oil." The heavy variety boils between 210° and 220° C., and is also called "nitro-benzene for red." It contains considerable quantities of toluene.

Nitro-benzene is either colourless or hock-coloured and boils between 205° C. and 210° C. It has a specific gravity of 1·187 at 15° C., and becomes solid at 3° C. Its flavour is absolutely the same as that of bitter almond oil, with a sweet burning taste; it is very poisonous both in the form of liquor and vapour. Its action in a gaseous state seems to be one of a strong intoxication which may increase up to unconsciousness. It is only very slightly soluble in water, but easily so in alcohol, benzene, and strong nitric acid. It will dissolve in the cold considerable quantities of nitro-cellulose with the formation of a jelly.

Nitro-benzene by itself is not an explosive under ordinary circumstances, as it cannot be exploded either by ignition or by a blow. If heated, it decomposes with the development of nitrous fumes, but nevertheless Berthelot and Vieille have proved that it can be detonated if suddenly heated by being thrown into a red-hot vessel in such quantities that it cannot materially alter the general temperature of the vessel. It burns with a strongly smoky flame. In connection with real explosive bodies, such as gun-cotton, nitro-glycerin, etc., it will detonate under the action of a fulminate of mercury detonator, but its influence is a retarding one, that is, it partly prevents explosion. It has the property of lowering the freezing-point of nitro-glycerin if added in small quantities, as before mentioned.

2. DI-NITRO-BENZENE [$C_6H_4(NO_2)_2$].

By re-nitrating nitro-benzene and by the direct action of strong nitric acid on benzene, a higher nitro-product—namely, di-nitro-benzene—is formed, in which two atoms of hydrogen of the benzene have been replaced by two nitro-groups. Its manufacture on a large scale is done in two ways: either nitro-benzene is made in a nitrating apparatus in the usual manner, and when the operation is finished the same quantity of mixed acid as at first is again added, or a mixed acid of 35 to 40 parts of nitric acid of 1·450 specific gravity and 60 parts of sulphuric acid of 1·840 specific gravity is prepared, and one part of benzene is nitrated with two parts of this mixed acid. The mixture is not cooled but allowed to become gradually heated up to 212° F. During nitration much acid vapour is formed which is caught in large condensing apparatus. When the process is finished the nitrated mixture is allowed to cool down and settle, and the waste acid separated from it in just the same way as with nitro-benzene. The di-nitro-benzene thus obtained is washed, first in cold and then in warm water. After this it is freed from water in filters, melted, and cast into cakes in sheet-iron moulds. The di-nitro-benzene obtained by this method of procedure is, as a rule, a mixture of meta-, ortho-, and para-di-nitro-benzene, but principally meta-di-nitro-benzene.

Pure di-nitro-benzene melts between 83° and 89° C., whilst the melting-point of ortho-di-nitro-benzene is 118° C., and that of para-di-nitro-benzene 172° C. Good di-nitro-benzene melts between 85° and 87° C. It is solid and hard, and crystallizes in long, brilliant prisms of a yellow colour. It should have no smell, and should not contain any nitro-benzene. Like the latter, it also is poisonous. By itself it is not an explosive, but on sudden heating it decomposes violently and under certain conditions will explode. In connection with some other bodies, especially those containing loosely bound oxygen, such as saltpetre, it forms a strong explosive. Di-nitro-benzene is soluble in warm water, alcohol, etc.

3. TRI-NITRO-BENZENE [$C_6H_3(NO_2)_3$].

By treating meta-di-nitro-benzene with nitric acid and Nordhausen sulphuric acid, tri-nitro-benzene is formed. As up till quite lately no use had been found for it, it was not manufactured on a large scale. Quite recently, however, the Chemische Fabrik of Griesheim has proposed using tri-nitro-benzene as an explosive like picric acid, and has at the same time patented a more convenient method of manufacturing it.

Tri-nitro-benzoic acid [$C_6H_2(NO_2)_3CO_2H$] is converted at 210° C., its melting-point, into carbon-dioxide and tri-nitro-benzene, but this transformation can be effected without danger and in a very simple way by heating the tri-nitro-benzoic acid with water or solvents containing water, like alcohol, soda solution, etc., to the boiling-point of these liquids. It remains to be shown what future there is for this explosive; undoubtedly it is weaker than picric acid.

4. DI-NITRO-TOLUENE [$C_6H_3(NO_2)_2CH_3 = C_7H_6(NO_2)_2$].

Substitution products can also be formed from toluene by nitration, but di-nitro-toluene alone has as yet found a practical application—namely, in the manufacture of the smokeless powder known as "Plastomenite." The manufacture of di-nitro-toluene is quite similar to that of di-nitro-benzene. It is advisable to start direct with toluene and to nitrate with strong nitric acid and highly-concentrated sulphuric acid. On nitrating, three isomeres of di-nitro-toluene are formed—ortho-, meta-, and para-di-nitro-toluene.

Di-nitro-toluene is a solid body crystallizing in needles. Its melting-point is about 71° C. It is soluble in boiling water and alcohol. Like di-nitro-benzene, it is not an explosive by itself, but it decomposes at temperatures above 300° C., and in conjunction with other real explosives it forms an explosive mixture.

5. NITRO-NAPHTHALENE [$C_{10}H_7(NO_2)$].

Naphthalene in conjunction with nitric acid forms four nitro-compounds by the substitution of nitro-groups for a corresponding number of hydrogen atoms. Nitro-naphthalene is manufactured in large cylindrical cast-iron vessels with water-jackets and agitators, similar to the nitrating apparatus used for nitro-benzene, but since naphthalene is a solid product, it is put into the acid mixture, and not *vice versâ*. For this purpose it is used either in the form of small flakes as it comes from the process of sublimation, or better still, in the form of a fine powder produced in incorporating-mills. The nitrating apparatus has usually an opening which can be closed by a cover, through which the naphthalene is introduced, whilst a gas-pipe is as usual provided. If naphthalene in flakes be used, it is put into the apparatus in small quantities through a sieve, its passage through the sieve being facilitated by pressing. At the temperature obtaining in the apparatus naphthalene again partially volatilizes and sublimes in the gas exit-pipe. The latter has, as a rule, a steam-jacket for the purpose of melting the naphthalene off again. The mixed acid generally consists of four parts of nitric acid of 1·400 specific gravity, and five parts of sulphuric acid of 1·840 specific gravity, or four parts of sulphuric acid, and a corresponding quantity of waste acid. The naphthalene is gradually introduced into the mixed acid, and the temperature so regulated that it does not fall below 160° F., the object of this being to prevent solidification of the nitro-naphthalene. The nitrated mixture is cooled in lead-lined tanks, whereby the nitro-naphthalene becomes solid. It is then melted with hot water and washed, then granulated in cold water and again washed, with constant stirring, in cold water until free from acidity.

Nitro-naphthalene crystallizes in needle-shaped crystals of yellow colour, melts at about 61° C., and is only slightly volatile when warmed or heated by steam. It is insoluble in water, but easily soluble in alcohol, ether, benzene, carbon di-sulphide, etc. It decomposes on being heated above 300° C. By itself it is not an explosive, and even in connection with oxygen carriers its use as an ingredient in the manufacture of explosives is quite exceptional.

6. DI-NITRO-NAPHTHALENE [$C_{10}H_6(NO_2)_2$].

This is made either from mono-nitro-naphthalene by treating the latter with highly-concentrated nitric acid in the cold, or by nitrating naphthalene at a temperature of about 70° C. with an acid mixture composed of one part of highly-concentrated nitric acid and two parts of sulphuric acid. Otherwise the process is the same as with the manufacture of mono-nitro-naphthalene.

In the "Ammonite" factory at Stanford-le-Hope, mono-nitro-naphthalene is first made by putting naphthalene into waste acid, and converting the product into di-nitro-naphthalene by re-nitrating with fresh acid mixture. Both operations are carried out in open enamelled cast-iron pans. The process is so regulated that no heating occurs, but this causes the operation to last from one to two days.

Di-nitro-naphthalene forms brilliant yellow needles. It melts at about 185° C., and is slightly soluble in ether and alcohol, less so in carbon di-sulphide and cold nitric acid, but easily soluble in hot xylene, benzene, acetic acid, and turpentine. Di-nitro-naphthalene crystallized from acetic acid does not melt until the temperature reaches 216° C. Di-nitro-naphthalene is chiefly used in the so-called safety explosives. Tri-nitro-naphthalene and tetra-nitro-naphthalene, which are formed by repeated nitration of di-nitro-naphthalene at higher temperatures, are at present not used in the explosives industry.

7. PICRIC ACID [$C_6H_2(NO_2)_3OH$].

Picric acid was discovered in 1788 by Hausmann, who obtained it by treating indigo with nitric acid. In 1795 Welter produced it by the action of nitric acid on silk. Later on it was found that it was produced by the reaction of nitric acid on various other bodies. Laurent, in 1843, first proved that it was tri-nitro-phenol, and that it was identical with the body produced by the reaction of nitric acid on either phenol or di-nitro-phenol. At present picric acid is manufactured exclusively from the phenol. Castellaz and Designolle originally made it by introducing slightly heated phenol into concentrated nitric acid, when a dark yellow paste was obtained, from the aqueous solution of which picric acid crystallized out. Direct nitration, however, gives too violent a reaction, and it is preferable therefore to first sulphonate the phenol—that is to say, mix it with sulphuric acid—and then to nitrate the phenol-sulphonic acid so obtained.

Formerly nitrating was also done in another manner—namely, by bringing the phenol-sulphonic acid in contact with sodium nitrate, when nitric acid was formed, which at once reacted upon the phenol. In this process sodium sulphate was obtained as a residue.

At present nitric acid is generally used for nitration, and gives a perfectly pure homogeneous product. According to the usual practice equal quantities by weight of crystallized carbolic acid and concentrated sulphuric acid are mixed together in large iron vessels, provided with a stirring gear and heated by steam to between 212° and 250° F. From time to time tests are made to determine whether a drop of the phenol-sulphonic acid is perfectly soluble in cold water. As soon as this stage is reached, it is allowed to cool and twice its quantity of water added. The nitration is done in earthenware receivers, standing in a water-bath which can be heated by steam. Into them are poured three parts of nitric acid of 1·400 specific gravity, and the phenol sulphonic acid allowed to run in gradually. In the beginning the reaction is very violent, and large quantities of hyponitric acid fumes are developed. By and by the reaction becomes slower, and finally heating is resorted to in order to completely drive off the hyponitric acid. The picric acid, which in the first stage is of a syrupy nature, separates on cooling in large crystals all caked together. These can then be either filtered, or better still, separated from the mother liquor in a centrifugal machine. They are then washed in a centrifugal machine with warm water and afterwards dissolved in hot water, again crystallized out, centrifugaled again, and finally the crystals are dried at about 95° F. If the picric acid is to be perfectly pure, it is converted into the sodium or potassium picrate (as a rule, the former, by adding soda solution), the sodium picrate precipitated is centrifugaled, washed with cold water, then dissolved in boiling water and decomposed again by an excess of sulphuric acid, when sodium bisulphate remains as a residue. The hyponitric acid developed in large quantities must be converted into nitric acid again in apparatus of sufficient dimensions. For this the author's condensing batteries in connection with Lunge's towers are well suited.

Where nitric acid is obtained as waste acid, for instance in the manufacture of nitro-glycerin and gun-cotton, it can be used instead of freshly-prepared nitric acid. The slight green coloration of such waste acids, due to a small percentage of hyponitric acid, is of no importance.

The process of formation of picric acid takes place according to the following formulæ :—

(*a*) Formation of phenol-sulphonic acid—

$$C_6H_5.OH + H_2SO_4 = C_6H_4(SO_3H)OH + H_2O.$$

(*b*) Formation of tri-nitro-phenol—

$$C_6H_4(SO_3H)OH + 3HNO_3 = C_6H_2(NO_2)_3OH + H_2SO_4 + 2H_2O.$$

Thus, on nitrating, sulphuric acid is again separated from the phenol-sulphonic acid, whilst three nitro-groups take the place of three atoms of hydrogen.

Picric acid forms long, flat, brilliant needles of a lemon colour, which melt at 122·5° C. If the heating be carefully prolonged, it sublimes without decomposition, but when suddenly heated it explodes. Although large quantities of picric acid have been ignited without exploding, yet it has been shown that under extraordinary circumstances picric acid may form very violent detonating mixtures. In 1887 a disastrous explosion of picric acid took place at the chemical works of Messrs. Roberts, Dale and Co., at Manchester, and experiments made by Dr. Dupré, and later on a larger scale by Sir Frederick Abel and Mr. W. Deering at Woolwich, showed that even very rough mixtures of picric acid and litharge or lead nitrate, strontia nitrate, etc., formed violently detonating mixtures, and that even the mere contact of litharge with burning picric acid at once produced detonation of the whole mass. Wet picric acid can be detonated by a primer of dry picric acid.

Picric acid has an extraordinarily bitter taste, and always gives an acid reaction. It is only soluble with difficulty in cold water. According to Marchand, one part of picric acid is soluble at

5° C.	in 166	parts of water
15° C.	in 86	„ „
20° C.	in 81	„ „
22½° C.	in 77	„ „
26° C.	in 73	„ „
77° C.	in 26	„ „
100° C.	in 20	„ „

It is soluble in alcohol, ether, and benzene, and gives a fast yellow colour to animal tissue such as silk, wool, the human skin, etc. According to Anschütz, picric acid can be determined quantitatively by acridine, since acridine picrate is hardly soluble in cold water.

Although picric acid does not contain a sufficient quantity of oxygen to cause complete combustion, yet it is one of the strongest explosives known, since at a high temperature or with a strong initial ignition it splits up completely. By itself, when only ignited, it burns with a strong deep black smoky flame, developing black bitter vapours, but does not explode. If picric acid in fine crystals be ignited in a closed vessel, it explodes with but little effect, similarly to black powder; but if fired by means of a strong detonator either in a compressed state or after it has been cast into a solid lump by melting, it detonates with great violence, and its force is far superior to that of the strongest dynamite or gun-cotton. A primer of dry gun-cotton has the same effect as a detonator.

Eugène Turpin was the first to discover, in 1886, the possibility of detonating picric acid by itself. Before this it was believed that on account of its deficiency of oxygen, an oxygen carrier must be added to the picric acid to make it capable of detonation. Borlinetto had suggested as far back as 1867 a mixture of 10 parts of picric acid, 10 parts of sodium nitrate, and 8½ parts of potassium chromate as a blasting powder, which could not be exploded by friction or shock. The use of picric acid mixed with potassium chlorate and various metallic oxides has also been suggested.

In or about the year 1886 picric acid mixed with collodion cotton, dissolved in ether, and formed into cartridges was made in France as an explosive, and known by the name of "Melinite." Later on Turpin showed that molten picric acid by itself explodes much more violently, and extensive experiments were then made simultaneously in France, Italy, and Great Britain, when it was found that the best method of detonating molten picric acid was the use of either very strong detonators or a primer of dry gun-cotton. The picric acid was melted at a carefully regulated temperature in an iron or earthenware vessel standing in an oil-bath. It was chiefly used for filling shells, and was cast direct from the melting-pot into the projectiles. After it had cooled, the gun-cotton primer was inserted. Picric acid so melted retained its name of "Melinite" in France, while in England it was called "Lyddite" (from the experiments having been made near Lydd).

It has since been found that picric acid in contact with the metal walls of the projectiles forms picric salts which do not possess sufficient stability, and its use has therefore been discontinued to a great extent. In its place stable salts of picric acid are used, which will be described in the following section.

8. PICRATES.

About the year 1869 Designolle, Brugère, and Abel simultaneously recommended the use of salts of picric acid for the manufacture of explosives, the potassium, sodium, and ammonium salts of the picric acid being found most suitable.

Designolle caused three kinds of powder to be made at Le Bouchet, consisting of a mixture of potassium picrate and saltpetre, with or without the addition of charcoal. Their composition according to Désortiaux was as follows:—

	Powder for Torpedoes and Shells.		Cannon Powder.			Rifle Powder.	
			Ordinary.		For large guns.		
Potassium picrate ...	55	50	16·4	9·6	9	28·6	22·9
Saltpetre	45	50	74·4	79·7	80	65·0	69·4
Charcoal	—	—	9·2	10·7	11	6·4	7·7

They were manufactured by pulverizing and mixing the ingredients together with 6 to 14 per cent. of water in stamp-mills for from three to six hours, and then granulating, sorting through sieves, polishing, and finally drying them.

The caking was done under varying pressure, according to the effect and the velocity of combustion desired. The effect of Designolle's powders was very satisfactory, and for a time they were in use in the French navy for charging torpedoes and shells. Their use has, however, been abandoned.

Fontaine made powder from potassium picrate and potassium chlorate, which gave a much greater effect than those previously mentioned. It was intended also for torpedo and shell charges, but proved to be very dangerous to manipulate.

Potassium picrate is made by dissolving picric acid in boiling water, and adding to it a hot solution of potassium carbonate. When cooled, potassium picrate separates out in golden yellow crystals, which are separated from the mother-liquors and washed.

In a similar manner, by using sodium carbonate in place of the potassium salt, sodium picrate can be made. Its properties are very similar to those of potassium picrate.

In 1869 Brugère and Abel simultaneously recommended a mixture of ammonium picrate and saltpetre as a powder. Brugère's powder consisted of 54 parts of ammonium picrate and 46 parts of saltpetre, and Abel's of 60 parts of saltpetre and 40 parts of ammonium picrate. When heated to 310° C., these powders burned with only half the velocity of black powder, but were said to have twice or three times the effect. Brugère further claimed as advantages possessed by his powder:—Small hygroscopicity, small residue, consisting of potassium carbonate only, which does not attack metals, and but little smoke, and that colourless. Experiments made with Brugère's powder in a Chassepot rifle showed that a charge of 2·6 grammes gave the same effect as the regulation charge of 5·5 grammes of black powder.

Brugère expressed the theoretical mode of decomposition of his powder by the following equation:—

$$C_6H_2(NO_2)_3(ONH_4)+2KNO_3=5CO_2+6N+6H+K_2CO_3;$$

and therefore 100 grammes of his powder should give 69·14 grammes of gas, which at 0° and 0·760 metre pressure would occupy a space of 52·05 litres. In a practical experiment he obtained 48 litres. The quantities of gas produced on the combustion of black powder, according to Bunsen and Schischkoff, and those found by Brugère, are in the proportion of 1 to 2½.

Powders made from ammonium picrate seem to be fairly stable. According to Abel, even when strongly moistened with water, no ammonium nitrate is formed. Mixtures of ammonium picrate with ammonium nitrate or potassium chlorate have also been proposed, but the former is hygroscopic and the latter dangerous to handle.

Ammonium picrate can be easily produced by saturating a hot solution of picric acid with a concentrated solution of ammonium, or by passing ammonia gas through a hot solution of picric acid. Carbonate or sulphate of ammonia may also be used. As soon as the solution is completely saturated it is allowed to cool, when ammonium picrate is obtained in the form of yellow needles, or prisms.

Picrates have in quite recent times again been carefully investigated with regard to their possible use in shells. It has been found that ammonium picrate is as suitable for filling shells as picric acid, and at the same time more stable, and that it does not give rise to the formation of dangerous kinds of picrates. A mixture of 432 parts of ammonium picrate with 568 parts of potassium nitrate was carefully investigated by Chief Engineer Lambert of the French Government. The dried and pulverized picrate was mixed with saltpetre by sifting fifty times, then moistened with 6 per cent. of water, and allowed to stand a few days. After this it was compressed like powder, broken with mallets and granulated. From experiments made in guns with this powder higher initial velocities and lower pressure than with black powder were obtained, but it was hygroscopic; yet, on the other hand, a certain percentage of moisture was necessary to avoid high gas pressures.

9. TRI-NITRO-CRESOL [$C_7H_4(NO_2)_3OH$].

Tri-nitro-cresol is similar to tri-nitro-phenol, and is made in the same manner as picric acid, but from cresol instead of phenol. It also is a yellow body crystallizing in needles, slightly soluble in cold water, more so in boiling water, alcohol, and ether, and melting at about 212° F. Tri-nitro-cresol is at present used in France under the name of "Cresylite," and also elsewhere, for filling shells and torpedoes. For this purpose it is melted like picric acid and cast into the projectiles. A primer of gun-cotton is used for detonating it.

In Austria the ammonium salt of tri-nitro-cresol is used in military blasting operations under the name of "Ecrasite." It can be made in a similar manner to ammonium picrate. The projectiles are either filled by ramming the loose explosive into them with wooden rods, or the explosive is compressed into the form of slabs. Ecrasite requires 2-gramme detonators to ensure its exploding properly. According to Austrian reports, ecrasite is of the colour of sulphur, and feels greasy to the touch; it is unaffected by moisture or differences of temperature, and does not explode by friction or blows, even from rifle-shots. It melts at 212° F., and when touched by an incandescent body or a flame, it only burns. Nevertheless, an accidental explosion happened in the laboratory of the Austrian Military Committee whilst a shell was being filled with ecrasite, the cause of which, however, has not been definitely ascertained.

The effect of ecrasite is said to be about double that of dynamite.

CHAPTER XIII

FULMINATE OF MERCURY

FULMINATE of mercury was discovered by Howard in 1799. The extremely explosive properties of this new body at first prevented an exact examination, and its composition was first settled by the famous investigations of Liebig and Gay-Lussac. Berzelius and Wöhler also investigated the composition, etc. of fulminate of mercury; and finally Kekulé determined with sufficient accuracy the rational constitution of the fulminates.

1. MANUFACTURE.

The materials required for the manufacture of mercury fulminate are—mercury, nitric acid, and alcohol. The strength of the two latter varies but little in the various processes. Howard took 5 parts of nitric acid of 1·3 specific gravity, and 10 parts of alcohol of 0·85 specific gravity, to each part of mercury. Liebig proposed that 3 parts of mercury should be dissolved in 36 parts of cold nitric acid of 1·34 specific gravity in a vessel with a capacity of at least 18 times the volume of this mixture, and to this, which contained nitrous acid in solution, 17 parts of alcohol of 80 to 85 per cent. were to be added, and the whole well shaken. In this method the reaction begins after a few minutes. A strongly refracting liquid separates and is mixed again with the rest by shaking, the mixture then blackens on account of the separation of mercury and is followed by a violent reaction, which is moderated by gradually adding a further 17 parts of alcohol. The separated mercury then disappears, and crystals of fulminate of mercury are formed and deposited in the vessel. There should then be no trace of free mercury left in the solution. The yield in this process varies between 118 and 128 per cent.

Chevalier suggested that in this process the vapours formed should be passed through several receivers and finally into a chimney.

At present a process devised by Chandelon is followed almost universally. According to his original method, one part of mercury was dissolved with slight heating in 10 parts of nitric acid of 1·4 specific gravity in a large vessel, and when the solution had attained a temperature of about 130° F., it was transferred to a flask containing 8·3 parts of alcohol of 0·83 specific gravity.

In France 3 parts of mercury (0·300 kilogramme) are dissolved in 30 parts of nitric acid of 1·383 specific gravity, and the solution poured into a large carboy containing 90 parts of 90 per cent. alcohol. Towards the end of the operation first 2·38 and then 1·58 parts of alcohol are added. The fulminate of mercury is washed for 24 hours and dried between blotting-paper. Experiments made in France show that when too large quantities of cold alcohol are added, a light white powder is obtained by reduction at the finish of the operation, which seems to be an explosive nitrate of mercury, and which impairs the quality of the fulminate of mercury.

Fig. 195.—One-50th full size.

In most factories fulminate of mercury is made by dissolving 1 part of mercury in 1 part of nitric acid of 1·380 specific gravity in a glass carboy, and then bringing the solution into a second glass carboy (A, Fig. 195), which contains 10 parts of alcohol, and stands on a ring-shaped straw mat (a). This carboy is connected with a series of receivers (C) standing in a trough (B) filled with water, the last of them being connected by a tube (D) either with a chimney or a condensing tower. After some time the reaction begins, showing itself at first by a slight development of gas only. Soon the liquid begins to boil up. White vapours of carbonic acid, nitric ether, acetic ether, etc., are given off at first, and then red vapours of nitric oxide.

After the reaction has lasted a quarter of an hour, the mercury fulminate separates out in small, slightly grey-coloured needles. The contents of the carboy are then poured on to a filter consisting of a wooden frame resting on a wooden stand and covered with cloth.

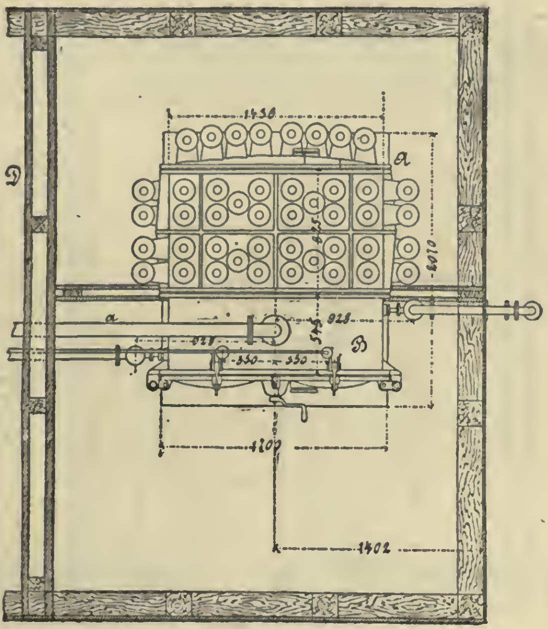

FIG. 196.

On this filter it is washed 10 or 12 times with water until it no longer reddens litmus. The filters are then placed in the open air away from direct sunlight and allowed to dry until they contain only 10 to 15 per cent. of moisture. The yield is about 125 parts of fulminate of mercury from each 100 parts of mercury. Theoretically, the yield should be 142 parts.

In the French artificers' school, according to Gody, 5 litres of alcohol of 90° on Gay-Lussac scale, or a mixture of 4 litres of alcohol and 1 litre of aldehyde obtained from the residue of the previous operation, are put into a carboy holding 40 litres. Then

0·450 kilogramme of mercury is dissolved in 1 kilogramme of nitric acid of 1·332 specific gravity in another receptacle, and 3½ kilogrammes of nitric acid are heated up to 80° C. (176° F.) in a glass carboy standing on a sand-bath. As soon as everything is ready, 3½ kilogrammes of nitric acid are first put into the carboy containing the alcohol by means of a long-necked funnel, and then the mercury solution added. The vapours given off are condensed in earthenware receivers. After 1¼ hours the reaction is finished, the solution is decanted from the mercury fulminate, and the latter well washed several times with rainwater. The yield is 0·500 kilogramme of dry fulminate of mercury, or about 111 per cent.

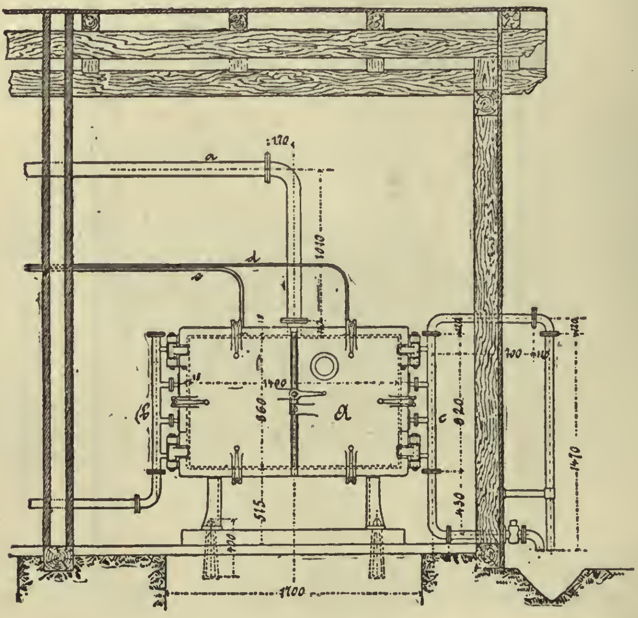

Fig. 197.

The liquid condensed in the receivers is neutralized with slaked lime and distilled off in a water-bath, whereby an ethereal liquid containing much aldehyde is obtained, which can be used again instead of alcohol. About 30 litres of this are obtained from 80 litres of the condensed products.

English experiments have shown that Chandelon's method gives a high yield and an excellent product, whilst if the materials are used in different proportions, either no fulminate at all, or only very small quantities of it are formed. For example, from 1 part of mercury, 8 parts of nitric acid of 1·37 specific gravity, and 6½ parts of alcohol of 0·896 specific gravity, no appreciable quantity of fulminate was obtained.

Stahlschmied found that fulminate of mercury was also formed when wood naphtha was used instead of alcohol. On the other hand, it was not formed by heating pure methylic alcohol with mercury nitrite and nitric acid.

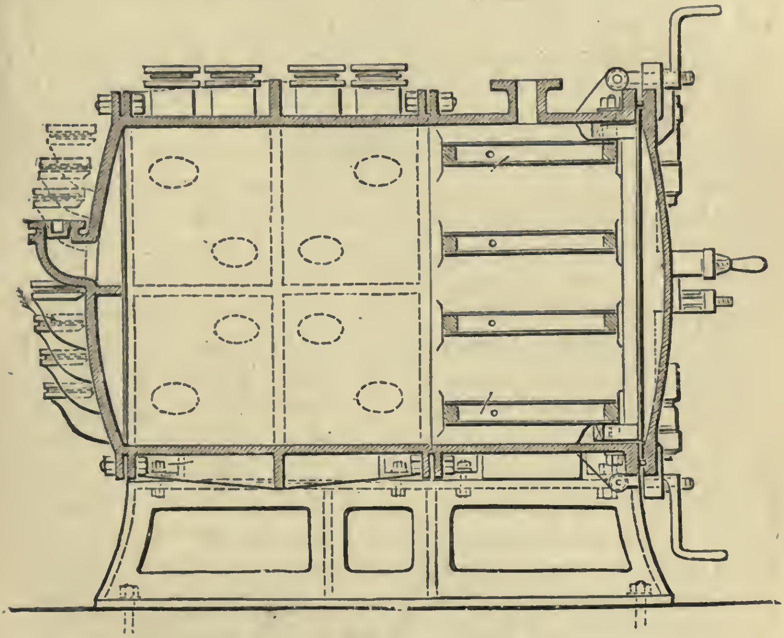

Fig. 198.

The finished product is usually wrapped up in paper parcels containing as a maximum 120 grains. It is sometimes packed in wooden boxes, but it is advisable to store it with about 15 per cent. of moisture in papier-maché boxes or entirely under water in vessels with a cloth tied over them.

If fulminate of mercury has to be dried, it is put into special drying-houses on wooden frames with string net bottoms, over which linen or paper is spread. The temperature is not allowed to rise above 104° F., and dry fulminate must be handled with great care.

Emil Passburg of Breslau has designed a vacuum apparatus for drying fulminate of mercury and similar bodies of an easily inflammable nature, which has been adopted by the royal firework laboratory at Spandau. Figs. 196, 197, and 198 show this in plan, elevation, and section respectively. It consists of a strong cast-iron casing divided into two compartments, the drying chamber proper (B) and the expansion chamber (A). There are four heating plates in the drying chamber, which can either be heated by steam or cooled by water. b is the inlet pipe for steam or water, either of which can be admitted at will by suitable taps. The discharge pipe (c) carries away the condensed water or the cooling-water, as the case may be. The fulminate to be dried is placed on the drying plates in flat trays. The drying chamber is closed by doors turning on hinges, and capable of being pressed home tightly by screws. Along the top and on the sides of the expansion chamber are forty-four large branch-pipes, on each of which a cast-iron cover is laid with an india-rubber ring between it and its seat. When in operation a vacuum of at least 28 inches is maintained in the apparatus by means of an air-pump. It is known that explosives will only burn with the greatest difficulty in a vacuous space. The idea underlying this apparatus is that only such quantities of fulminate of mercury are dried in the apparatus that in case of an explosion the gases evolved can first expand into the vacuous expansion chamber, and if this be not sufficient to prevent dangerous pressure being set up in the apparatus, the safety-valves, which are kept tight by the pressure of the atmosphere when there is a vacuum in the apparatus, will be thrown from their seats, and thus give a free exit to the gases. A tube (d) is connected with a vacuum gauge in the engine-room, and a tube (e) with an air-tap in order to admit air when the drying is finished. As soon as this has been done, steam is shut off and cold water allowed to run through the heating plate, in order to rapidly cool the fulminate. The expansion chamber is protected by a wooden wall from the drying part proper of the apparatus, so that in case of the safety-valves being thrown about, damage to the workmen is prevented. The apparatus is said to work very well and to dry rapidly, because much higher temperatures can be employed in the apparatus on account of the vacuum.

2. PROPERTIES AND CONSTITUTION.

According to the care bestowed on its production, fulminate of mercury is either grey or whitish grey, the amount of colour being due to intimately mixed particles of metallic mercury. It can be purified by dissolving in boiling distilled water, and when the liquid has become clear, by decanting and cooling. From this liquid fulminate of mercury crystallizes out in slightly yellowish-white crystals.

Fulminate of mercury crystals under the microscope are seen to be octahedra, forming frequently needles entangled together. They are almost insoluble in cold water, and 130 parts of hot water are required to dissolve 1 part. From such a solution fulminate of mercury crystallizes out in a beautiful slightly yellowish-coloured mass with a silky gloss. It has a sweetish-acid, metallic taste, and like all mercury compounds, is very poisonous.

Liebig and Gay-Lussac, who determined the composition of fulminate of mercury by analysis, considered it to be a mercuric oxide fulminate, and wrote its formula in the old notation as $2HgO.C_4N_2O_2$. But their results have been frequently questioned. Thus Berzelius considered the fulminate of mercury to be the salt of a paired acid containing mercury. Gerhardt was probably the first to suspect it of containing a nitro-group. Kekulé demonstrated with a fair amount of accuracy that fulminate of mercury should have the following rational formula (new notation), $C(NO_2)(CN)Hg$. In support of his opinion as to the constitution of fulminate of mercury, he relied on its reactions with chlorine, bromine, and sulphuretted hydrogen.

The most important property of mercury fulminate is that of exploding with extraordinary violence. By a moderate blow and by friction with hard bodies detonation takes place with a red flame. Since its decomposition into nitrogen, carbonic oxide, and mercury vapours takes place almost momentarily, the explosion is extremely violent. If mercury fulminate be heated slowly, beginning at 158° F., it explodes at 305·6° F. (152° C.); or if rapidly heated at 368·6° F. (187° C.), according to Hess, or according to Leygue and Champion, at 392° F. (200° C.). If fulminate of mercury, lying free, be ignited by a burning body, it puffs off with only a slight detonation. It detonates by an induction coil spark. The ease with which explosive decomposition can be produced varies according to circumstances. Thus the dry substance can be exploded without fail by a blow between iron plates, but less easily between iron and lead. A blow of wood upon wood is as a rule without effect. The larger the crystals the more easily they can be detonated.

Its explosibility can be reduced or even entirely prevented by moistening. If the fulminate contain from 5 to 30 per cent. of water, only those particles which are directly struck by a strong blow explode, and the decomposition is not communicated to the other parts. On the other hand, according to experiments made by Messrs. Cross and Bevan, it is quite possible for fulminate of mercury suspended in water to explode, if the whole be enclosed in a metallic wrapping and single particles struck directly.

The German factory inspectors have noted cases in which wet fulminate of mercury has exploded during manufacture. Thus, in one case explosion occurred just as the workman took the still off its stand to decant the liquid from the mercury. The still had small cracks passing from the helm to its body which probably caused it to break as it was removed, thus allowing the fulminate to fall. The height of the fall was about 2 feet. The inspectors have also reported on another case, in which a dry mixture of mercury fulminate and potassium chlorate exploded and caused another similar, but wet, mixture (containing from 15 to 17 per cent. of water) standing at about 7 feet distance to explode. Parts of the wet charge were thrown about and exploded the next day under the influence of the sun's heat, although the latter was not transmitted directly but through a lead plate. The inspectors arrived at the conclusion that drying mercury fulminate increases its explosibility, and that wet fulminate, even if entirely under water, can explode whether the heating be by blow or by friction.

Experiments on exploding fulminate by increasing the temperature or by friction show that a pure mixture consisting of two parts of mercury fulminate and one part of potassium chlorate, if no shaking take place, can be heated to 293° F. It then only decomposes gradually and becomes brown, without any explosive action taking place. When subjected, however, to vibration at the same time, the mass explodes violently. On heating to a higher temperature (311° F.) explosion takes place after some time, and the more the temperature is increased, the sooner explosion takes place.

On pressing, mercury fulminate behaves differently, according to whether it be mixed with quartz sand or with charcoal powder. A sample of pure fulminate does not explode at the highest pressure to which it has been subjected, which was calculated to be 105,000 lbs. per square inch in these experiments. Samples mixed with sand and coke powder explode at once, even under the least pressure. Similarly pure fulminate does not explode when rubbed on a smooth surface of soft lead or American cloth on wood, or when rubbed between a bronze face and cedar wood, whilst impure fulminate explodes. On being rubbed on a steel surface pure fulminate only ignites when the surface is roughened.

It has not yet been finally settled whether it is possible to explode fulminate of mercury by sympathy or not. Some affirm this to be possible, whilst others deny it. A case has been previously mentioned in which a dry mixture exploded a wet mixture standing over two yards away. In the reports of the German factory inspectors, experiments are mentioned which should prove that such ignition cannot take place at a distance, but it seems that these experiments were made with too small quantities to be convincing, because the effect from a distance will depend not only on the distance, but also very largely on the quantity exploded.

When introducing a detonating fuse made with fulminate of mercury, Hess and Dietl made experiments on the stability of fulminate of mercury. The usual determination of stability, which is based upon the liberation of nitrous acid by heating, is impossible with mercury fulminate, because on decomposing it consumes, as it were, the nitro-group in itself. Hess and Dietl therefore based their stability test upon the fact that fulminate of mercury on prolonged exposure to a temperature of from 90° to 95° C. was gradually transformed into a brownish-yellow powder of unaltered crystalline shape of its particles, which was not explosive, and even burnt with difficulty. Hess and Dietl attributed this property to a transposition of the atoms in the molecule, perhaps to an isomerization or polymerization. The test was made by exposing 0·5 gramme of loose fulminate of mercury in a test-tube on a water air bath to a temperature of from 90° to 95° C. Wet fulminate of mercury lost its power of explosion within 75½ hours; when dried in a desiccator after 81 hours; while that washed with alcohol and ether and dried by a current of air stood 97 hours. Traces of nitric acid which remained in the fulminate from the washing, or were added artificially, had the effect of making the mercury fulminate retain its explosive properties for from 14 to 22 hours longer.

Fulminate of mercury in a rarefied space behaves like gun-cotton. Heeren found that on applying a wire made incandescent by an electric current, only those parts adhering immediately to the part touched exploded, and that without flame. Parts of the mass further off were thrown about undecomposed. From these observations Ph. Neumann recommended granulating and mixing fulminate in vacuo, but it would seem that he has not succeeded in making an apparatus for doing so.

Fulminate of mercury is principally used for filling percussion caps, the manufacture of which will be described later. Fulminate of mercury cannot be used by itself as an explosive for rifles or blasting because it is too dangerous to manipulate in large quantities, and also because of its excessively sudden action. It decomposes so very nearly instantaneously that in the short time given the inertia of the projectile is not overcome, and even the strongest gun-barrels are broken. It is only in very small charges, placed together with a pellet in a metal cap, that it is used in toy guns and pistols.

The destructive action of fulminate of mercury as compared with the propulsive one of gunpowder was clearly proved by an experiment made by Sir Frederick Abel. A bomb loaded with 6½ grammes of fulminate of mercury was crushed on explosion nearly to dust, whereas a small bomb loaded with 49·7 grammes of gunpowder was broken into several large pieces which were thrown about to great distances. In the first case the broken particles remained near the point of explosion, proving that by far the largest part of the force of the explosion was utilized in overcoming the internal cohesion of the iron. Von Uchatius also found that the gas pressure set up in the interior of a gun on exploding small charges of fulminate of mercury in it was excessively high.

For these reasons fulminate of mercury is almost exclusively used as a priming material, being mixed as a rule with other burning bodies or mixtures such as potassium chlorate, black powder, or a mixture of saltpetre and sulphur, etc. The object of these admixtures is partly to retard the rate of decomposition, thus making the effect more lasting, but chiefly to increase the volume of the gases liberated on decomposition. By this the longer blow-pipe-like flame is obtained, which penetrates further into the interstices of the powder to be ignited, and thus renders its ignition more sure and complete.

The importance of intimately mixing the fulminate of mercury with the powder to be ignited, or at least of enclosing it perfectly in a certain space, is shown by a well-known fact, namely, that if the fulminate of mercury be exploded in the open air merely in contact with black powder, the latter is thrown about without being ignited.

CHAPTER XIV

FULMINATE OF SILVER AND FULMINATE OF GOLD

PARTLY on account of their composition, and partly on account of their very highly explosive properties, the fulminates of silver and gold are closely related to fulminate of mercury, and although their industrial importance is insignificant, a description of their manufacture cannot be passed over.

The compound analogous to fulminate of mercury,

$$C_2Ag_2N_2O_2 = C\,(NO_2)\,(CN)\,Ag_2,$$

is known under the name of Howard's or Brugnatelli's fulminate of silver. Gay-Lussac and Liebig made it by dissolving 1 part of silver in 20 parts of nitric acid of a specific gravity of from 1·36 to 1·38. To this solution they added 27 parts of alcohol of between 85 and 90 per cent., and heated the mixture to ebullition, after which another 27 parts of alcohol were added and the mixture allowed to cool.

Fulminate of silver can also be obtained by introducing nitrous acid (made from starch-flour and nitric acid) into an alcoholic solution of silver nitrate, and also by boiling mercuric nitrate in water, together with some silver and platinum filings.

Fulminate of silver, which is highly poisonous, crystallizes in opaque, silky, white needles which are slightly soluble in cold water, but more so in hot; 36 parts of boiling water will dissolve 1 part of fulminate of silver. The crystals are placed on a filter and washed with cold water until they no longer give an acid reaction. The filter whilst still wet is torn into strips, which are laid on more filter paper. They are then dried in a slightly warm place. The dried compound is stored in small quantities in cardboard boxes.

On account of its very high explosive properties, which surpass those of fulminate of mercury, the utmost precaution is required in its manufacture, especially in filtering and drying it. The precautions to be used follow as a matter of course from the properties of the compound. Since it explodes under a slight blow between two hard bodies even under water, it must be stirred with wooden rods instead of glass ones. The dried product can only be handled with sheets of Bristol board, and any crushing that may be necessary must be done in small quantities either between the fingers or by corks in porcelain mortars. Fulminate of silver can be heated to 130° C. without explosion taking place. It detonates with a violent piercing sound if ignited by a burning body. Concentrated sulphuric acid and chlorine cause it to explode. In vacuo, the explosive properties of fulminate of silver are, according to Heeren, considerably inferior. It only burns with a flame without exploding.

On account of its great sensitiveness to friction, its use in percussion caps is not to be thought of, and it has hitherto only been used in toy crackers and such like. In these a small quantity of fulminate of silver is put on to a strip of Bristol board, and another one with a rough surface fixed over it. The friction caused by pulling the two apart causes explosion. Throw-down crackers contain glass beads on which fulminate of silver wrapped round with blotting-paper is fixed. It has also been used for "cracker-matches."

Another compound, which has the name of "Berthollet's fulminate of silver," exploded even under the slightest pressure. Its use is therefore absolutely excluded. It is formed when newly-precipitated oxide of silver is left standing for 24 hours in an excess of concentrated ammonium hydrate. The black powder obtained is carefully dried between blotting-paper.

The so-called fulminate of gold is a compound which can be obtained in a similar way. It also is distinguished by its highly explosive properties.

CHAPTER XV

UTILIZATION OF WASTE ACIDS

From the nitration of various explosives there remains a mixture of sulphuric and nitric acids which, on account of its insufficient stability, must be dealt with as soon as possible, and which contains too great a quantity of valuable products to be thrown away.

The following may be taken as the average composition of these waste acids:—

	From Nitro-glycerin manufacture.	From Gun-cotton manufacture.	From Nitro-benzene, Nitro-naphthalene, Picric acid, etc.
Nitric acid monohydrate	10	10	1
Sulphuric acid monohydrate ...	70	80	65
Water	20	10	34
	100	100	100

The waste acid resulting from the manufacture of nitrobenzene, nitro-naphthalene, etc., contains too little nitric acid to make a separation of the two acids practically possible. It is therefore either used direct for the manufacture of nitric acid, or is at once re-concentrated. The waste acid from the manufacture of gun-cotton only contains a small quantity of gun-cotton, which, when the waste acid is used for the manufacture of nitric acid, decomposes gradually, and thus does not constitute a danger to the stills. Apart from this there are certain difficulties attending the denitration of the waste acid from gun-cotton manufacture, and as it contains comparatively little water, it is always more advantageous to use it direct for the manufacture of nitric acid than to dilute it by denitration. On account of the copious nitrous fumes from the decomposition of the gun-cotton, very efficient apparatus for condensation must be used, otherwise the nitric acid obtained will contain far too much hyponitric acid.

The waste acid from the manufacture of nitro-glycerin is always denitrated. Even after all precautions have been taken in the two separation processes, it still contains drops of nitro-glycerin, which float on the top of the waste acid. These if mixed in the stills with the nitrate may give rise to explosions when they come in contact with the walls of the stills. It is therefore inadvisable to use such waste acid directly for making nitric acid.

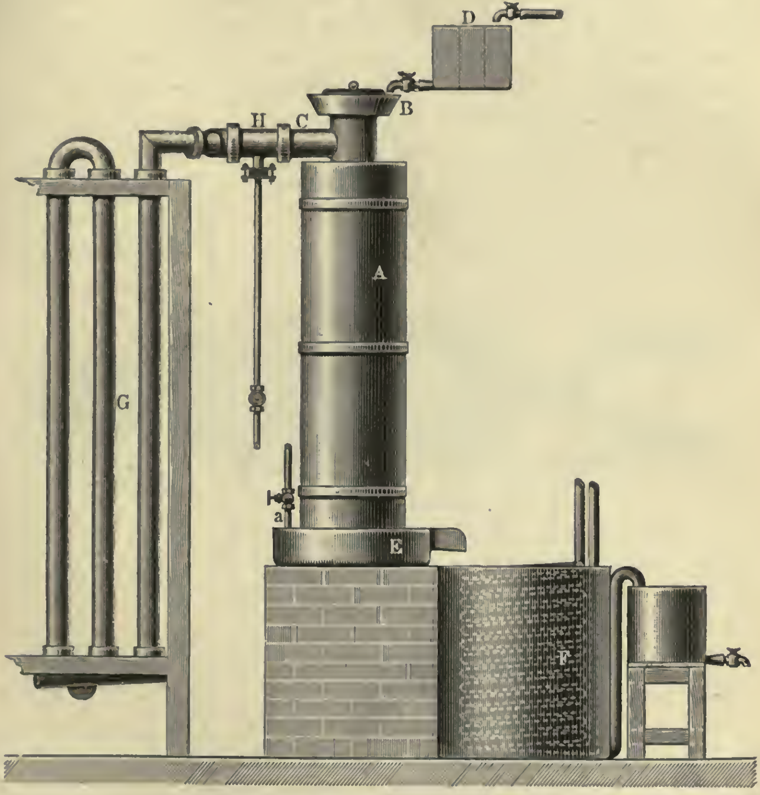

FIG. 199.—One-40th full size.

(*a*) DENITRATION.

If waste acid, which may be considered as nitrous vitriol, containing much nitric acid be heated, the nitric acid can be driven out of it. On account of the high temperature required for this, the nitric acid is transformed into hyponitric acid. The required heat can be either introduced from external sources or decomposition of the waste acid may be started by introducing steam, and the heat liberated used for driving off the nitric acid. This method is greatly assisted by the organic bodies which the waste acids always contain. Formerly denitration was done in cast-iron, still-shaped vessels which were provided with a suitable furnace for heating, and the nitrous acid issuing was condensed in earthenware receivers. Horizontal boiling drums have also been used, into which steam and compressed air were blown in order to aid the external fire in eliminating all traces ot nitric acid. The denitrating process most generally used at the present time is carried out in apparatus called denitrators. These are either high lead cylinders with a stonework lining set in tar mortar, or better still, cylinders made from a single block of acid-resisting stone, as illustrated in Figs. 199 and 200. These cylinders (A) are nearly filled with pieces of quartz, broken earthenware, and similar material presenting a large surface. A steam pipe (a) passes up into the cylinder, from below. On the top there is a feeding arrangement (B) with a water lute and a fume-pipe (C) to carry off the hyponitric acid vapours.

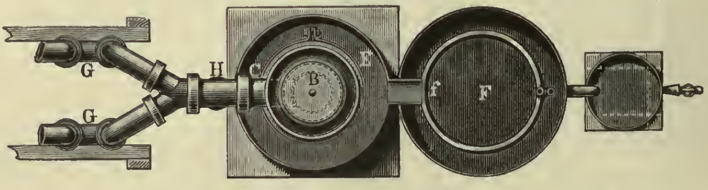

Fig. 200.—One-40th full size.

The waste acid before going into the denitrator first passes through a small separating vessel (D) in which any traces of nitroglycerin suspended in the acid are caught. The waste acid is then run in a thin stream on to the feeding arrangement. The acid as it runs slowly downwards meets a current of steam which, with the aid of the organic compounds contained in the acid soon starts decomposition. This, when once started, proceeds continually, and only requires a small jet of steam to maintain it.

The sulphuric acid freed from the nitric acid runs out below either through a discharge pipe or through an overflow arrangement (E) into a cooling vessel (F), in which it is brought to normal temperature by means of a current of cold water running through a lead coil (f). The gaseous hyponitric acid passes away through C, and is transformed into nitric acid by the action of atmospheric or compressed air, and the steam carried mechanically with it. For this purpose the Guttmann-Rohrmann battery (G) in connection with a Rohrmann injector (H) and a Lunge tower is a most suitable apparatus.

Perfect work with this process depends chiefly on giving sufficient space and time for the transformation of hyponitric into nitric acid to take place, as the air blown in cannot at once act upon the whole of the hyponitric acid, and as it also has a certain velocity, it would carry with it part of the nitric acid if the condensing arrangements were insufficient. On the other hand, it is necessary that the movement should be constantly reversed in order to obtain a thorough mixing of the hyponitric acid, steam, and air. This is effected very advantageously by the apparatus above mentioned, in which the hyponitric acid is aspired by the injector, at once mixed with air and further thoroughly mixed by the zigzag direction it is forced to follow in passing through the battery.

A double battery of 40 pipes is necessary for a denitrator capable of dealing with between 2 and 2½ tons of waste acids in 24 hours. This battery only transforms about half of the hyponitric into nitric acid. The other half passes to the Lunge tower, arriving in a state already fairly free from water. If water be allowed to run on to Lunge's tower in a very thin stream, or drop by drop only, the resulting nitric acid is not excessively dilute, and the process can be so regulated that the acid from the whole of the apparatus has a mean specific gravity of from 1·357 to 1·383. In spite of all precautions, the nitric acid so obtained still contains about ½ per cent. of hyponitric acid, and is therefore of a more or less green colour.

The sulphuric acid coming from the denitrating apparatus has, as a rule, a specific gravity of 1·635. It has a more or less dark colour, due to the organic matters in the waste acid. If the secondary separation is carefully done and the waste acid is stored for a fairly long time, it will contain but little organic matter, and the recovered sulphuric acid will only be of a slightly brown colour. With a high percentage of organic matter it is sometimes jet black. The presence of such organic matters shows itself on denitration by a strongly-marked smell of caramel.

(*b*) CONCENTRATING THE RECOVERED SULPHURIC ACID.

The recovered sulphuric acid requires careful treatment in concentrating. It contains, according to its colour, a varying

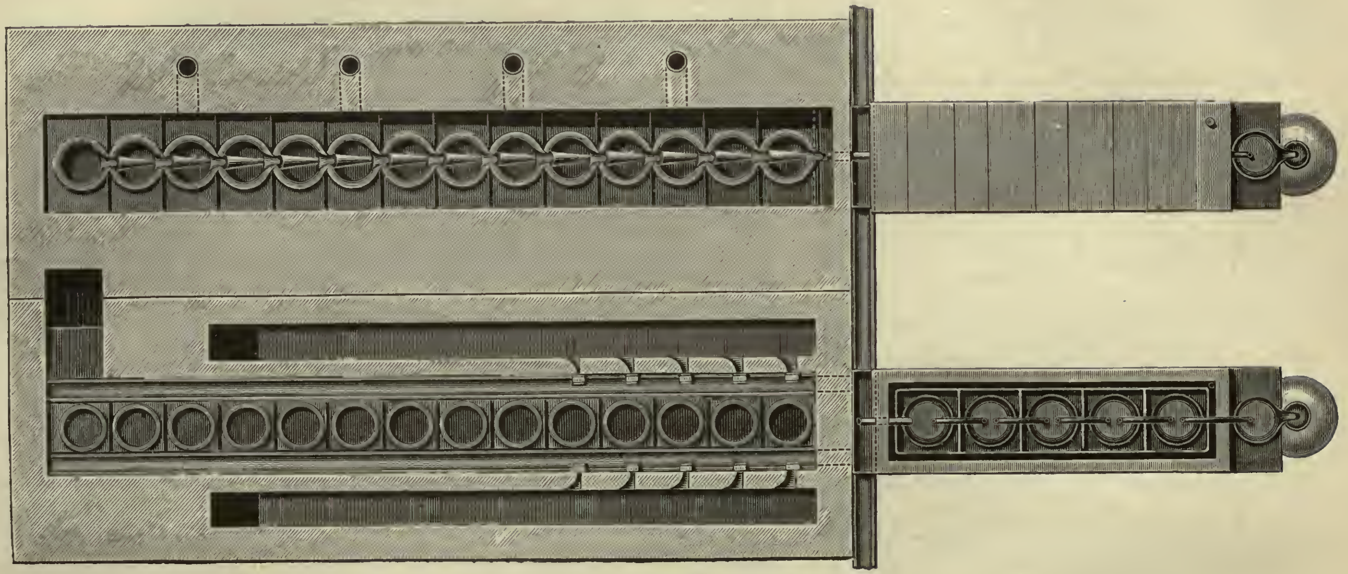

FIG. 201.—One-48th full size.

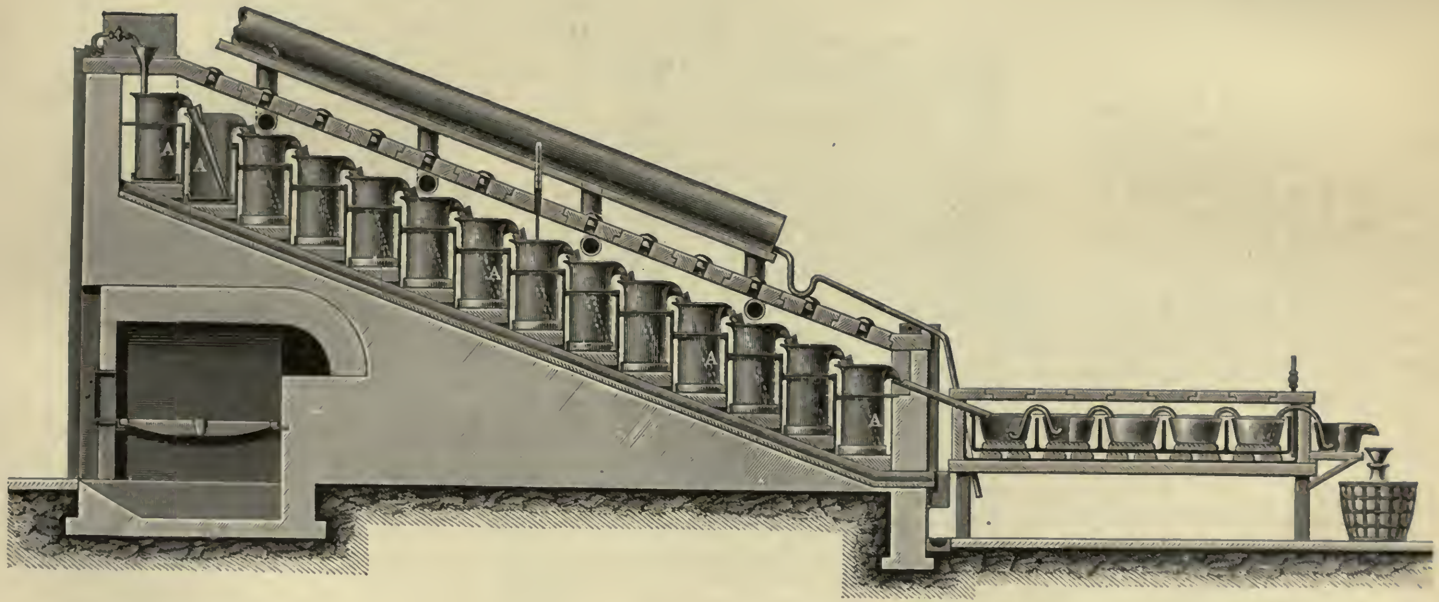

FIG. 202.—One-48th full size.

percentage of charred organic matter and other impurities, which separate on concentration and form a crust of residue in the concentrating apparatus. It will be sufficient to refer the reader to Professor Lunge's work on sulphuric acid and alkali for information in general on the concentration of sulphuric acid. On account of the possibility of traces of nitric acid being present in the recovered sulphuric acid, it is not advisable to let it run over platinum apparatus. For some time it was therefore usual to partially concentrate it in leaden pans and then to let it run into cast-iron pans provided with a hood for carrying off the fumes, or into cast-iron pans arranged on steps in a similar manner to Delplace's platinum apparatus. In these cast-iron pans the acid can be concentrated up to about 96 per cent. monohydrate; they have, however, a comparatively short life, since the covers are soon destroyed by the acid vapours, and the pans themselves crack after a comparatively short time through the crust which cakes on to them, and thus become unserviceable.

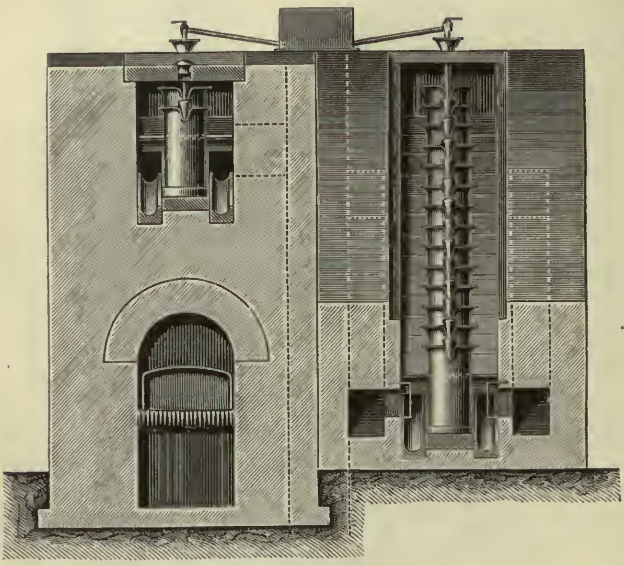

Fig. 203.—One-48th full size.

Within the last few years attempts at constructing more suitable apparatus have been made—Kessler's apparatus, Negrier's, and other similar ones for example; but concentrating beyond 95 per cent. monohydrate in these apparatus is either impossible, or cannot be effected without risk of destroying the concentrating vessels themselves.

The most advantageous concentrating apparatus is, the author believes, that of Mr. T. G. Webb in Manchester, which is illustrated in plan, longitudinal section, and cross section in Figs. 200, 201, and 202 respectively.[1] The apparatus is based upon the plan, adopted amongst others by Negrier, of arranging a number of vessels or pans in steps, and letting the acid run from one vessel into the next in an inverse direction to the current of hot gases. In Webb's apparatus every detail has been carefully attended to, so that it works in a most satisfactory manner. Briefly, it consists of a number of lipped deep porcelain vessels resting in flat cast-iron sand-baths, which in turn stand on a series of stone steps in a flue of large cross section (Fig. 203). The lower part of each pot stands by itself in a separate compartment formed by vertical and horizontal cast-iron plates (Fig. 202). The porcelain vessels or pots are so arranged that the lower part only is exposed to the action of the flue gases, whilst the top is in the main flue along which the acid vapours pass. In each vessel is a funnel-shaped tube of porcelain (Fig. 202) which is cut open on the top and the bottom to prevent cracking from unequal expansion. These tubes stand under the lips of the vessels, and thus receive the acid overflowing from one vessel and conduct it to the bottom of the next one, whence it rises and again overflows into the next tube, and so on.

The brick setting is so arranged that the hot gases from the grate pass through two flues (Fig. 201 below, 203 on the right) on to the lowest part of the apparatus. There are five openings on either side, the size of each being larger than the one behind it. Opposite each opening is a baffle-plate to prevent the hot gases striking directly on the porcelain vessels. The hot gases passing through these openings find their way round the lower parts of the vessels up into the main flue, and are conducted thence through a second flue (Fig. 201, lower part) to the chimney. The water vapours go through an earthenware or lead pipe, and as far as they have not been condensed therein, on to a washing arrangement (coke-tower, coke-box, etc.), whilst the condensed products run into a carboy through a syphon-shaped tube. The acid is

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[1] The engraver has unfortunately omitted the proper reference letters on the woodcuts, but the author believes that, together with the following description, they will be quite intelligible to all technical men.

introduced from a storage vessel on the top of the apparatus (Figs. 202 and 203), and is partly heated therein, and from thence it runs through a discharge tap and funnel into the first vessel. The concentrated acid runs through a pipe into a suitable cooling apparatus, which in this case is a number of earthenware vessels (Figs. 201 and 202) connected with each other by syphons, which stand in a lead vessel surrounded by a wooden tank. The cooling-water first runs into the wooden tank and thence into the lead vessel. The object of this double cooling jacket is that if a cooling vessel cracks, the acid runs into the inner vessel, and the outer one is not attacked. The discharge channel for the gases is covered by flagstones (Fig. 202), each of which is fitted with a stopper to allow a thermometer to be dipped into the acid; it also serves for lifting off the cover. Along each side of the main flue at the bottom is a gutter made of earthenware plates (Fig. 203) leading to a main gutter (Fig. 201), the object of which is to at once carry off any acid in case of a vessel breaking.

With this apparatus even quite black waste acid from nitroglycerin or nitro-naphthalene manufacture can be concentrated and recovered as perfectly clear acid. Experience has shown that up to a certain temperature, corresponding to the degree of impurity, the acid remains black, but as soon as it is heated above this temperature, it suddenly becomes clear, the impurities being deposited on the bottom of the vessel in the form of mud. The vessels must therefore be cleaned out from time to time. This can be done either by lifting a flagstone cover and ladling out the mud, or by stopping the whole apparatus about once in six weeks. If chamber acid or Glover-tower acid has to be concentrated, such large quantities of impurities will not occur, and the apparatus will work much longer without stoppages. There is no difficulty in obtaining sulphuric acid of over 97½ per cent. monohydrate from waste acid with this apparatus. Webb, and also Levinstein, formerly used glass instead of porcelain vessels in a similar apparatus, but they were extremely difficult to handle, and sometimes cracked by the dozen. According to the author's experience, the amount of breakage with porcelain vessels is so far quite insignificant.

The yield of acid depends upon the degree of concentration. If acid of not over 95½ per cent. monohydrate is all that is required, about two tons in 24 hours can be obtained by a double apparatus, such as the one here illustrated; when concentrating to

97½ per cent. monohydrate, the loss rises to over 25 per cent., and about 1½ tons per day is the maximum output obtainable. The loss is of course recovered as weak acid in the condensing apparatus.

The consumption of coke for acid of 97½ per cent. is about 25 per cent. by weight of the concentrated acid produced; with acid of 95½ per cent. it falls to 12 per cent., and even less.

CHAPTER XVI

DYNAMITE

UNDER the general name of Dynamite are included all mixtures of nitro-glycerin with bodies which not only completely absorb it, but also keep it absorbed under all ordinary conditions —that is to say, bodies from which the nitro-glycerin will not separate out when once absorbed if the mixture be subjected to either moderate pressure or increase of temperature, frost, or moisture of air.

Originally certain native mineral absorbing materials were used, because the object of absorbing the nitro-glycerin was in the first place to obviate the inconvenience and danger attending the charging of bore-holes with a liquid. Later on the value of a combustible absorbent was recognized, and the preparation of effective combustible absorbents was attempted by making suitable mixtures. Dynamites can, therefore, be divided into two classes, namely, those with *active* and those with *inactive* absorbents.

The first experiments with absorbing materials date from the year 1863. In Dingler's *Polytechnisches Journal* of January 1864, Mr. B. Turley of Ammeberg, a mining engineer, gave an account of one of Mr. Alfred Nobel's attempts, which consisted in filling sheet-zinc cartridge cases with ordinary gunpowder and then filling the interstitial space with nitro-glycerin. He closed the cartridges with a cork, and filled the space between them and the sides of the bore-holes with gunpowder.

In the *Scientific American* of 1866, Prof. Seeley proposed mixing nitro-glycerin with sand. Later on he also proposed mixing it with some powder which would neutralize any trace of acidity.

1. DYNAMITES WITH INACTIVE ABSORBENTS.

The first practical step towards the dynamite of the present ay, and at the same time the first use of this name for blasting material, was due to Mr. Alfred Nobel, who in 1866 found that "kieselguhr" possessed a very high absorbing capacity and at the same time the property of properly holding nitro-glycerin when once absorbed. Dynamite made with kieselguhr is used in large quantities, and is known either as kieselguhr dynamite or simply as dynamite. Its manufacture has, however, been entirely discontinued in some countries, in Austria and Italy for instance, and very much reduced in other countries.

The properties and the preliminary preparation of kieselguhr by calcining, grinding, and sifting have already been described in Vol. I. p. 110.

Kieselguhr dynamite as a rule consists of 75 parts of nitro-glycerin and 25 parts of calcined kieselguhr, with the addition of half a part of calcined soda, which is added with the idea of neutralizing any traces of acidity that may be developed during storage. For special purposes dynamite containing 80 per cent. of nitro-glycerin and 20 per cent. of a highly absorbing kind of kieselguhr has been made. Sometimes dynamite containing only 70 per cent. of nitro-glycerin is asked for. In Europe kieselguhr dynamite containing from 70 to 80 per cent. of nitro-glycerin is called dynamite No. 1; in Great Britain and the Colonies it is called dynamite or kieselguhr dynamite simply, lower grade qualities not being used; in America it is known as Giant Powder No. 1.

The colour of dynamite varies according to the kind of kieselguhr it has been made with. When white slimed guhr, burned in muffle furnaces, is used, its colour is a delicate pink. If grey kieselguhr or white guhr that has been calcined in an open flame furnace, when particles of carbon get mixed with it, be used, the dynamite will be of a dirty brown colour, sometimes with visible black specks of carbon. In order to keep a uniform colour in spite of any accidents in calcining and the use of various kinds of kieselguhr, it is usual on the Continent to add about 0·25 per cent. of burnt ochre to the dynamite.

Good dynamite is of about the consistency of fresh mould. It should not feel greasy to the touch, and a cartridge should not show any traces of nitro-glycerin on the inside of the wrapper. The outside of a cartridge when the wrapper is removed should be perfectly smooth, but not pasty, and when a cartridge is broken in two, it should have a granular fracture without traces of nitro-glycerin being visible.

One of the earliest imitations of Nobel's dynamite was one made at Eperies in Hungary under the name of "Fulgurite," the absorbent in which was magnesium carbonate. In America dynamites are still made with this absorbent.

Another dynamite of early date was the "white dynamite" of Charles Diller of St. Lambrecht, which had "lime-guhr" as the absorbent.

Both of these had the advantage of requiring no addition of soda, as the absorbent itself acted as a neutralizer, and consequently the dynamite possessed a high degree of stability under heat. The absorbing capacity of both is however low, 65 per cent. at most.

During the siege of Paris in 1870, Champion proposed making dynamite from 55 per cent. of nitro-glycerin and 45 per cent. of boghead coal ashes, as no other absorbing material was available. Later Randanite and also chalk, magnesium carbonate, and various silicates were used as absorbents.

In the powder factory of Vonges the following dynamites were made:—

	1	2	3	Special.
Nitro-glycerin	75·0	50·0	30	90
Randanite	20·8	—	—	1
Silica from Vierzon	3·8	48·0	—	—
Magnesium sub-carbonate ...	0·4	—	—	1
Chalk from Meudon	—	1·5	—	—
Red ochre	—	0·5	—	—
Silica from Launois	—	—	60	—
Blast furnace slags	—	—	4	—
Calcium carbonate	—	—	1	—
Yellow ochre	—	—	5	—
Special silica	—	—	—	8
	100	100	100	100

Many other absorbents have been tried besides the above, but kieselguhr and, more rarely, magnesium carbonate are practically the only absorbents in use at the present time. The exceptions are those bodies which are added to the absorbents for special purposes.

In 1878 Th. S. Huntley and R. W. Kessel of Cardiff proposed using 25 parts of burned gypsum as the absorbent. It was said to have the property of making the dynamite waterproof. More recently attempts have been made to make a dynamite which will not ignite fire-damp by its explosion. Besides the water cartridges proposed by Abel and Smethurst, where a paper casing filled with water is put round the dynamite cartridge, Mr. Müller, the manager of the Cologne dynamite factory, has made so-called fire-damp dynamite, which has proved very effective. He mixes 10 parts of kieselguhr dynamite containing 77 per cent. of nitro-glycerin with 7 parts of soda, and forms the mass into cartridges. The idea with this dynamite is that the soda, which possesses a large amount of water of crystallization, will at the moment of the explosion form a jacket of steam round the explosive gases. The fire-damp dynamite contains 25·90 per cent. of water of crystallization and 45·29 per cent. of nitro-glycerin; thus, as far as its protection against fire is concerned, it should be decidedly effective, and experiments have proved this to be so. On the other hand, its effect in working will of necessity be inferior, and the protection it affords is therefore not without additional cost. Similar fire-damp dynamites have also been made in which magnesium sulphate is used in place of soda.

2. DYNAMITES WITH ACTIVE ABSORBENTS.

The first typical dynamite in which nitro-glycerin was absorbed by a combustible or explosive substance was that made by the process used by Mr. Alfred Nobel, which is referred to on p. 186, in which he filled cartridges of black powder with nitro-glycerin. Later on saltpetre or wood pulp were added to dynamites with inactive absorbents, and sometimes a mixture of both, until finally the inactive absorbent was entirely abandoned and an absorbing mixture used which on explosion would decompose as completely as possible into carbonic acid and water.

The dynamites with inactive and active absorbents most largely used at present are the following:—

Kieselguhr dynamite, called "Giant Powder" in America:—

75 parts of nitro-glycerin,
25 ,, of kieselguhr,
0·5 ,, of soda.

Lithofracteur of Krebs Brothers and Co.:—

54½ parts of nitro-glycerin,
14¾ ,, of barium nitrate,
2 ,, of manganese,
2 ,, of soda,
2 ,, of wood meal,
1 ,, of bran,
7 ,, of sulphur,
16¾ ,, of kieselguhr.

Rhexite of Carl Diller:—

64 parts of nitro-glycerin,
11 ,, of decayed wood,
7 ,, of wood meal,
18 ,, of sodium nitrate.

Meganite of Wilhelm Schückher and Co.:—

60 parts of nitro-glycerin,
10 ,, of nitrated wood,
10 ,, of nitrated vegetable ivory,
20 ,, of sodium nitrate.

Dynamite of Vonges (France):—

75·0 parts of nitro-glycerin,
20·8 ,, of randanite (decayed feldspar),
3·8 ,, of quartz from Vierzon,
0·4 ,, of magnesium carbonate.

Carbonite (from Schmidt and Bichel):—

25 parts of nitro-glycerin,
40½ ,, of wood meal,
34 ,, of sodium nitrate,
½ ,, of sodium carbonate.

Stonite (from Schmidt and Bichel):—

68 parts of nitro-glycerin,
20 ,, of kieselguhr,
4 ,, of wood meal,
8 ,, of potassium nitrate.

Hercules Powder (America):—

40 parts of nitro-glycerin,
45 ,, of sodium nitrate,
11 ,, of wood-pulp,
1 ,, of sodium chloride,
1 ,, of magnesium carbonate,
2 ,, of moisture.

Vulcan Powder (America):—

30·0 parts of nitro-glycerin,
52·5 ,, of sodium nitrate,
7·0 ,, of sulphur,
10·5 ,, of charcoal.

Safety Nitro-Powder (America):—

68·81 parts of nitro-glycerin,
18·35 ,, of sodium nitrate,
12·84 ,, of wood-pulp.

Judson Powder (America):—

5 parts of nitro-glycerin,
64 „ of sodium nitrate,
16 „ of sulphur,
15 „ of cannel coal.

Atlas Powder (America):—

75 parts of nitro-glycerin,
2 „ of sodium nitrate,
21 „ of wood fibre,
2 „ of magnesium carbonate.

Vigorite (America):—

30 parts of nitro-glycerin,
49 „ of potassium chlorate,
7 „ of potassium nitrate,
9 „ of wood pulp,
5 „ of magnesium carbonate, moisture, etc.

Pulverulent Ammonium-Dynamite:—

20 parts of nitro-glycerin,
25 „ of ammonium nitrate,
36 „ of sodium nitrate,
18½ „ of roasted rye flour.

Dynamite No. 3 for giant mines:—

15 parts of nitro-glycerin,
85 „ of absorbing powder consisting of—
  84·5 parts of sodium nitrate,
  15·0 „ of coal,
  0·5 „ of sodium carbonate.

Carbo-Dynamite of Reid and Borland:—

90 parts of nitro-glycerin,
10 „ of charcoal made from cork.

It is said that the latter absorbent gives the carbo-dynamite the property of absorbing even over 90 per cent. of nitro-glycerin, which it will retain perfectly absorbed even if kept under water for a prolonged time. The inventors intended making weaker dynamites by adding water in place of some of the nitro-glycerin, as the charcoal will retain both perfectly absorbed. In experiments made at Treherbert in Wales, carbo-dynamite gave very good results, with almost no noxious gases. At the same time the water added is said to make it a safety dynamite that will not ignite fire-damp. Carbo-dynamite, however, does not appear to have been extensively used as yet.

As in former chapters, the author does not intend to describe or even enumerate all the varieties of dynamite with active absorbents, which have been invented and tried at various times. Their number is very large, and all materials in any way suitable have been used as absorbents. Those desirous of fuller information on them will find it in the late Colonel Cundill's 'Dictionary of Explosives,' or in Désortiaux's, a revised translation of it. At present their only interest is historical.

3. MANUFACTURE OF DYNAMITE.

(*a*) Mixing.

The nitro-glycerin is brought in weighed quantities into the mixing-house in buckets made either of hard gutta-percha or lacquered compressed wood pulp. The calcined kieselguhr or other absorbent is brought in in tight bags or cases. Frequently nitro-glycerin is not carried about the factory in a liquid state, but is at once poured over the weighed quantity of absorbent and superficially mixed in the store-house, so that a more or less thick paste has to be carried, and there is no danger of nitro-glycerin being spilt on the way. Where this is not advisable, it is of advantage to have a special tram-line from the store to the mixing-house, and to have suitable trolleys for carrying a number of the nitro-glycerin buckets. The trolleys should be divided into compartments by wooden bars, each compartment holding one bucket fairly tight, and so fixed that it cannot shake about. It is best to entirely line the trolleys on the inside with sheet-zinc, so that if any nitro-glycerin is spilt it can be easily taken up with a sponge.

The nitro-glycerin and absorbing material are mixed by hand. As the workmen get so accustomed to handling nitro-glycerin, it is superfluous to take the precaution of wearing india-rubber gloves. These are uncomfortable and hot, and so do not find favour with the workmen themselves. They also have the disadvantage of rendering it very difficult for the workman to feel when the mixing is perfect, and inferior work of course is the result.

The process of mixing requires a man with some practice, otherwise some parts of a charge will contain very little or no nitro-glycerin, and others too much.

After this hand-mixing the dynamite is rubbed through wire sieves. For this purpose large troughs about the height of a table and lined with thin sheet-lead or sometimes only ordinary wooden cases are used, but on account of the difficulty in keeping the latter clean they cannot be recommended. A wooden frame to which a brass wire gauze is fixed at the bottom is put over these troughs. Sometimes two sieves are used instead of one; a coarse one with about three meshes per linear inch, and a finer one with about seven meshes per linear inch. Rubbing through the coarser sieve mixes the mass more intimately than the hand alone, whilst the action of the finer one is a better distribution of the nitro-glycerin in the mass. This operation is done by hand, the loose dynamite being put on to the sieve with a wooden spatula and pressed through with the palm of the hand. It can also be done with india-rubber gloves; but, as previously said, their use is not popular with the men.

After rubbing through the sieve the dynamite should be in the form of fine grains. It must be neither too greasy nor too dry, and the judging of this can only be done by an expert. If the mass be too dry, it can only be worked with great difficulty in the cartridge press, and if too damp, the dynamite smears and the nitro-glycerin soon sweats out. Dynamite that is too moist can be improved by the addition of a little absorbent, or if too dry by the addition of a little nitro-glycerin. Kieselguhr dynamite that is too dry can be improved by repeatedly rubbing it through sieves. It appears that the fine capillary tubes of the kieselguhr are again broken by more frequent rubbing, or that the nitro-glycerin adhering to the particles of kieselguhr is made to exude.

The loose dynamite is taken from the mixing-house to the cartridge huts in small charges, either in india-rubber bags, or, better still, in wooden boxes lined with sheet-zinc or merely varnished.

(*b*) FORMING CARTRIDGES.

In order to facilitate the charging of bore-holes, dynamite is made up into the shape of cylinders. The diameter of these cylinders, or cartridges as they are commonly called, should be rather smaller than that of the bore-holes, and since as a rule bore-holes are rarely made with drills of over 1¼ inches, the largest diameter of cartridges in the trade is 1 inch, while those with a diameter of ⅞ and ¾ of an inch are most generally used. From its physical nature a dynamite cylinder requires some protection, and it is therefore wrapped in waterproof paper, as a rule in parchment or paraffined paper.

The dynamite cylinder is usually made by pressing. There are two kinds of presses—those which press the whole of the cartridge into the ready-made case at one stroke, which are known as intermittent presses, and those which make a continuous thick cord of dynamite, which is broken off from time to time to the required size and wrapped in paper. These are known as continuous presses. An intermittent cartridge press, which was formerly used, is illustrated in Fig. 204. It consists of a funnel (a) which

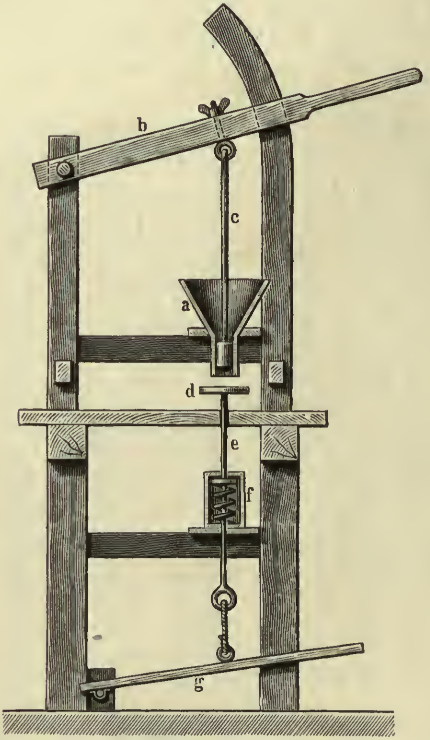


Fig. 204.—One-20th full size.

takes up the loose dynamite and a lever (b) hinged at one end to the frame of the press. A pressing bar (c) is hung to the shorter arm of this lever, of a slightly smaller diameter than the tube of the funnel. The cartridge paper is wrapped round the tube of the funnel, which has a length corresponding to that of the cartridge to be made. The cartridge paper is then closed below the tube by bending it round, and a small plate (d) which is connected by means of a bar (e) with a spiral spring (f) is brought up against it.

The latter constantly presses the plate against the funnel, and the case enclosing the spiral is connected with a foot lever (g) by means of another bar. When the funnel is filled with dynamite and a cartridge paper wrapped round the tube, the dynamite falls into the funnel tube, partly automatically and partly by being aided with a short wooden stick. As the lever is brought down the pressing bar enters the funnel and presses the dynamite contained in the tube into the cartridge case. At the same time the plate held upwards by the spring presents a certain resistance, and the dynamite is therefore compressed to the proper degree. The plate gives way under the pressure of the lever, this yielding being assisted by the workman moving the foot lever downwards.

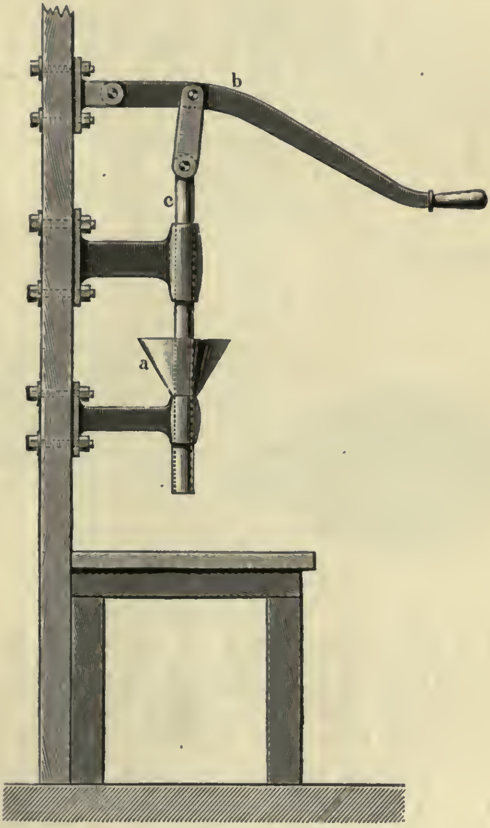
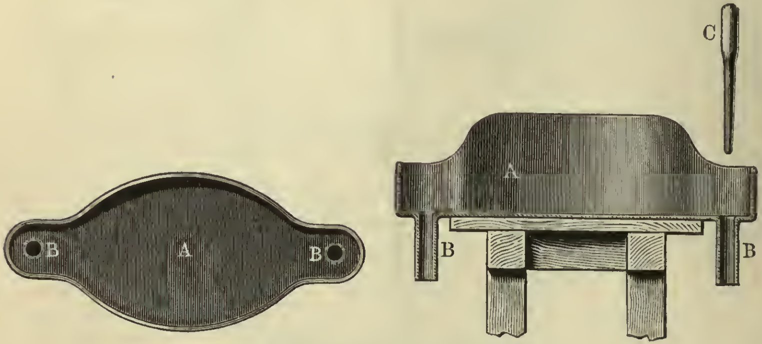


Fig. 205.—One-20th full size.

Another intermittent press which can be understood from the drawing without further explanation is illustrated in Fig. 205. This kind of press is still used in some German factories. There is no pressure acting against the cartridge. The workman simply holds the cartridge paper with his hands, and the whole mass is at once pressed into the wrapper, which is then closed on the top, also by hand.

Amongst the intermittent cartridge presses belongs the apparatus for the manufacture of cartridges from pulverulent dynamite, which is illustrated in Figs. 206 and 207. It consists of a trough of sheet-lead (A) which is slightly narrowed at each opposite end, where there is a tube (B). The dynamite is put into the trough, and the cartridge paper wrapped round the tube and held by the workman with his left hand. In his right hand he has a long pointed piece of wood (C), with which he rakes the dynamite into the tube and presses it by a few dextrous strokes, thus forming the cartridge. This apparatus is only used with dynamites which on account of their pulverulent state would cause too much friction in an ordinary press, and could not therefore be pressed through it. Sometimes, though rarely, these pulverulent dynamites are formed into cartridges in screw presses similar to those which will be described later in connection with gelatine dynamite.



Figs. 206 and 207.—One-20th full size.

A continuous press constructed by the author for the manufacture of dynamite is illustrated in Fig. 208. It consists of a strong wall-plate (a) cast in one piece with three arms (b). Each of these arms has a stout projection (c). The two upper ones are perforated and a round or square bar (d) goes through them, the upper end of which is screwed. A cylindrical box (e) is fixed to the top boss, and over it there is a second cylinder (f) closed at the top. These two enclose a spiral spring, and the one slides neatly within the other. The outer cylinder is attached by nuts to the screwed end of d. Below the upper box there is an adjustable stop (h) fixed with a screw; the upper part of this stop is slightly curved so as to work smoothly against the lever (i). The latter is carried by a link (k) hung from the back plate, and where the bar passes through it, it is thicker and provided with a slot. It will be clear that as the lever is pulled down the spiral spring is compressed, and the upper box is moved downwards, and that when the lever is released, the spiral spring lifts the bar

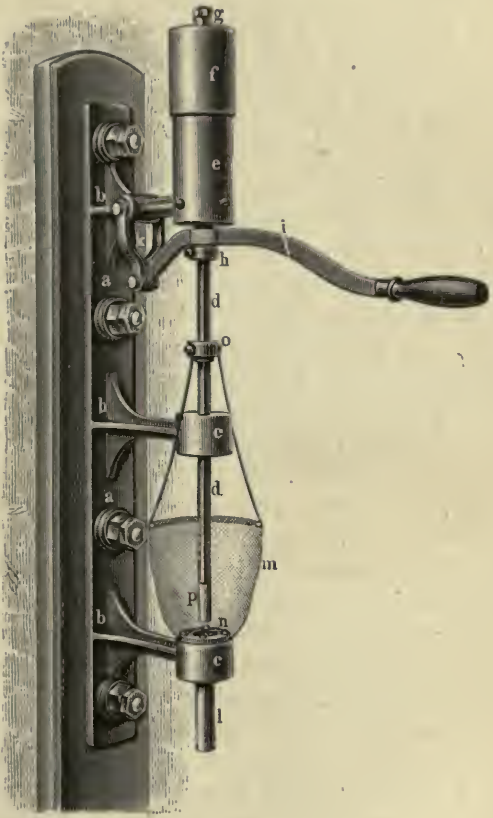


Fig. 208.

again. The second boss acts merely as a guide. The hole through the lowest arm is screwed to take the upper end of a tube (l). A cloth bag (m) is fixed by means of a washer (n) to the top of the lowest boss, whilst the upper part of the bag itself is attached to the pressing bar by means of straps and a ring (o), so that at each down-stroke of the lever the bag folds inwards, and with each up-stroke is pulled straight, with the result that the dynamite which is contained in it is constantly shaken about and so falls into the pressing tube. A piston (p) of lignum vitæ or ivory is screwed on to the lower end of the press bar. This piston is slightly tapered, and its upper end, which is the larger, is of slightly smaller diameter than the bore of the pressing tube. The workman standing in front of the press gives a succession of short quick down-strokes, allowing the spring to lift the pressing bar between each. The workman actuates the press as above by a rather peculiar rising and falling movement, which he gives to his body from the feet. This enables him to work by the full weight of his body instead of by the muscles of the arm. If the dynamite be not too dry, it will issue from the pressing tube in the form of a continuous cylinder. The workman only draws the bar down about three-quarters of an inch at each stroke, so that the pressure and the friction of the dynamite in the tube is not excessive. The mass not being exposed to much vibration, the compressed dynamite has sufficient cohesion to support a length of some 5 or 6 inches hanging free from the end of the tube. As soon as it is about 4 inches long, the workman breaks it off by holding the last two fingers of the hand under the cartridge and pressing sideways with the other three in a peculiar manner. The cylinder of dynamite thus broken off is at once wrapped in its paper covering by hand. As a rule, the work is so arranged that one workman presses while two others, one to the right and one to the left, alternately break off the cartridges and wrap them in their papers. Should the cartridge break off from any cause before the workman can reach it, the piece falls into the store-box for loose dynamite, which stands below the press.

The advantages of continuous presses over intermittent ones are that their output is greater, while they are considerably less dangerous. With intermittent presses explosions are frequent, whereas with continuous presses they can only occur through the greatest negligence. With intermittent presses a layer of dynamite 4 inches deep is tightly jammed in a tube, and to force it out at one stroke requires considerable pressure. Should a foreign particle by any accident get into the dynamite, or even particles of the absorbent which have been burned too hard, or should the pressing piston not be properly guided vertically, considerable friction, and consequently heating, takes place against the side of the tube and may cause explosion. On the other hand, with continuous presses a thin layer of the dynamite only, not more than ½ or ¾ inch, is pressed out at a time. The pressure required is therefore not so large, and above all does not last so long, so that there is no time for dangerous heating to take place.

(*c*) Packing.

The wrappers of the cartridges should be closed so as to be as waterproof as possible, in order that when subsequently used they may withstand the action of moisture as long as possible. Water, as is known, is always used in drilling bore-holes, and these are not always properly dried afterwards.

Originally dynamite was always wrapped in parchment paper, and in Great Britain and the Colonies this mode of packing is still generally used. On the Continent, packing in paraffin-waxed paper, which is cheaper, is adopted. Parchment paper gives protection for some time against the influence of mere damp, but very little against moisture. The result is that on account of the osmotic action of parchment paper nitro-glycerin will ooze out from dynamite packed in it, after being for a short period in water, and be replaced by water. Parchment paper should be carefully freed from acid due to the manufacture and should not be too stiff. This can, as a rule, be avoided by adding a little glycerin to the last washing-water. On the other hand, it must not be too soft, or else the cartridge will lose its shape and not keep its cylindrical form.

Paraffined paper is as a rule made in the dynamite factories themselves. A suitable apparatus for making it is illustrated in Fig. 209. It consists of a cast or wrought-iron pan (a), in which two iron rollers (b) are arranged in seats (c), so that they can be easily lifted. In the pan is a lead coil (d) through which exhaust steam circulates. To the ceiling and on the floor of the room, or on suitable frames, iron rolls (e) are fixed at convenient distances apart. The paper in a single length is rolled on a revolving drum (f), and from this it passes under the two rolls in the pan and to a roll near the floor and another on the ceiling in a zigzag manner, and finally through a pair of rolls (g, g_1) fixed on a table. By turning a handle (h) which is fixed on one of the rolls, the whole mechanism is put in motion. On the table a lever cutting knife is fixed in a hinge (i).

Into the pan either soft paraffin or a mixture of 20 parts of paraffin wax, 60 parts of resin, and 30 parts of tallow are introduced. This is chosen so that after cooling the coating on the paper shall not be too hard, but sufficiently pliable and not crack. The paraffin wax is melted by the heat from the waste steam. As the paper passes through the melted wax, it takes up, partly by absorption and partly by adhesion, a thin layer of paraffin, which as it passes over the rolls is cooled by the surrounding air, and can be used at once. On this table a baffle (k) is fixed, the position depending on the length of sheets required. As soon as the paper butts against k, the lever knife is brought down, and the sheet cut. Sometimes the paper is not cut at once, but passes from the last roll straight to a printing press which has a printing roll with the trade-marks cast on it at suitable distances apart, so that the paper is printed at the same time. It can then be cut to the required sizes. As a rule, parchment paper is purchased already cut from the makers. The shape of the cartridge paper is a square with a piece cut off obliquely at one end, so that one side is about 1¼ inches shorter than the opposite one. This enables the ends of the cartridges to be more easily closed. Formerly three sizes of cartridges were made—namely, for 4, 2, and 1 inch lengths; at present only 4-inch and 1-inch cartridges are as a rule made. The latter are known as primers.

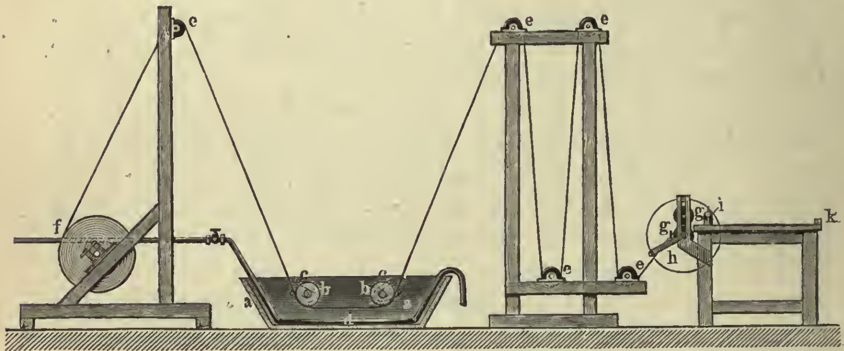


FIG. 209.—One-50th full size.

Every 5 lbs. (on the Continent 2½ kilogrammes) of cartridges are put into cardboard boxes, the weight being regulated by putting in more or less primers. The cardboard boxes are generally made in the factory itself. They are simple boxes made of straw board in the usual manner by scoring and cutting, the ends of which are closed by a simple wire fastener, or by means of resin-glue. A loose piece of cardboard is used as the cover, or the shape of the cardboard for the box is so chosen that, on folding it, pieces remain to act as the cover. In each of the cardboard boxes printed slips are put, containing instructions and advice on the use of dynamite and precautions to be observed in manipulating it. Each box is then wrapped either in waterproof or in ordinary paper. The waterproof paper is made either by soaking paper in oil or painting it over with an asphalt solution.

With very hygroscopic dynamites a somewhat porous wrapping paper is used, and after the box has been tied with a string, it is dipped two or three times into melted paraffin wax, so that the whole box is coated. Sometimes the cardboard box is even closed by pasting on paper strips, and then dipping it in paraffin, and in order to make them easy to open, a special arrangement is provided consisting of a piece of twine laid under the paper strap, which on drawing tears open the latter.

Ten boxes, thus altogether 50 lbs. (25 kilogrammes on the Continent), are put into a wooden case, with which in Great Britain and Switzerland only brass or zinc nails may be used. In Austria wooden pins only must be used. In Great Britain dynamite must be so packed that either the case or the boxes, or both together, are waterproof, and therefore, as a rule, a waterproof lining of india-rubber cloth is put into the case. Ten cardboard boxes are put into it, and the edges of the covering flap of the lining are smeared over with a solution of india-rubber in benzene, thus cementing down the cover. The cases are then marked, either by branding or stencilling, on the cover with the name of the manufacturer, and on the ends with the number of the case and the diameter of the cartridges.

For military purposes dynamite is enclosed in tin boxes with a small tubular socket on the cover for inserting the detonator. For subaqueous blasting similar tin boxes are sometimes used, and sometimes sausage skins or bags made of india-rubber cloth.

The packing of cartridges first into the boxes and then in cases is, as a rule, done in special houses, and the nailing down of the lid is sometimes done in a separate room isolated from the packing-house.

4. PROPERTIES OF DYNAMITE.

(*a*) PHYSICAL PROPERTIES.

Kieselguhr dynamite is usually orange-yellow or reddish-brown, or when coloured with ochre, a dark red mass without odour, and plastic like fresh mould. With other kinds the colour of the dynamite is of course different.

The density of kieselguhr dynamite, which in this paragraph is alone referred to, is when in cartridges 1·6; that of dynamite Nos. 2 and 3 (with active absorbents) between 1·2 and 1·3.

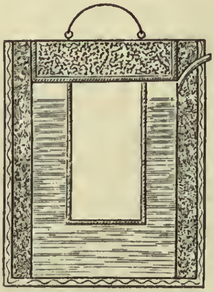


FIG. 210.

Dynamite has most of the physical properties of nitro-glycerin, and is equally poisonous. It freezes at temperatures below 46° F. into a hard mass, the outer surface of the cartridge becoming whitish, on account of the nitro-glycerin contracting and leaving the outer layer of kieselguhr. The consistency of the cartridge consequently becomes slightly loose, and as a matter of fact it expands a little. When a cartridge is thawed before being used, as it has to be, the nitro-glycerin would not distribute itself again properly through the absorbent, but would partly ooze out, had not this originally been considered in making the dynamite.

Thawing is done in special warming-pans, one of which is illustrated in Fig. 210. They consist of double sheet-zinc vessels, in the space between which warm water of 160° F. at most is put. The outer vessel and the cover are lagged with non-conducting material to prevent loss of heat.

(b) CHEMICAL PROPERTIES.

Dynamite being a mechanical mixture, its chemical properties are of necessity dependent on its components.

The chemical examination of dynamite is best done according to the directions given by Col. Hess in the 'Mittheilungen des k. k. technischen und administrativen Militär Comité's,' of 1881, which are as follows:—

(1) *QUALITATIVE EXAMINATION.*

Finely-divided dynamite is treated with ether free from water, by which nitro-glycerin and any paraffin, sulphur, or resin which may be present are dissolved. The ether is driven off from the extract on a water-bath at about 30° C. If a drop of the oil remaining be absorbed with a piece of blotting-paper, placed on an anvil and struck by a hammer, explosion will take place, if the oil be nitro-glycerin. On dissolving the oil in alcohol, and adding to it a solution of ammonium sulphide in alcohol, polysulphide is first formed, and then sulphur separates. If a drop of the oil is put into a solution of iron sulphate to which hydrochloric acid has been added, and heated, the nitric oxide reaction is obtained. If a drop of the nitro-glycerin be put into a solution of potassium iodide and starch paste, to which some dilute sulphuric acid and zinc shavings have been added, the liquid turns blue. If the oily residue be evidently uniform, its specific gravity about 1·6, and there is no peculiar smell, it may be assumed that it consists of nitro-glycerin only. If impure ether have been used its impurities will be found in the residue. If more than traces of sulphur, paraffin, or resin be present, they separate from the nitro-glycerin. Sulphur deposits in crystals and its identity is established by its smell and flame when ignited.

If the extract, after having been freed from ether, be treated with cold alcohol, paraffin remains. Any resin present can be saponified by boiling it with soda solution, and then isolated from the soap by precipitating with hydrochloric acid. If all three bodies be present at the same time in the dynamite, the nitro-glycerin is first decanted and the residue carefully pressed between blotting-paper. By boiling with soda solution the resin is then first separated and the residue washed with water and dried. A part of it is treated with aqua regia to transform any sulphur present into sulphuric acid, which is determined as barium sulphate; another part is boiled with ammonium sulphide solution, whereby sulphur dissolves forming ammonium polysulphide, whilst the paraffin floats on the liquid after cooling in the form of a crust; it can then be washed with water, dried, and reserved for further examination.

The residue from the extraction with ether is then first examined under a microscope, when kieselguhr, wood pulp, and charcoal can be recognized by their structure and coloration. Lignite and coal are identified by the nature of their products on dry distillation. Lignite yields acetic acid or ammonium acetate. If the residue be calcined, any mineral substances present will be found side by side with the wood or coal ashes, and can be determined by the ordinary analytical means.

(2) *QUANTITATIVE EXAMINATION.*

A weighed quantity of nitro-glycerin is extracted in a Soxhlet's extraction apparatus, if there be no other materials than nitro-glycerin in the dynamite that the ether will extract. For this purpose a weighed quantity of dynamite is put over calcium chloride in a desiccator until constant weight is obtained. The use of sulphuric acid desiccators is not advisable on account of possible accidents from spilling. Drying in the desiccator is best effected in the small thistle-funnel used for the extraction.

Soxhlet's apparatus is illustrated in Fig. 211. It consists of a glass flask (B), into which a glass tube passes through a cork. A funnel (A) is fused on to this tube, which takes the small thistle funnel into which the dynamite is put; it is closed at its upper end with a perforated cork which serves for connecting a Liebig cooler (C). From the bottom of the funnel (A), which is closed below, a glass tube (b) fused to the outlet tube branches off and is bent into a loop. Another wider tube (a) is connected with the top of the funnel and the outlet pipe. The flask is put on a water-bath, and the whole carried on a strong iron stand. The water is kept at a temperature of about 50° C. and the flask filled to about one-half with ether, which must be free from water. The ether as it evaporates rises into the funnel and is cooled in the Liebig condenser, whence it drops down on to the dynamite in the thistle funnel and extracts the nitro-glycerin.

As soon as the extract reaches the level of the highest point of the loop, the latter acts like a syphon, and draws the whole contents of the funnel suddenly into the flask. This operation is repeated about ten times, with fresh ether each time, until it is judged that the whole of the nitro-glycerin has been extracted.

The disadvantage of this method is that if the flask has not a very long neck, and the apparatus is not well protected from radiant heat, the ether in the funnel evaporates, and also that the nitro-glycerin evaporates from the extract itself in small traces, and therefore the percentage of nitro-glycerin found is always less than

FIG. 211.

it ought to be. It is preferable, therefore, to put the dynamite in a covered funnel on to a dried and weighed linen filter, and to extract it by frequent infusions of ether; but in this case also, care has to be taken that the ether does not rise on the sides of the vessel, and thus cause a loss of nitro-glycerin.

The ether-extract is collected in a small weighed beaker and freed from ether on a water-bath at a low temperature. During evaporating there is a moment when the hitherto clear liquid begins to become milky on account of partial separation of nitro-glycerin from the ether. From this moment the operation has to be carefully watched until the milkiness again disappears. The beaker is then at once taken off the water-bath. The nitro-glycerin left now contains only small traces of ether and possibly of acetic ether. In order to free it from these and from any moisture adhering, it is placed over calcium chloride under the receiver of an air-pump. The nitro-glycerin may then be considered as pure and weighed as soon as all smell of ether, acetic ether, etc., has vanished, or as soon as after the first exhaustion there is no appreciable difference of weight between two weighings.

If, in addition to nitro-glycerin, paraffin, resin, sulphur, etc. be present in the dynamite, the extract, after having been freed from ether and dried, is weighed and heated in soda solution on a water-bath. This dissolves any resin which can be separated from the rest by decanting and washing with distilled water. The resin is precipitated from the solution by hydrochloric acid, collected on a weighed filter, dried at 100° C., and washed, dried, and again weighed.

The nitro-glycerin in the residue from the previous operation is dissolved in strong alcohol and decanted, and the remaining mixture of the paraffin and sulphur washed with strong alcohol, dried and weighed. In order to separate the sulphur from the paraffin, the mixture is heated in an aqueous solution of ammonium sulphide, cooled down, the layer of paraffin perforated, and the liquid below decanted. The paraffin is then washed with water, dried and weighed. The quantity of nitro-glycerin and sulphur is found by difference.

The residue from the extraction, as far as it consists of fire-resisting materials only, is dried in a dry current of air at 60° C. and weighed, then calcined, and after being cooled, again weighed, in order to find by the difference whether any small quantities of organic matters were contained in the residue. If it contain other components as well, the residue is first dried on a filter and weighed, then treated with hot distilled water and the solution evaporated, again dried at 120° C., and the residue weighed. If carbonates be present, the extract, if it do not contain chlorates, is again dissolved in water, neutralized with nitric acid, evaporated on a water-bath, dried at 120° C., and again weighed. In this way the carbonates are transformed into nitrates without altering any extracted organic matter present, and from the difference of the last two weighings the quantity of carbonic acid is calculated. The dry mass is then calcined, and when cool moistened with nitric acid, freed from the free nitric acid, again calcined, and finally weighed after cooling. All salts which can be extracted by water will then be present as nitrates, whilst all extracted organic matters will have been destroyed. The difference between the last two weighings therefore gives the percentage of extracted organic matter.

If the watery extract contain chlorates as well, the carbonic acid must be determined as calcium carbonate by precipitating with calcium nitrate, and in part of it, by evaporating and calcining, the chloric acid must be determined as silver chloride. The quantity of nitric acid is found in a second part of the extract by transforming it into ammonia. In the residue from the extraction with water, only charcoal, or similar organic matters, and insoluble absorbents can be present. The organic matter is eliminated by calcining, and the rest weighed after cooling, whereby the weight of the insoluble mineral matter is obtained. Should several mineral absorbents be present at the same time, they can be determined singly from the residue by the usual methods. If several organic absorbents be present, their proportion can only be approximately estimated by an elementary organic analysis.

It has frequently been proposed to make a direct determination of the nitrogen in dynamite, and to calculate from it the percentage of nitro-glycerin. This can be easily done with kieselguhr dynamite, and similar ones containing only a mineral absorbent, but with these only, because, as previously mentioned, the percentage of nitrogen in pure nitro-glycerin is always the same.

(3) *HEAT TEST.*

This is carried out in the manner indicated in the Appendix. Dynamite should stand it at least fifteen minutes.

(4) *EXUDATION TEST.*

For prolonged storage and transporting in warm climates, it is of great importance that dynamite should be examined with regard to its capacity for retaining the nitro-glycerin permanently absorbed. In this country the Government regulations require that dynamite shall not exude any nitro-glycerin after it has been frozen and thawed three successive times; also that no nitro-glycerin shall exude from the dynamite after exposure to a temperature between 85° and 90° F. for six consecutive days and nights.

(5) *EXTRACTING NITRO-GLYCERIN.*

If, for some reason, whether to determine nitrogen or for experimental purposes, pure nitro-glycerin be required, it can be obtained in the simplest and quickest manner from ordinary kieselguhr dynamite by putting a ball of glass wool into the point of a glass funnel and pressing on to it fairly tightly some loose dynamite. On to this distilled water is poured, and the whole allowed to stand for some time over a beaker. After about half-an-hour the water begins to displace the nitro-glycerin from the dynamite, and the nitro-glycerin runs into the beaker. Since it has passed through the kieselguhr, it is at the same time perfectly clear. It will also contain no water if the beaker be taken away in time.

(*c*) Mechanical Properties.

(1) *SPONTANEOUS DECOMPOSITION.*

Formerly dynamite as well as nitro-glycerin was believed to be liable to spontaneous decomposition, and most marvellous statements have been made, such as that by the mere violent slamming of doors dynamite could explode which by some process of decomposition had been brought to a highly sensitive state.

Even at the present time there is still a State regulation in Austria which requires that a piece of litmus paper shall be packed in each box of dynamite, and that the cartridges themselves shall be tested from time to time for acidity by means of litmus paper. Now-a-days manufacturers no longer think of working up acid, or imperfectly washed, nitro-glycerin, and experience has shown that dynamite made with pure nitro-glycerin can be stored not only in the climate of Europe, but also in tropical ones, for an indefinite time without fear of decomposition. It is quite conceivable that if irrational absorbents—such as chlorates, for instance—be used, the stability of the dynamite is less certain, but in this country the use of such materials is prohibited by the terms of licence, and will probably be restricted, or entirely forbidden, elsewhere in the future.

(2) *THE ACTION OF HEAT ON DYNAMITE.*

Kieselguhr- and most other dynamites can generally be ignited by an incandescent body or by flame, and will then burn away more or less quickly, without exploding. H. M. Inspectors of Explosives and the Austrian Technical and Administrative Military Committee have made many experiments in this direction. Whole cases have been firmly bound up and ignited by a fuse. The dynamite merely burned away, the gas developed breaking open the cover. Bolley, Pestalozzi, and Kundt of Zurich have also made similar experiments. Half a cartridge of dynamite when put on a piece of sheet-iron and exposed to fire simply ignited and burned away without explosion.

This absence of danger in dynamite is however only relative. It only obtains for small quantities of carefully made dynamite and under ordinary circumstances of handling. As soon as larger quantities of dynamite are ignited—as for instance in the 10 cwts. experiments of H. M. Inspectors of Explosives in a light wood shed—some cases may burn away fiercely, but the rest of the contents of the building are gradually brought to a sufficiently high temperature to explode the whole mass at once. It can also be supposed with perfect certainty that if a dynamite cartridge be put on a hot or incandescent metal plate, it will explode within a very short time, because by doing this it is very quickly heated above its temperature of explosion. With over-saturated dynamite a peculiar action takes place. If a long roll of dynamite containing more nitro-glycerin than the absorbent can properly hold, be put on to a plank and ignited at one end, it will, as a rule, burn away quietly. If a second one be put on to the plank, part of it will burn away but the rest will explode, and the same will take place with all following charges. Whether this can be ascribed to an altered state of vibration of the air, or whether over-saturated dynamite can be more easily brought to a critical temperature, has not yet been settled. At any rate, a general warning not to heat or burn dynamite, either in large or in small quantities, must be given, because danger of explosion is not quite excluded.

Dr. Auguste Dupré has informed the author that a very simple and effective means of destroying large quantities of dynamite or other explosive is to lay it in small quantities in the form of long rolls and pour paraffin oil over them. If they are then ignited, the combustion will take place without danger. The author has used this method with great advantage.

Dynamite cannot be exposed for a long time to a comparatively high temperature without losing nitro-glycerin. Exposed on an uncovered watch-glass to a temperature of 70° C., nitro-glycerin evaporates completely, according to Hess, within 100 hours, but the author found that even at 40° C. considerable quantities (10 per cent. and more) can be driven off in a few days.

If slowly heated, dynamite explodes at 180° C., or if rapidly heated at 230° C. Dynamite containing saltpetre, or absorbents of the nature of blasting powder, will sometimes explode at lower temperatures.

(3) *THE EFFECT OF SHOCK.*

According to experiments made by Colonel Hess, dynamite detonates under a blow of 0·75 kilogramme-metre, if struck between steel and steel. In the frozen state the work required is 1 kilogramme-metre. The explosion is considerably promoted by the nature of the base on which it rests, and the implements producing the blow. According to the experiments of Bolley, Pestalozzi, and Kundt, previously mentioned, dynamite explodes between iron and iron, iron and another metal, between stone and stone more rarely, and not at all between wood and wood. When so treated, only the part directly struck as a rule explodes, but if the base consist of iron or steel, and the mass be not too large in proportion to the blow, and if at the same time a sufficient propagation of the explosive wave be possible, the whole is sure to explode. Use is made of this property, especially in military engineering, in destroying iron girders by tying either cartridges on both sides or on the ends, if not too long, of a girder, when only the charge on one side is exploded by a detonator, the second one exploding by sympathy. This action can be demonstrated in a small way by putting a dynamite cartridge into each end of a pipe, when, if one of them be detonated, the other explodes by sympathy. In this case both the metal of the pipe and the air in the bore serve as the medium for propagating the explosion.

Dynamite cartridges, and still more whole cases of dynamite, will stand very rough treatment under ordinary circumstances, because the mass is soft enough to absorb a blow of considerable force without harm. Dynamite packed in cases has been dropped down in quarries from heights of 100 and 140 feet. It has also been tied to the buffers of railway trucks which were then rolled down an incline against stops without more happening to the cartridges than their being crushed to a shapeless mass. Bullets fired from a rifle through packets of dynamite only explode them at distances below 60 paces.

Frozen dynamite cannot be exploded in the ordinary manner, and even very strong detonators are not sufficient, but it can be done with great certainty if gun-cotton impregnated with some nitro-glycerin be used as a primer. Frozen dynamite is also fairly insensitive to a blow, but on breaking or crushing it, caution is necessary (*vide* nitro-glycerin, p. 142). Rifle-shots only explode frozen dynamite at distances below 50 paces.

(4) *THE ACTION OF LIGHT AND ELECTRICITY.*

If dynamite be exposed to heat for a prolonged time, especially to the direct rays of the sun concentrated at one spot by some suitable means, as, for instance, by faults in glass windows or by reflection from highly-polished curved surfaces, it may undergo chemical transformation in the same way as nitro-glycerin, and if the disturbing cause be proportionately higher, explosion may ensue. Dynamite seems to be fairly insensitive to the action of static electricity. Currents of high tension—for instance, the discharge from a Leyden jar—only throw the particles about; but if a wet twine be inserted and sparks of an induction apparatus passed along it, the dynamite is ignited. Anyhow, it has been ascertained that a strong electric discharge, a flash of lightning for instance, can explode dynamite, and magazines containing dynamite have repeatedly been blown up through being struck by lightning. This subject will be more fully dealt with later on.

(5) *THE ACTION OF WATER AND MOISTURE.*

If dynamite be immersed in water for a considerable time, or if it be allowed to remain in a wet bore-hole, an osmotic action, which is assisted by the parchment paper wrapping of the cartridges, takes place.

If this action be allowed to continue, water from the outside will entirely replace the nitro-glycerin. As previously mentioned, this property is made use of for obtaining small quantities of nitro-glycerin. In mines it is a disadvantage, because the nitro-glycerin oozing out filters into the fissures of the rock, and may later on give rise to accidents. For subaqueous blasting, for instance, in rivers, under the sea, etc., on account of this property dynamite is, as a rule, used in tins or india-rubber bags.

In moist air well-made dynamite will take up but little water, about ¼ per cent. per day at most, and no nitro-glycerin should exude. Dynamites with active absorbents are affected to a greater extent, and the absorption of moisture will sometimes amount to several per cents. of the whole.

5. GUN-COTTON DYNAMITE.

About the year 1867 both Mr. Trauzl and Sir Frederick Abel proposed mixing nitro-glycerin with pulped gun-cotton. Trauzl's gun-cotton dynamite consisted of 73 parts of nitro-glycerin, 25 parts of gun-cotton, and 2 parts of charcoal. After making it was immersed in water for four days, during which time it absorbed 15 per cent. of moisture, but could still be exploded by a strong detonator.

Abel's gun-cotton dynamite, which he called "Glyoxiline," consisted of gun-cotton and saltpetre soaked in nitro-glycerin. Both kinds of dynamite behaved well in general, and had an effect corresponding to their composition; but when the gelatine dynamites were invented, they went out of use entirely.

In 1868 Schultze recommended soaking his gunpowder (wood nitro-cellulose) in nitro-glycerin, and he called the resulting explosive "Dualine," but it was not used for long.

CHAPTER XVII

BLASTING GELATINE AND GELATINE DYNAMITE

In 1875 Mr. Alfred Nobel patented a process according to which, by mixing that variety of gun-cotton known in the trade as collodion cotton, or soluble gun-cotton, with warm nitro-glycerin, he dissolved the collodion cotton in the nitro-glycerin, and obtained a jelly- or rubber-like mass, which he called "Blasting Gelatine."

By making a thin, more syrup-like jelly, and mixing it with various absorbing powders, he also made the so-called gelatine dynamite, the characteristics of which approached more nearly to those of kieselguhr dynamite, and which had a less violent action than pure blasting gelatine, so that it was more adapted to the requirements of practical work.

Great though the difficulties were in introducing this explosive in the beginning, it has nevertheless completely replaced kieselguhr dynamite in some countries, and nearly so in most others. At the present time it is probably the most suitable explosive in use for almost all blasting operations.

1. BLASTING GELATINE.

(*a*) MANUFACTURE.

The composition of blasting gelatine remained practically the same as that originally used by Nobel. It is only the relative proportion of the two ingredients that is slightly varied in order to meet the different regulations obtaining in various countries as to plasticity and absence of exudation. In Austria, where blasting gelatine was first experimented with on a large scale, the normal composition is 93 per cent. of nitro-glycerin and 7 per cent. of gun-cotton, and these proportions have since been adopted in Germany. In Switzerland 91 parts of nitro-glycerin and 9 parts of collodion cotton are taken, together with 1 part of soda. In Italy 90 parts of nitro-glycerin to 8 parts of collodion cotton are used, 2 parts of methylic alcohol being added to promote solution. Until about 1886 all attempts at manufacturing blasting gelatine which would satisfy all the requirements of the inspectors of explosives failed. The conditions they insisted on its fulfilling as regards heat test, liquefaction, and exudation of nitro-glycerin when subjected to a high temperature and freezing, were so stringent that it was impossible with the knowledge then available to make a blasting gelatine that would fulfil them. Increased experience, however, now enables factories to produce blasting gelatine which not only fulfils all the conditions imposed in England, but also without trouble the far more stringent ones obtaining in Australia. The proportions used in Great Britain are between 90 and 91 per cent. of nitro-glycerin, and between 9 and 10 per cent. of soluble gun-cotton.

The collodion cotton used in the manufacture of blasting gelatine should be completely soluble in ether-alcohol, and should have as high a percentage of nitrogen as possible. These conditions are not easily fulfilled under all circumstances, and the manufacture of a really suitable gun-cotton is carried to perfection in only a few factories. The most suitable soluble gun-cotton is that consisting exclusively of tetra- and penta-nitro-cellulose.

Chardonnet examined the various kinds of gun-cotton under the microscope with polarized light, and found appreciable differences in the colours given by the different varieties. He found that the colour given by tetra- and penta-nitro-cellulose, and also by a mixture of both, is materially different from that given by higher or lower nitrated celluloses, but it would seem that in order to appreciate the distinction exactly, more than ordinary familiarity with the method is required.

The collodion cotton used should be as dry as possible. Now-a-days, when this elementary condition of gelatinization is so well known to every manufacturer, it seems hard to realize that, even in the famous dynamite factory of the Nobel Company at Ardeer, it was formerly believed that nitro-cellulose coming fresh from the centrifugal machines—that is, containing about 30 per cent. of water—could be used for the manufacture of blasting gelatine; on the other hand, it will be seen later, in connection with smokeless powder, that singularly enough a large quantity of water in working a mixture of gelatine does not give rise to any difficulty.

In most factories, especially in those where the daily production is small, blasting gelatine is made in trough-shaped or square boxes of sheet copper or lead, surrounded by a water-jacket. These boxes are heated by continually circulating water at about 122° F. through the jackets. Formerly temperatures from 158° and even to 176° were used, but on account of an accident the German factory inspectors have insisted upon the temperature not exceeding 122° F., and it has, in fact, been proved that a higher temperature is not required.

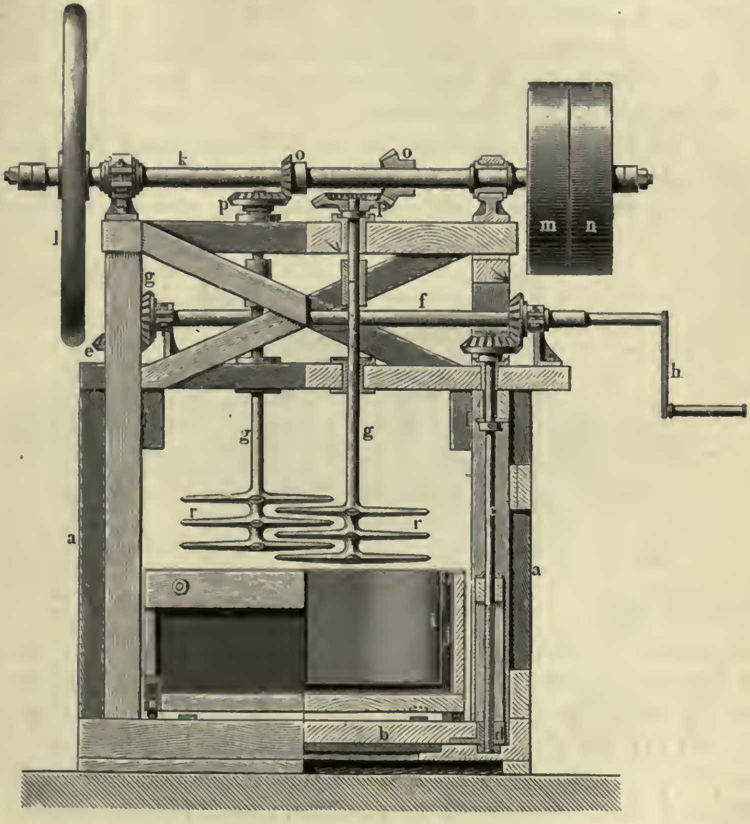


Fig. 212.—One-20th full size.

In most factories on the Continent the mixing of the gelatine is still done by hand. The nitro-glycerin stands in the troughs until it is heated to 122° F., which takes from half to one hour, the collodion cotton is then added, and the mixture stirred with a wooden spade until the mass is thoroughly mixed. The gelatine is allowed to remain at the above temperature, and is only stirred from time to time. After two hours it is kneaded by hand like bread-dough until it assumes a uniform ointment-like consistency. After cooling it forms a more or less thick jelly.

In larger factories, special kneading machines are used which make a more perfect gelatine, and at the same time can deal with very large quantities. Mr. George McRoberts, in a paper read before the Society of Chemical Industry in London in May 1890, gave a description of the apparatus used at Ardeer, which is illustrated in Figs. 212 and 213.

It consists of an oak frame (a) in which a platform (b) can be lifted or lowered by means of two screws (c). The latter work in nuts (d) fixed on the side of the platform, and are continued upwards with unscrewed shafts on which cog-wheels (e) are fixed. A horizontal shaft (f) carried on the top of the apparatus is geared to the shafts by means of two cog-wheels (g). By turning the handle (h) the platform can therefore be lifted or lowered. A copper pan surrounded by a water-jacket (j), with four wheels attached to it, is run on to the platform, and the jacket connected by an india-rubber pipe with the warm-water supply pipe. The platform and pan are then lifted by turning the handle (h) until the stirrers dip into the liquid, when the former are revolved. The temperature of the mixture is kept at between 104° and 113° F., and the whole operation is finished within an hour.

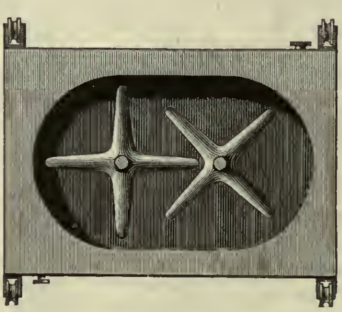


Fig. 213.—One-20th full size.

The machine has so far proved well adapted for the work, but there is always the possibility of dangerous breakages. In general, therefore, the kneading machines of Werner and

Pfleiderer are preferred for making blasting gelatine and smokeless powder. They are fully described in connection with the latter on p. 240.

The finished blasting gelatine is worked into cartridges, but it is not possible to work such a tough mass in the presses used in the manufacture of kieselguhr dynamite, and therefore other means had to be found. Now-a-days the cartridges are generally made by an archimedean screw machine, such as is used in brick-, sausage-, and chocolate-factories, for instance.

The machine used at Ardeer, according to McRoberts' paper, is illustrated in Fig. 214. It consists of a funnel-shaped case (a), with a filling funnel (b) and a nozzle (c) corresponding in size to the diameter of the cartridge to be made. Inside and concentric with the case is a shaft (e) with a screw blade (f) on it, this shaft being carried on two bearings (d) outside the casing. When the shaft is rotated, the screw blade gradually forces the mass introduced through the funnel out through the nozzle in the form of a long cylinder. On the interior of the casing there are a number of grooves (g) which prevent the mass from simply turning round with the screw.

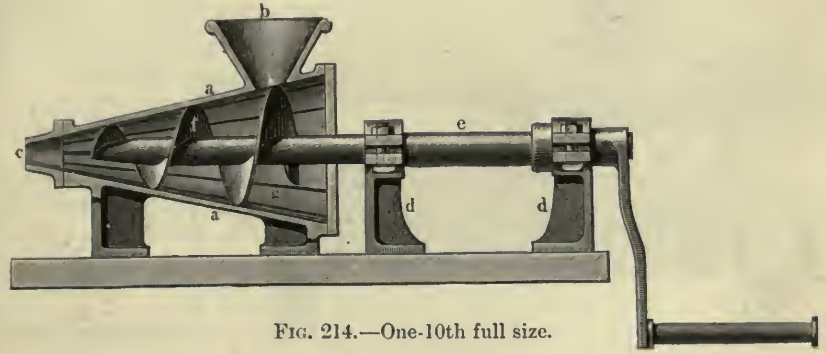


Fig. 214.—One-10th full size.

Similar machines, but with a cylindrical casing and screw blade (Fig. 215), are used in almost all the other factories. As a rule they have a cap (c) with two or more nozzles (d), so that several cylinders of the material are pressed out at the same time. The shaft (f) and with it the screw (g) can be rotated in the two bearings (k) and the cover (e) by means of the handle (l). Two collars (h), fixed by means of wing nuts (i), keep the shaft in its place. The whole is mounted on a base plate (m), and this in turn on a table. The cylindrical presses do not subject the gelatine to such a high pressure as the conical ones, and a special construction of the screws usually adopted makes them draw in the entering mass more easily.

The cylinder of gelatine issuing from the presses is cut, either with a single-bladed bronze knife by the eye, or with a double bladed one with the two blades spaced apart, according to the length of the cartridge to be cut off. The cut pieces are wrapped in parchment or paraffin paper, and then packed into the usual boxes and cases in the same manner as ordinary dynamite.

In order to entirely avoid heating in the manufacture of blasting gelatine, the Deutsche Sprengstoff-Actien-Gesellschaft of Hamburg patented a process, according to which they dissolved some picric acid in the nitro-glycerin and mixed collodion cotton with this by frequent stirring. By this process pure tetra-nitro-cellulose is said to dissolve completely in two days. This process has not yet been adopted in practice, since picric acid must always give an acid reaction to the mixture, and this is against the regulations at present in force. Besides this, there is no need for making gelatine by a cold process, and the few disadvantages of the manufacture by the hot process are far outweighed by the much quicker rate of work it allows.

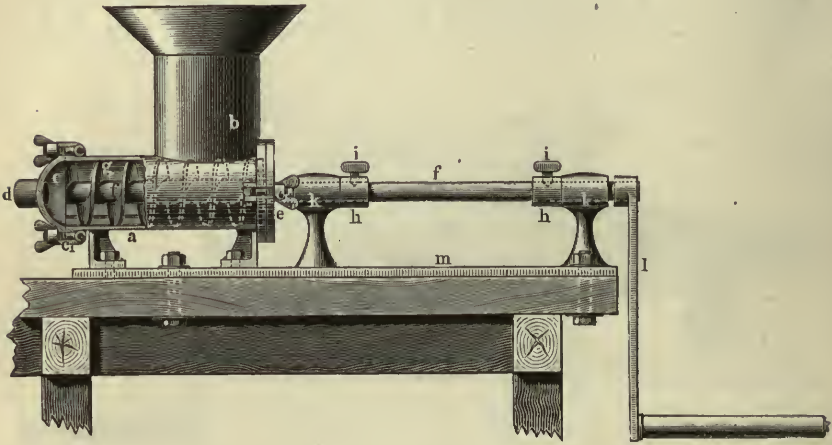


FIG. 215.—One-8th full size.

Although blasting gelatine has proved to be less sensitive to shocks than dynamite, the Austrian military authorities have nevertheless tried to devise a military explosive which should be practically insensitive to mechanical effects, especially rifle-shots for short ranges. The researches carried out by Siersch and Roth in connection with Trauzl and Colonel Hess have finally led to the addition of camphor to the nitro-glycerin, as is done in the manufacture of celluloid. It dissolves the nitro-glycerin and gives an extraordinary degree of safety to the blasting gelatine. The normal composition of this product, which was called "War Blasting Gelatine," was fixed in Austria as follows:—

96 per cent. of gelatine consisting of { 90 per cent. nitro-glycerin
{ 10 „ „ soluble gun-cotton
4 „ „ of camphor

In Italy, where it is also used, it is made of 100 parts of gelatine, consisting of 92 parts of nitro-glycerin and 8 parts of soluble gun-cotton, to which 5 parts of camphor are added. The use of this war blasting gelatine was discontinued in Austria three years ago, and ecrasite adopted in its place. In order to explode the camphorated blasting gelatine, the use of a special primer was necessary, which ensured a more violent detonation on account of its giving a larger number of vibrations. The proper mixture for the primer was found to be 60 parts of nitro-glycerin and 40 parts of nitro-hydro-cellulose. In Italy primers of pure gun-cotton are used.

(*b*) PROPERTIES.

Blasting gelatine is an amber-coloured translucent elastic mass, which can be cut, bent, and pressed without altering its shape. The older the blasting gelatine is, the harder up to a certain point it becomes. Well-made blasting gelatine will, even after repeated freezing and thawing, and storing for six days and nights at a temperature of 90° F., not exude any nitro-glycerin. Not only does it not run into a paste, but its height does not diminish by more than one quarter, and the edges maintain their sharp form. It should stand the heat test for at least 10 minutes, and very often it will stand it for from 40 to 60 minutes.

The manufacture of blasting gelatine to the above requirements is not at all an easy matter, and only a very few factories are able to regularly make such a product. The blasting gelatine made on the Continent is not so good as that made in England, probably on account of the less stringent regulations.

Blasting gelatine explodes under a blow of only 25·3 foot pounds (3½ kilogramme-metres) between steel and steel. In some experiments made by H. M. Inspectors of Explosives, soft blasting gelatine was exploded between brass plates by a weight of 56 lbs. falling from a height of 12 feet, and frozen blasting gelatine by the same weight falling from a height of only 1 foot. Gelatine dynamite behaved in the same manner when soft as when frozen, the drop required to cause explosion with a 56 lbs. weight being 8 feet and 7 feet respectively.

For this reason strong detonators are required to explode blasting gelatine. The strength of detonator required varies with the percentage of gun-cotton, and in some cases detonators containing charges of 1 gramme and more of fulminate are required. Camphorated blasting gelatine has been exposed to a temperature of 158° F. for eight days without exploding or showing signs of dangerous decomposition. It is extremely insensitive to shock, and cannot be exploded with certainty either by detonators containing charges of 2 grammes or primers of compressed gun-cotton.

The Austrian Military Committee made experiments on the effect of rifle-shots on blasting gelatine. Captain Wawra's report on these was published in the Journal of the Committee in 1879. These experiments showed that soft blasting gelatine containing 4 per cent. of camphor could not be exploded with certainty at a distance of 150 paces. On the other hand, it has been shown, and it also follows from experiments made in this country, that frozen blasting gelatine is far more sensitive than not only unfrozen blasting gelatine, but also than kieselguhr dynamite.

Blasting gelatine can be kept for an almost indefinite time under water. After some time its surface gets to look milky, and the mass becomes turbid; its blasting effect, however, is not effected by storage under water.

If pure blasting gelatine be heated slowly, beginning at 60° C., it will explode, according to experiments of Hess, at 204° C. On rapid heating it explodes at 240° C. Blasting gelatine containing camphor cannot be exploded by gradual heating, and only burns away with sparking; at least Hess did not succeed in exploding it even at 330° C. If ignited in a frozen state, even small quantities of blasting gelatine may explode.

2. GELATINE DYNAMITE.

Pure blasting gelatine is only used for extraordinarily tough rock and for military purposes. For general purposes its effect is too violent, and suitable mixtures were therefore made soon after it was invented in order to weaken its effect. The simplest and safest way of doing this is to make a thinner gelatine (about the thickness of syrup) and knead into it an absorbing powder of suitable composition. The most varied bodies have been proposed as absorbing powders, but now-a-days a mixture of potassium, or sodium nitrate with wood pulp, is generally preferred. In France and Italy rye-flour is sometimes used in the place of wood pulp. Sometimes an addition of burnt ochre is made in order to give it a similar colour to kieselguhr dynamite.

For mining purposes a mixture is made which is sold here as gelatine dynamite, and in Germany as miners' blasting gelatine, and which consists of about 80 per cent. of blasting gelatine containing 5 per cent. of gun-cotton, and of 20 per cent. of absorbing powder. Formerly various intermediate stages between this gelatine dynamite and the now more usual "Gelignite" were made, but have since been abandoned.

Gelignite (or, as it is called in Germany, gelatine dynamite) has the following composition:—

		PER CENT.
65 per cent. gelatine of ...	$96\frac{2}{13}$ per cent. nitro-glycerin...	=62·500
	$3\frac{11}{13}$ „ collodion cotton	= 2·500
35 per cent. absorbing powder	75 parts sodium nitrate ...	=26·250
	24 „ wood pulp	= 8·400
	1 „ soda	= 0·350
		100·000

Sometimes, in order to increase the solidity of the gelatine, a little more gun-cotton is added. Gelatine dynamites weaker in action are sometimes made containing less gelatine and more absorbing powder.

In France and Italy ammonium nitrate is preferred in place of the potassium and sodium nitrate, and in fact greater effect is thereby obtained; but there are the disadvantages that the dynamite gradually absorbs moisture, and that the ammonium nitrate becomes acid through the volatilization of ammonia, which when liberated impairs the stability of the dynamite and weakens the retention of the nitro-glycerin.

The admixture of the absorbents with the gelatine is done either by hand or in kneading machines, as in the manufacture of the latter, and the whole process of making gelatine dynamites is exactly the same as that of blasting gelatine.

3. CHEMICAL EXAMINATION OF BLASTING GELATINE AND GELATINE DYNAMITE.

The analysis of blasting gelatine and the gelatine dynamites is conducted practically on the same lines as the examination of dynamites, but with special consideration to the gelatinous state of the explosive due to the nitro-cellulose it contains.

(*a*) Qualitative Examination.

A disc is cut from the centre of the cartridge and divided as finely as possible into small cubes by means of a horn or wooden knife. These are put into a small flask, and a mixture of 2 parts of ether free from water and 1 part of alcohol, also free from water, is poured over it. Nitro-glycerin, collodion cotton, and any admixture of camphor, resin, paraffin, or sulphur is dissolved out. An excess of chloroform is then added which precipitates the collodion cotton. It is allowed to settle, the liquid part then decanted, and the residue pressed between filter paper. The separated collodion cotton is recognized by its inflammability, its property of exploding when struck between a hammer and anvil, and by its behaviour with boiling sodium sulphide solution, in which it dissolves. If boiled with hydrochloric acid and iron sulphate, the nitric oxide reaction should appear. The decanted solution is heated to 30° C., which drives off the greater part of the ether, alcohol, and chloroform. The temperature is then raised to 80° in order to completely drive off these solvents. The residue is then treated in the same way as described in the analysis of dynamite. If the presence of tri-nitro-cellulose in the residue after it has been freed from nitrates and chlorates be suspected, it can be demonstrated either—

(1) By examining it under a microscope with polarized light, when tri-nitro-cellulose will have a dull appearance, whilst the non-nitrated cellulose will give vivid colours.

(2) By boiling the residue with hydrochloric acid and iron sulphate, when tri-nitro-cellulose will give the nitric oxide reaction.

(3) By boiling the residue in potassium solution to which alcohol has been added and filtering, and then slightly acidulating the filtrate with sulphuric acid, when free nitric acid or hyponitric acid will be recognized if tri-nitro-cellulose be present.

(*b*) QUANTITATIVE EXAMINATION.

A sample is prepared, cut up with a horn spatula into small cubes, as before, and left standing for five or six days in a desiccator over calcium chloride in order to determine the percentage of moisture.

The best means of extracting the nitro-glycerin and the collodion cotton from the gelatinous explosive is to treat it with ether-alcohol. Hess has also recommended the use of Soxhlet's extracting apparatus for this purpose; but it has been found that in order to completely dissolve the nitro-compounds from a sample of a few grammes only, it is necessary to syphon off at least fifty times, which would take about 24 hours. Moreover, in spite of all precautions, it is hardly possible to avoid the evaporation of some of the nitro-glycerin, and consequently the use of extraction apparatus has been discontinued of late, and an Erlenmeyer flask used, into which about 5 grammes of the gelatinous explosive in a finely-divided state are put, 200 cubic centimetres of ether alcohol being then added to dissolve it. The dissolving is done by letting it stand a few hours with frequent shaking, or overnight. An excess of chloroform is next added to the solution, and the mixture filtered through a dried and weighed linen filter. The filtrate is freed from ether alcohol and chloroform on a water-bath of about 30° C. If camphor be present in the residue, the latter is shaken up with carbon di-sulphide. The ether extract can also be filtered at once, and the nitro-cellulose precipitated by chloroform in the filtrate only, but on account of the gelatinous nature of the solution this method is not quite exact.

The residue on the filter may still contain tri-nitro-cellulose and absorbing powder. It is therefore dried at 60° C. in a current of dry air and weighed. The analysis is then proceeded with as described for dynamite in order to determine the soluble salts (saltpetre, chlorates, etc.) and organic extractive matter. Part of the residue is then boiled with a concentrated solution of sodium sulphide, which dissolves any tri-nitro-cellulose. The solution is filtered through a weighed filter, carefully washed with water, dried and weighed, and the percentage of tri-nitro-cellulose calculated from the loss of weight. From another part of the residue any carbonate of calcium or magnesium is extracted by dilute hydrochloric acid, and the bases determined in the extract. The washed, dried, and weighed residue then contains the organic and mineral absorbents. The latter can be determined by calcining. Since extracting the nitro-compounds from gelatine dynamite never gives quite exact results, it is better to determine the absorbents with larger quantities. It is advisable to prepare a larger sample of the explosive, and to always use parts of it separately to singly determine the components. Much more exact results will be obtained in this manner, and an analysis can be finished in a much shorter time.

Dr. Scheiding has proposed the following method :—The blasting gelatine is first treated with ether alone, which extracts the nitro-glycerin only. The saltpetre is then extracted with boiling water, and the nitro-cellulose from the residue by cold acetic ether. Should the percentage of tri-nitro-cellulose have to be determined, he first extracts with ether alcohol and then with acetic ether.

4. EXPLOSIVES RELATED TO GELATINE DYNAMITE.

Attempts have repeatedly been made to obtain a gelatinous explosive by dissolving nitrated wood, vegetable ivory, jute, etc., either in nitro-glycerin or in nitro-benzene, etc., instead of nitro-glycerin. Amongst the latter was "Kinetite," which consisted of nitro-benzene and collodion cotton with the admixture of potassium chlorate, potassium nitrate, and antimony sulphide. It could only be exploded by very strong detonators, but on the other hand it was not free from the objections peculiar to all chlorate powders, and therefore neither it nor any other similar explosive could maintain its position for long.

CHAPTER XVIII

SPRENGEL'S EXPLOSIVES AND SAFETY BLASTING MATERIALS.

DR. HERMANN SPRENGEL in 1873 described a large number of explosives till then unknown, which on account of the peculiarity of their composition created considerable sensation. They were of two kinds—liquid and solid. The liquid ones were in general solutions of nitrated hydro-carbons,—chiefly nitro-benzene or picric acid, in nitric acid. These mixtures could be exploded with considerable effect by a detonator. Sprengel himself declared these liquid explosives to be impracticable; nevertheless, later on patents were taken by Hellhoff for "Hellhoffite," and by Turpin for "Panclastite," which were essentially the mixtures suggested by Sprengel.

It was intended to produce these explosives where they were to be used, so that two bodies which in themselves were harmless could be carried to the spot at which the explosive was required and transformed into a violently acting explosive by merely mixing them together. Objections were soon raised to this through fear of possible injury to the workmen and danger in mixing, and therefore the use of glass vessels or sheet-lead cases for wrapping these explosives was proposed. Although their effect quite fulfilled expectations, and was in some cases excellent, these explosives never got beyond the experimental stage, and at the present time have quite disappeared.

The same has happened to the solid or neutral explosives suggested by Sprengel, in which potassium chlorate in the place of nitric acid was used as the oxygen carrier. In these explosives the potassium chlorate was moistened with water and then compressed into a porous cake which was to be immersed in a hydro-carbon or a nitrated hydro-carbon—such as carbon di-sulphide, benzene, nitro-benzene, and paraffin oil—at the place where the explosive was wanted. None of them, however, gained a firm footing in Europe. They were used in America for blasting operations at the Hell Gate in New York Harbour, and still are employed for larger works under the name of "Rackarock," which consists of potassium chlorate and nitro-benzene. There is also "Rackarock special," which, in addition to chlorate of potash and nitro-benzene, contains from 12 to 16 per cent. of picric acid.

Black powder and similar explosives which burn slowly but develop a large quantity of heat are not suitable or safe to use in fiery or dusty mines, as they ignite the gases and coal-dust and cause disastrous explosions. Highly explosive bodies like dynamite and gelatine dynamite are rather more safe under these conditions because they explode in a much shorter time; nevertheless they also have proved to be untrustworthy in such mines. According to a report by Mallard and Le Chatelier to the French Committee of Explosives, a mixture of fire-damp and air is only ignited by explosives when these on explosion develop a greater heat than 2200° C. The Commission found that mixtures of equal parts of dynamite and soda crystals, or dynamite and glauber salts, ammonia alum, or ammonium chloride will not ignite fire-damp. The most advantageous mixtures tried, however, consisted of nitroglycerin or gun-cotton with ammonium nitrate. Such mixtures have received attention previously in the chapter on dynamite. Another form of safety explosives made from nitro-compounds of aromatic hydro-carbons are described below.

1. ROBURITE.

Roburite was invented by Dr. C. Roth of Charlottenburg, and according to his patent is made by heating 1 part of naphthalene for three hours with 5 parts of sodium nitrate and 6 parts of concentrated sulphuric acid. The resulting nitro-naphthalene is washed, dried, and carefully mixed with 0·8 part of potassium chlorate; 5 parts of concentrated hydrochloric acid are then gradually run in, and the mixture heated on a water-bath. According to another modification 5 parts of tar are gradually introduced into 15 parts of nitric acid of 1·45 specific gravity and 12 parts of hydrochloric acid, which are kept cool during the first part of the process. Towards the end the mixture is heated and each part of the resulting plastic mass again nitrated in a mixture of 5 parts of nitric acid and 15 parts of sulphuric acid. The chloro-nitro compounds thus formed are then mixed with potassium chlorate, saltpetre, or some similar oxygen carrier. For instance, 1 part of chloro-nitro-naphthalene and 2 parts of saltpetre were used as a mixture.

It appears that nitro-naphthalene has been since abandoned and chloro-di-nitro-benzene mixed with ammonium nitrate used instead. Whether chloro-di-nitro-benzene is still used, or whether di-nitro-benzene alone has replaced it, is not quite certain. In this country, according to the terms of the licence under which it is made, roburite is considered as containing chloro-di-nitro-benzene. In Germany it is said to consist of di-nitro-benzene and ammonium nitrate only, and to be made by melting di-nitro-benzene and gradually introducing it into the ammonium nitrate.

2. BELLITE.

Bellite was invented by Charles Lamm of Rotebro near Stockholm, and according to his patent consists of 1 part of di-nitro-benzene and 1·9 parts of ammonium nitrate, or 1 part of tri-nitro-naphthalene and 2·57 parts of ammonium nitrate.

The meta-di-nitro-benzene which he used is obtained by dissolving di-nitro-benzene in alcohol. As the solution cools meta-di-nitro-benzene crystallizes out, whilst ortho- and para-di-nitro-benzene remain in solution. The two components of bellite are mixed together in a steam-heated drum, whereby the meta-di-nitro-benzene, which melts at 194° F., is deposited on the ammonium nitrate particles in the form of an unbroken coating. Before the mass has quite cooled it is compressed into cartridges. When cold these are hard and have a specific gravity of 1·25 and a melting-point of 90° C.

3. SECURITE.

According to the patent of F. Schöneweg of Dudweiler, securite is made by dissolving ammonium nitrate and potassium oxalate or ammonium oxalate in water, drying the mixture at 176° F., and adding 10 parts of nitro-benzene or 20 parts of di-nitro-benzene. It is manufactured both in this country and in Germany.

4. AMMONITE (Favier's Explosive or Miners' Safety Explosive).

P. A. Favier of Paris patented the use of ammonium nitrate in connection with paraffin wax, resin, and nitro-naphthalene. This explosive has in the course of time undergone various modifications, and according to the 'Génie Civil,' 1891, p. 241, the following five varieties were then manufactured:—

		PER CENT.
(*a*) Grisounite for coal (temperature of explosion, 1480° C.)	Sodium nitrate ...	95·5
	Tri-nitro-naphthalene....	4·5
(*b*) Grisounite for rock (temperature of explosion, 1785° C.)	Sodium nitrate	92
	Tri-nitro-naphthalene ...	8
(*c*) Favier's Powder No. 1.	Ammonium nitrate ...	88
	Di-nitro-naphthalene ...	12
(*d*) Favier's Powder No. 2.	Ammonium nitrate ...	44
	Sodium nitrate ...	40
	Di-nitro-naphthalene ...	16
(*e*) Favier's Powder No. 3.	Sodium nitrate ...	75
	Mono-nitro-naphthalene	25

Latterly the Powder No. 1, consisting of ammonium nitrate and di-nitro-naphthalene, has alone found favour, although the manufacture of di-nitro-naphthalene is more difficult than that of mono-nitro-naphthalene. When mono-nitro-naphthalene is used many misfires occur, whilst with di-nitro-naphthalene they are no longer feared.

In France ammonite is made as follows:—The nitrate is dried in a steam-heated tube through which it is passed by means of a screw. It is then pounded in a heated mortar and sprinkled with the molten di-nitro-naphthalene. The resulting cake is first put into a roller granulating machine, and then sifted. The larger grains are formed under moderate pressure into hollow cylinders which are filled with the fine grains in a loose state. The cylinders so prepared are dipped in paraffin and the whole wrapped in paraffin paper.

In the English factory at Stanford-le-Hope the dried materials are incorporated on heated incorporating-mills, and then mixed in sifting machines fitted with rotating brushes. Only loose powder is used, and it is pressed by means of a screw filling machine into thin cases of tinfoil, similar to those used for artists' colours. The cartridge-filling machine is illustrated in Figs. 216 and 217. It consists of a hollow wooden block (a), into which three brass tubes (b) are let in; these are half cut away where they are inside the hollow in the block. In these tubes endless screws (c) revolve, which fill the ammonite into cases (f), which are slipped over the tube. The case rests against a small slide (g), which is held up to the tube by a weight (i), the motion of which is limited by means of a stop (h). On their outer ends the screws

(c) are fitted with cog-wheels (e) connected with each other by means of intermediate wheels (e_1), so that on turning the handle (d) all the screws revolve simultaneously. The cases have a small closed nipple, the top of which can be cut off to insert a detonator. The other end is closed by a screwed cap after the cases are filled. After closing they are dipped into paraffin wax, which penetrating the thread on the cap makes a thoroughly waterproof joint.

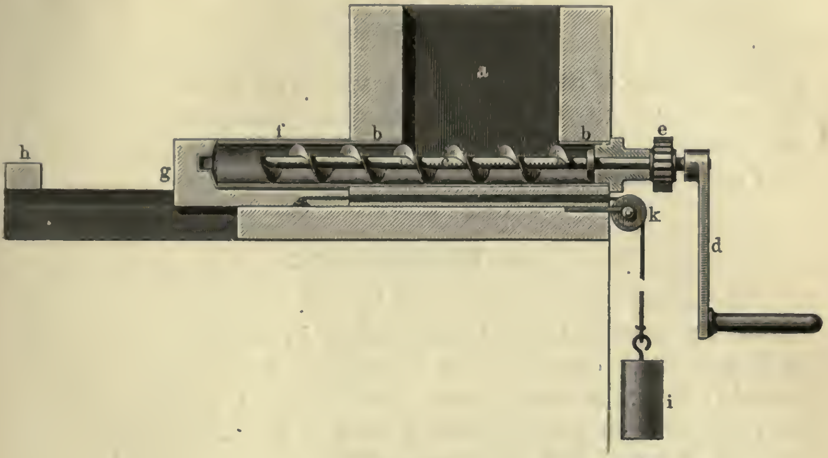


FIG. 216.

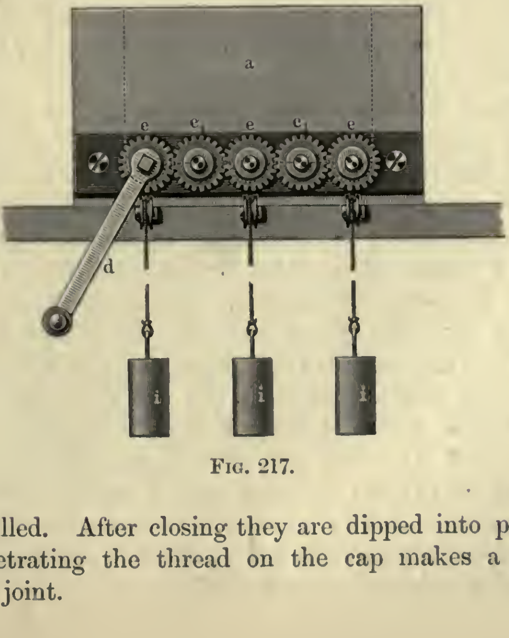


FIG. 217.

In order to insert the detonator the nipple is cut open, the detonator is put in with a fuse, and the whole squeezed together by means of detonator pliers.

5. ROMITE.

R. Sjöberg of Stockholm has patented an explosive consisting of ammonium nitrate, nitro-naphthalene, potassium chlorate, and ammonium carbonate in various proportions, but has not obtained any great success with it.

6. WESTPHALITE.

The safety explosive patented by the "Westphälisch-Anhaltische Sprengstoff-Actien-Gesellschaft" in Coswig, and called "Westphalite," consists of a mixture of resin and ammonium nitrate; 6½ parts of resin are dissolved in 3½ parts of alcohol, and the solution poured into 90 parts of ammonium nitrate and treated in a ball mill for from 1 to 2 hours. By moistening the ammonium nitrate it can be ground to a finer powder than in a dry state, and at the same time a thorough mixture can take place. The mixture is then heated in order to drive out the solvents, further ground, and put into cartridges.

In order to promote solution of the resin about 10 parts of nitric ethylic ether are added to the alcohol. That coming from the manufacture of mercury fulminate is used.

For filling cartridges with Westphalite, the same company patented the apparatus illustrated in Fig. 218. Its chief object is to give the cartridges a uniform and adjustable density of loading, and for this purpose the cartridge cases are submitted to the continuous action of a shaking apparatus. The machine consists of a funnel (A), in which a measuring drum (B) provided with radial compartments revolves. The compartments are alternately filled from the funnel with the pulverulent material and discharge their contents into the prolongation of a discharge tube (C), and thence into the cartridge (E). The cartridge rests on a frame (D) which is moved up and down between guides (G) by means of a cam (F). The cam-shaft, and at the same time the measuring drum (B), are actuated by link chains which rest on a pulley (H), provided with a handle. Since the cam-shaft obtains a greater velocity than the measuring drum, the former will obtain a large number of blows for each filling.

7. CARBONITE.

Under this name Messrs. Schmidt and Bichel, of Schlebusch, formerly made an explosive which consisted of 1 part of sulphuretted tar oil, 0·5 part of nitro-cumene, and 9 to 10 parts of sodium nitrate. The sulphuretted tar oil was produced by heating tar oil with pulverized sulphur. This explosive has since been given up, and now-a-days the name "Carbonite" is only used for a certain dynamite, a description of which is to be found in the chapter on dynamite on p. 190.

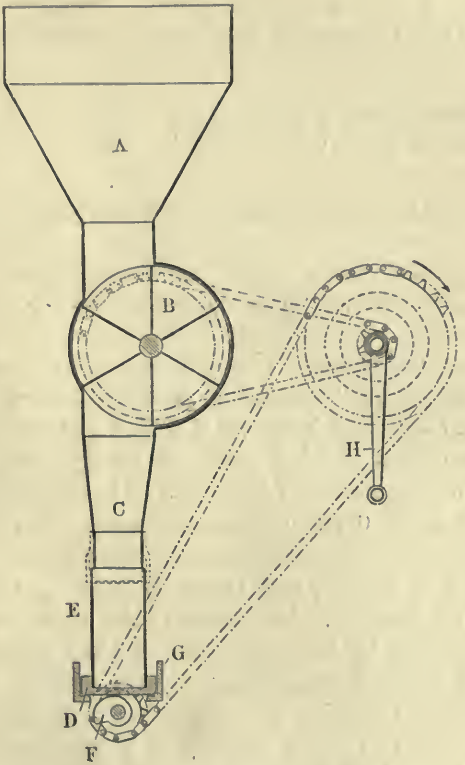


Fig. 218.

8. FRENCH BLASTING MATERIALS MADE FROM GUN-COTTON AND AMMONIUM NITRATE.

In consequence of the researches of Mallard and Le Chatelier, which are mentioned on p. 226, the French Government have issued for sale blasting materials made of gun-cotton and ammonium nitrate intended for use in fiery mines. They are made in two qualities, and their respective composition is as follows :—

		FOR ROCK.	FOR COAL SEAMS.
Collodion cotton	calculated as dry weight	20	9·5
Ammonium nitrate		80	90·5

They correspond to the following composition (according to Vieille's formulæ) :—

20/80 powder $C_{24}H_{32}N_8O_{36} + 127{\cdot}4\ NH_4NO_3$.
9·5/90·5 „ $C_{24}H_{32}N_8O_{36} + 50{\cdot}4\ NH_4NO_3$.

The prime materials are the collodion cotton and ammonium nitrate of commerce. The latter is sifted through sieves of $\frac{1}{8}$ inch mesh and contains between 2 and 3 per cent. of moisture. The collodion cotton is dried to about 12 per cent. of moisture. The mixture of the materials is worked in incorporating-mills, and then contains about 6 per cent. of moisture. The runners weigh half a ton and make ten revolutions per minute.

The charge is 22 lbs., and takes an hour to work, including charging and discharging. At the end of the operation the runners are given one turn by hand, whereby a kind of cake is formed. This is passed through three leather sieves without a granulating disc, then the mass forming grains about $\frac{1}{8}$ inch diameter is dried during 24 hours, and again worked in a granulating apparatus, consisting of three leather sieves, but this time a granulating disc is used. The grains are sorted into two sizes, those of between 3 millimetres and 0·65 millimetre, and those below the latter size. The larger-sized grains are dried for three hours and taken to the press in tightly-closed boxes. The real specific gravity is 1·47, and their gravimetric density 0·50. The grains below 0·65 millimetre are used for primers, as will be mentioned later on, while the grains between 3 millimetres and 0·65 millimetre are compressed into cartridges with a central core. Two kinds of cartridges are made of the following proportions :—

WEIGHT OF THE CARTRIDGES.	CYLINDERS				DIAMETER OF THE CORE.	WEIGHT OF THE PULVERULENT CHARGE OF THE CORE.
	Number for one cartridge.	Weight.	Height.	Diameter.		
Grammes.		Grammes.	Millimetres.	Millimetres.	Millimetres.	Grammes.
100	3	28	53	30·2	13	16
50	2	21·5	39·5	30·2	13	7

The real specific gravity of the compressed cylinders is 0·906, and the average specific gravity of the cartridges 0·878.

The cylinders are rolled in paper forming a cartridge, and both the sides and one end are glued. After this the cartridges are dipped into molten paraffin, the pulverulent mass is put into the central core, and compressed by knocking it against the table. The other end of the cartridge is then closed and also dipped into paraffin. After this the cartridges are again coated twice with paraffin. Fourteen kilogrammes of these cartridges are put into a case made of sheet-zinc, which is soldered with easily fusible solder.

On using these cartridges one end is opened and a detonator of 2½ grammes charge of fulminate inserted into the pulverulent core. Detonators with a lower charge will not detonate this explosive with certainty.

By regulations issued on the 1st August, 1890, the French Minister of Public Works prescribed that in future only the following explosives shall be used in fiery mines:—

(1) A mixture of dynamite No. 1 of 75 per cent. nitro-glycerin and ammonium nitrate, in which the quantity of dynamite is not more than 40 per cent. for use in rock, and not more than 20 per cent. in coal seams.

(2) A mixture of blasting gelatine (91·7 per cent. of nitro-glycerin and 8·3 per cent. of collodion cotton) and ammonium nitrate, in which the percentage of blasting gelatine is not more than 20 per cent. for work in rock, and not more than 12 per cent. for work in coal seams.

(3) Mixtures of collodion cotton and ammonium nitrate, with 20 per cent. and 9½ per cent. collodion cotton respectively for use in rock and coal.

(4) Mixtures of di-nitro-benzene and ammonium nitrate containing not more than 10 per cent. di-nitro-benzene for work in rock.

In general the regulations state that only such explosives shall be used in fiery mines, the products of explosion from which do not contain combustible matter such as hydrogen, carbonic oxide, carbon, etc., and that the temperature of explosion shall not be over 1900° C. for work in rock, and not over 1500° for work in coal seams. The composition must be written on the cartridge, so that the temperature of explosion can be calculated from the well-known thermo-chemical formulæ.

9. PROPERTIES AND BEHAVIOUR OF THE SAFETY EXPLOSIVES.

The various safety explosives enumerated above have in general proved satisfactory in fiery mines, since they either do not ignite the gases at all, or only under quite exceptional conditions. They are also not subject to the defect of freezing at comparatively high temperatures, like dynamite, and therefore do not require thawing. Further, they are only sensitive to shock in a very slight degree. As a rule, they burn away harmlessly, even in large quantities, and on account of these properties their carriage by ordinary goods trains has been permitted for some time in Germany. On the other hand, they have the disadvantage of low specific gravity, and consequently require comparatively large bore-holes. Their effect is much inferior to that of dynamite, and therefore they cannot be used with advantage in hard rock, where the cost of wide bore-holes is a great consideration.

Both in the manufacture and in the use of many of these explosives complaints about poisonous vapours have been heard, and even some cases of death ascribed to them, and there is hardly any doubt that to weak constitutions or to people already suffering from certain diseases, inhaling nitro-benzene and nitro-naphthalene vapours is not without danger. These nitro-hydrocarbons are fairly volatile near their melting temperature, and even considerably below it, and H. M. Inspectors of Explosives have therefore issued special instructions as to minimizing the development of these vapours during manufacture.

CHAPTER XIX

SMOKELESS POWDERS

PROBABLY the first attempt to use as a propelling agent an explosive which burns without developing smoke was made by Captain von Lenck of the Austrian army, who tried to adapt gun-cotton for use in guns; but, as previously mentioned, his experiments were not successful.

Schultze, in his powder made from wood nitro-cellulose, avoided the extraordinary rapidity of combustion of gun-cotton by using another prime material than cotton and by suitable admixtures. This powder has, however, only been used for sporting purposes.

The first step towards a smokeless powder according to modern ideas was the invention of "E. C." powder by Messrs. Reid and Johnson in 1882, and the next that of "J. B." powder by Messrs. Judson and Borland. Both of them contained gun-cotton in an unaltered state, and were distinguished from it only by their external appearance—that is to say, by being in the form of grains with a smooth surface which was produced by suitable solvents.

The present smokeless powder industry only dates from the manufacture of the "Poudre B" by Vieille in 1886, and the ballistite by Nobel.

The smokeless powders at present known can be divided broadly into three classes:—

(1) Powders in which gun-cotton, either the so-called insoluble or the soluble variety, alone is used, which by aid of a solvent has been converted into a horny substance and then is formed into flakes or cords.

(2) Powders in which a mixture of nitro-glycerin and either di- or tri-nitro-cellulose is transformed into a similar horn-like substance either with or without the aid of a solvent.

(3) Powders which contain nitro-derivatives of the aromatic

hydro-carbons either by themselves or in connection with nitro-cellulose.

Suggestions have been made for smokeless powders of other kinds—such in which nitro-cellulose is combined with an oxygen carrier, and also such which contain oxygen carriers without nitro-cellulose. The latter have been suggested most frequently, but so far they have not attained any appreciable success.

1. E. C. POWDER.

In 1882 Messrs. Reid and Johnson patented a process for making a gunpowder from nitro-cellulose, which they called E. C. Powder (Explosives Company Powder). They first formed grains by putting pulped nitro-cellulose into barrels, spraying water over it, and agglomerating it by revolving the barrel. The grains were then dried, moistened with ether (50 to 80 parts by volume to 100 parts of grains), again dried, and the grains which adhered slightly to each other separated by rubbing them through a sieve.

In order to colour the powder, aurine (rosolic acid) was added; this gave it a reddish-yellow colour. The grains through the above treatment were coated with a hard skin, consisting of a thin layer of collodion. More recently a kind of E. C. powder has been made, which is completely soaked with ether, whereby a hard grain giving a more uniform effect is obtained.

The E. C. powder is still largely used in Great Britain and the Colonies for sporting purposes. In its behaviour it is very similar to the modern smokeless powders.

2. J. B. POWDER.

This powder obtained its name from its inventors, Messrs. Judson and Borland. They used di-nitro-cellulose sufficiently wetted with water to lose its inflammability, and this was then mixed with potassium or barium nitrate, and granulated either by rubbing through sieves or by revolving it in drums. After this the grains were dried and then moistened with a quantity of a solution of camphor in benzene sufficient to work the process without an excess. The camphor solution was said to boil at 212° F., and not to have a gelatinizing or disintegrating effect on the powder, either by itself or when this was mixed with camphor.

In practical working the inventors took

	PARTS.
Di-nitro-cellulose	68
Barium nitrate	25
Potassium nitrate	6
Ultramarine	1
	100

These were intimately mixed and granulated in a drum, whilst water was admitted in a fine spray. After drying, 100 parts of the powder were moistened with a solution of 10 parts of camphor in 50 parts of petroleum spirit, which was allowed to act upon it for 15 minutes. The powder moistened by the solvent was then heated by hot water in vessels provided with a water-jacket, a cover, a stirring arrangement, and an outlet pipe. The evaporated benzene was recovered in a condensing apparatus, and the powder remained in the vessel impregnated with solid crystalline camphor. The heating was then continued until the bulk of the camphor sublimed on the still head. The powder was taken out and heated to 212° F. in an open vessel, and finally exposed at ordinary temperature to the air, so as to absorb moisture from it.

J. B. powder had a silvery grey colour, and the grains were fairly hard and uniform. For some time it was used as a sporting powder, but appears to have quite died out now.

3. GUN-COTTON POWDERS.

The first military smokeless powder, which was made in France in 1886 by Vieille, seems to have been composed of picric acid and gun-cotton. It was, however, very soon abandoned, and the so-called "Poudre B," which consists of gun-cotton only, adopted.

Speaking generally, pure gun-cotton powders now only differ in their mode of manufacture and the form given to the finished powder.

(*a*) FLAKE POWDERS.

The prime materials for the manufacture of flake powders for military purposes are chiefly tri-nitro-cellulose and acetic ether, or acetone. For sporting powder di-nitro-cellulose is chiefly used, and as the solvent a mixture of sulphuric ether and alcohol. The nitro-cellulose is used in a finely-pulped state, and is first freed as completely as possible from water. To effect this it is exposed at a temperature of 40° C. for some days in drying-houses.

In Austria drying is hastened by the addition of strong alcohol, which constitutes the so-called "alcoholizing process." In this process the alcohol absorbs the whole of the water contained in the nitro-cellulose, and so becomes diluted, and as the boiling-points of strong alcohol and slightly diluted alcohol are very nearly the same, but both much lower than that of water, and as both are thus much more easily volatilized at low temperatures, the drying can be done more rapidly. The consumption of alcohol need only be very small, since nitro-cellulose as it comes from the centrifugal machines contains about 30 per cent. of water, and of this about 80 per cent. can be driven off in a comparatively very short time by heating, and it is only the last

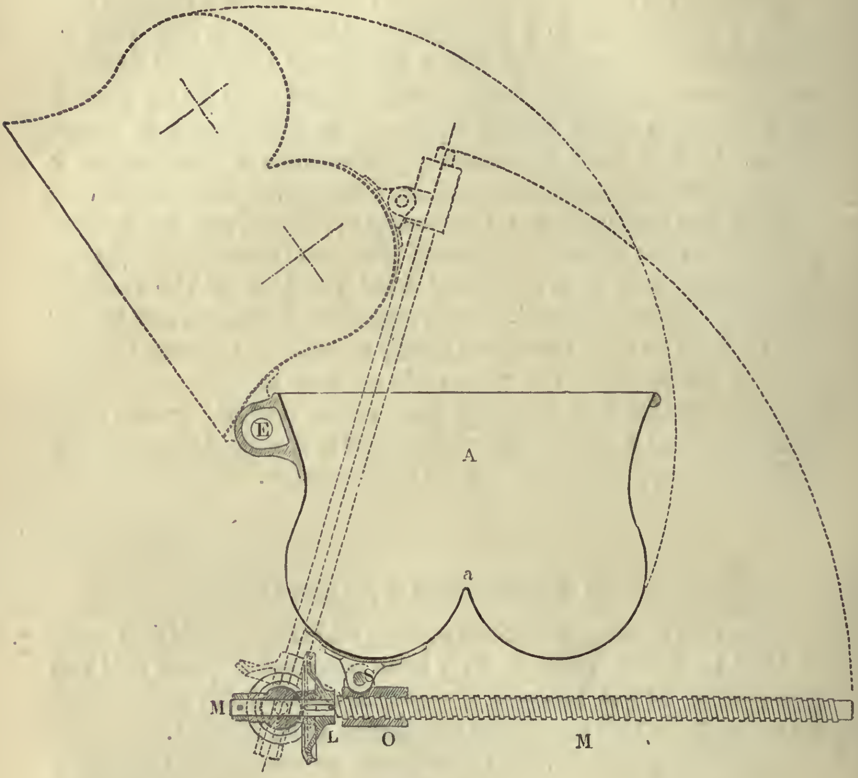


Fig. 219.

traces of moisture that are stubbornly retained, so that an addition of a small percentage is quite sufficient to effect the drying.

It is interesting to note that although this process has been used in Austria since the year 1891 without having been published, it was patented in this country by A. N. Durnford in 1892, the specifications of which patent were only published in 1894.

The nitro-cellulose to be used should be in the form of a fine powder not containing any coarse grains. Small lumps of unpulped gun-cotton only dissolve very slowly in the solvents used because they are surrounded on the outside with a coating of dissolved gun-cotton, which prevents the solvent from penetrating further.

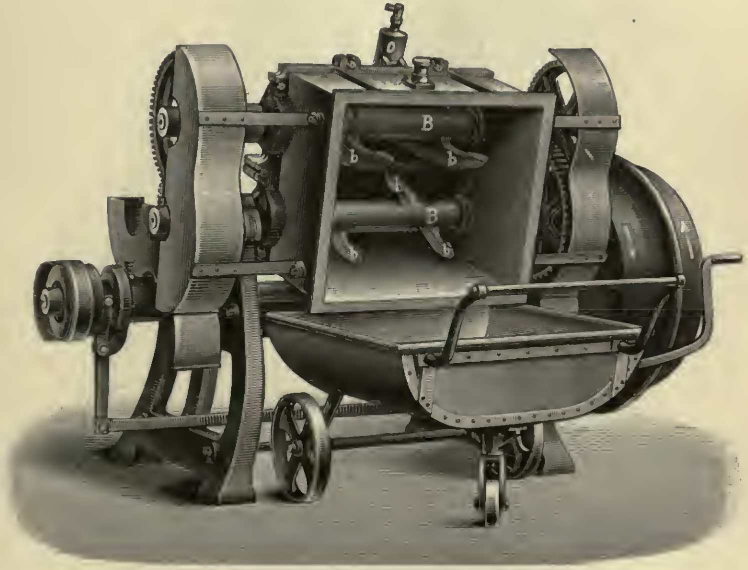


Fig. 220.

In France, the gun-cotton was formerly spread out in a thin layer in flat ebonite vessels and the solvent poured over it. If the gun-cotton were put into the ether, it would at first float on it, which would hinder the dissolving and also give rise to the formation of matted lumps. The ebonite vessels were put under glass covers and there left to themselves until the solution was completed. A current of air at 121° F. was then passed over to drive off the ether, which was condensed in suitable apparatus.

At present kneading machines are almost exclusively used, those of Werner, Pfleiderer and Perkins, of London and Cannstatt, being most usual, and it is necessary to describe them in detail in order that their action in the manufacture of the powder may be understood.

In principle, the kneading machines consist of a trough (Figs. 219 and 221), the upper part of which is rectangular, whilst the lower part consists of two half cylinders lying side by side. Where the two semi-cylinders touch, a ridge (a, in Fig. 219) is formed at the centre of the bottom of the trough. In the trough two kneading blades revolve, one in each half cylinder. These blades are either helical or consist of four screw stirring-arms mounted on two shafts. With both kinds of kneading arms the shape is so chosen that if they are revolved towards each other they force parts of the material being mixed against each other and against the central ridge on the bottom of the trough, and also push it forward over the surface of the cylinder. The clearance between the paddles and the cylindrical surfaces of the bottom being small, they act on the whole contents, so that nothing remains on the side of the trough, and yet there is no friction of metal against metal. Both paddles are revolved by a train of cog-wheels, the sizes being so chosen that one paddle turns with about double

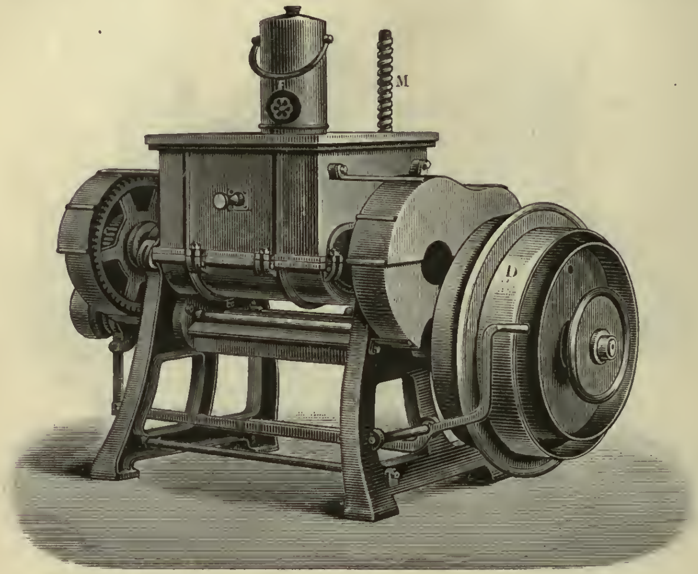


Fig. 221.

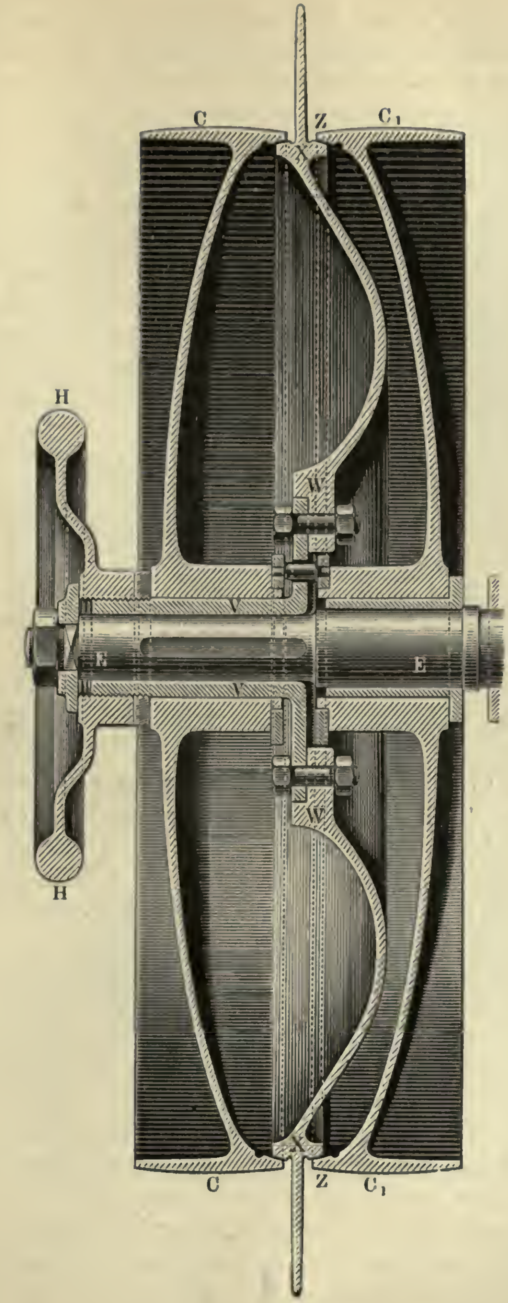


Fig. 222.

the velocity of the other. The result of this is that the kneading action is alternately stronger on the one side or the other, and the material is therefore constantly changed over, and the different parts of it are subjected to a complex varying action of turning, pressing, squeezing, and pushing against each other.

Small machines worked by a crank handle and fly-wheel are made, but as a rule they are driven by power. Since, in order to increase the effect of the kneading, the direction of motion of the paddles should be reversible at will—that is to say, that they should also be movable in an inverse sense or away from each other—Werner, Pfleiderer and Perkins have patented the reversing gear (D) which is illustrated in Fig. 222, on a large scale. It consists of two pulleys (C—C_1) driven one by an open and the other by a crossed belt from a shaft. Between these two pulleys the plate (W), with cast-on friction cones (X—X) attached to it, moves and the cones grip the rims (Z—Z) of the pulleys. The plate is firmly fixed to a cylinder (V) by a large flange and screws. The cylinder revolves with the shaft (E), and can be moved along it. A hand wheel (H), the boss of which is screwed, throws one or the other of the friction cones in gear with a pulley, so that either the open or crossed belt drives the machine at will, one driving it one way and the other in the opposite direction. When the wheel is in its middle position, neither cone is in gear, and the machine is therefore stopped.

In order to empty the trough it can be tilted on the front driving shaft. This is done in the smaller machines by hand, and by a mechanical arrangement in the larger sizes. Since the driving shaft remains in its bearings the paddle continues to revolve, whether the trough stand horizontally or vertically, and the emptying is thereby done by the machine itself.

The emptying arrangement for the trough is shown in Fig. 219, and the emptied trough in Fig. 220. At the bottom of the trough a nut is fixed by a hinge (S). Through this a screw spindle (M) passes to which a bevel-wheel is fixed by means of a key, which is driven by means of another bevel-wheel. The spindle rotates in a nut (O), which will then travel along the screw. As the trough is held fast by the hinge (E), and the screw spindle (M) by another hinge, the trough is tilted over.

In the latest designs the arrangement is slightly different in detail, but the principle remains unaltered. The cog-wheels are revolved either by a small handle or are connected by a gear-chain connected with the main shaft by a special foot lever arrangement.

For special purposes the kneading machine may be fitted with a jacket, through which warm or cold water can be circulated. The machine is as a rule made of cast-iron with a wrought-iron

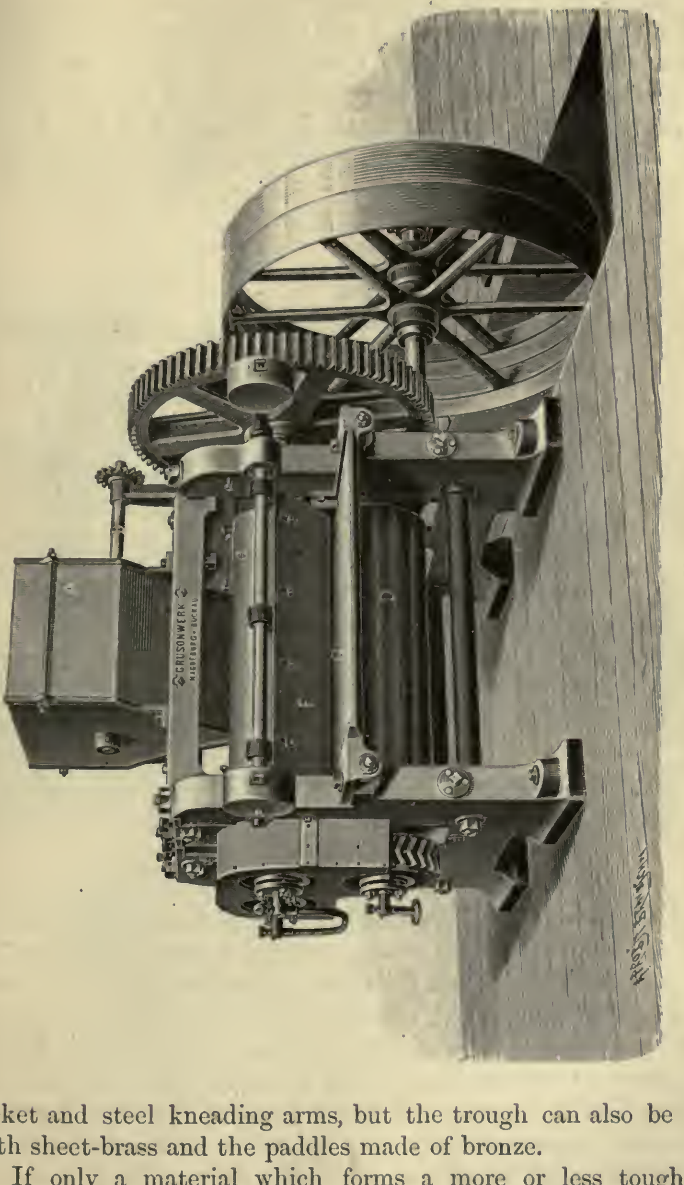


Fig. 223.

jacket and steel kneading arms, but the trough can also be lined with sheet-brass and the paddles made of bronze.

If only a material which forms a more or less tough, not dropping dough, is to be kneaded, nothing can leak out where the shaft passes through the trough. The case is different with such mixtures which have solid or liquid components, like blasting gelatine and similar materials. For reasons of safety these shafts must not fit quite tight in the sides of the trough, because they would require constant greasing and excessive friction would take place, which, under favourable circumstances, would finally give rise to dangerous heating of the material. There must, therefore, be a slight clearance round the shaft, about $\frac{1}{50}$ inch being sufficient. With a liquid material, gelatine dynamite, for instance, and also with some smokeless powders, some of the mass gradually works out through the small interstices and travels along on the shaft. In order to prevent it getting to the bearings of the shaft, a ring is fixed on the shaft between these and the sides of the trough, which carries any material leaking out downwards and lets it fall through an opening, whence it can be removed from time to time. For the purpose of better cleaning, the trough usually consists of two parts which join at the centre of the shaft, and are fixed to each other by bolts.

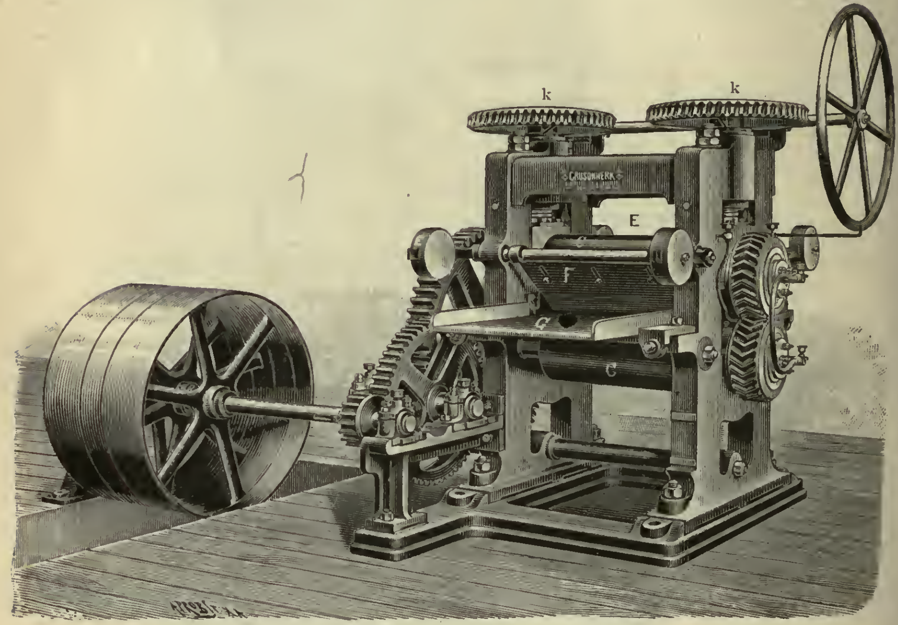


FIG. 224.

The two surfaces are exactly ground together in order to make a perfect joint. Making the casing in halves allows the upper half to be taken off and the kneading arms to be removed.

In order to introduce the easily volatile solvents used in making smokeless powders into the machine without allowing the vapours to escape into the open air, the machine is sometimes provided with a cover, as illustrated in Fig. 221, on to which a vessel holding the solvent fits. The vessel is tightly closed, and by turning a valve the solvent can be run into the apparatus. As a rule, this precaution is hardly necessary, since it is sufficient to put on to the machine a tightly-fitting, easily removable cover, and to prevent the development of heat during kneading by introducing cooling-water through the jacket, this also preventing the solvent from evaporating.

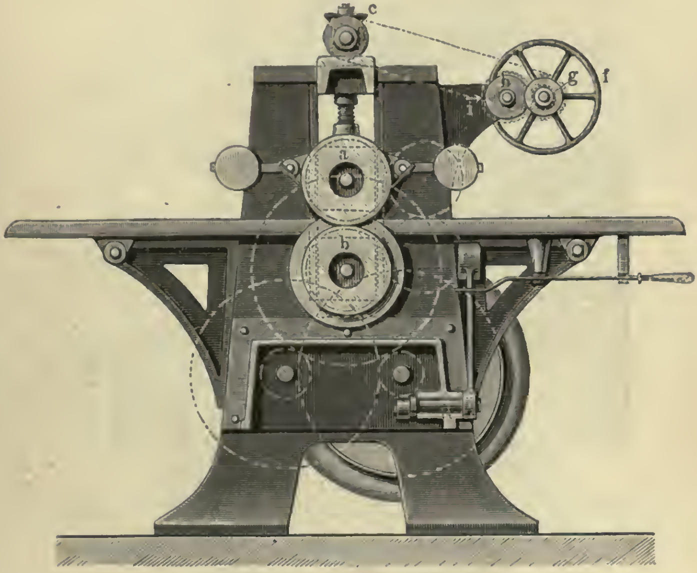


Fig. 225.—One-20th full size.

The kneading machines used for the manufacture of smokeless powders are, as a rule, made entirely of iron, since there is no fear at all of explosion.

The quantity of nitro-cellulose to be treated is put into the machine and the calculated quantity of solvent (acetic ether, sulphuric ether, or acetone) poured over it. After a few revolutions of the machine, the solvent so completely penetrates the nitrocellulose that the dissolving action begins. This is materially assisted by the kneading and squeezing of single particles between the sides of the trough and the kneading blades. The nitro-cellulose is thereby finely divided in the solvent, and after a short time the mass takes the form of a dough.

The operation of kneading lasts a longer or shorter time, according to the solubility of the nitro-cellulose and the quantity of the solvent used. Since so far it has not been possible to recover the solvent from the finished powder in a perfectly satisfactory manner, as little as possible is used, and the kneading continued a little longer. When this is done the time for kneading a charge is between 6 and 8 hours.

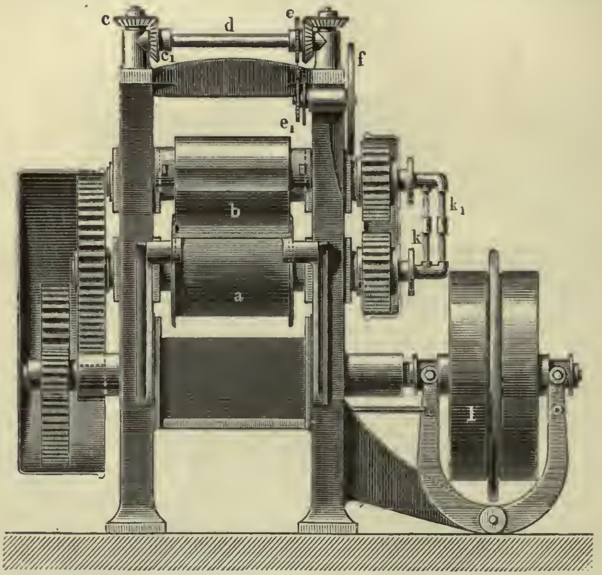


Fig. 226.—One-20th full size.

The quantity of powder usually worked at one operation is between 1 and 2 cwts., but there is no reason why larger quantities should not be treated at the same time.

The nitro-cellulose in this pasty state, both on account of the solvents and its jelly-like consistency, has completely lost its explosibility, but it is easily inflammable, and therefore suitable precautions for keeping away all fire and light must be taken.

The powder coming from the kneading machine is put through preliminary rolls, a suitable form made by F. Krupp, Grusonwerk, being illustrated in Fig. 223. The paste is introduced through a hopper (A) on the top of the machine, and is pushed down by a feeding arrangement (B) inside. The machine has two rolls (C) made of chilled cast-iron placed side by side horizontally, and a third one (D) below them opposite the front roll. The rolls are heated by steam, and can be fixed at suitable distances apart by means of an adjusting arrangement. The dough first passes between the two upper rolls, and then between an upper and the lower one. As it leaves these it is guided by a pair of scrapers (F) held by counter-weights (E_1) against the front upper roll, which prevents the sheet formed from rising on the roll and being carried round by it, and brings it on to the table (G) in front. The rolled sheets are then placed in a drying-house, where the bulk of the solvent is driven out. Since the vapours of the solvent are only driven off with some difficulty, some parts of the sheet expand and thus blisters are formed. In order to eliminate these and also to make the solution of the nitro-cellulose as perfect as possible, the sheets are put through a pair of final rollers.

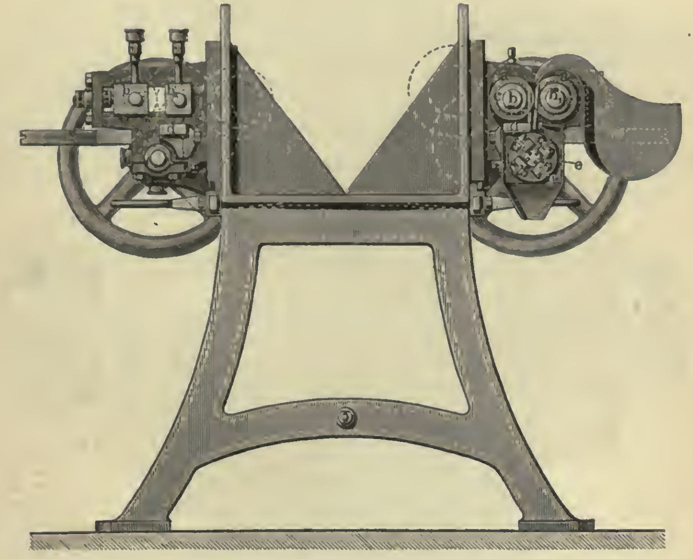


Fig. 227.—One-15th full size.

This machine is illustrated in Fig. 224 and is also made by Krupp, Grusonwerk. Figs. 225 and 226 show a machine made for the same purpose by Werner, Pfleiderer and Perkins. In both there is a pair of steam-heated rolls (c and a, b) actuated by cog-wheels, and scrapers (F) are pressed against the rolls by means of counter-weights. In Krupp's final rolls the fixing of the rolls against each other is done by drawing up or letting down the upper bearings by means of two screws, which are moved by a worm- and wheel-gear (k) turned by means of a hand-wheel. A pointer on the frame of the rolls and an index-plate on the hand-wheel show exactly the distance of the rolls apart.

With Werner, Pfleiderer and Perkins's rolls, the upper roll (a) is lifted by means of two cog-wheels (c and c_1) fixed on a shaft. On the cog-wheel shaft (d) there is a sprocket-wheel (e), which is geared by a chain to a second one (e_1), which is turned by a hand-wheel (f). A cog-wheel (g) fixed on the shaft of the hand-wheel moves another cog-wheel (h) provided with an index, on which, by means of a fixed dial (i), the position of the rolls can be read off. The rolling apparatus has in it cocks for steam or cooling water (k and k_1), and is revolved by a reversing apparatus (l).

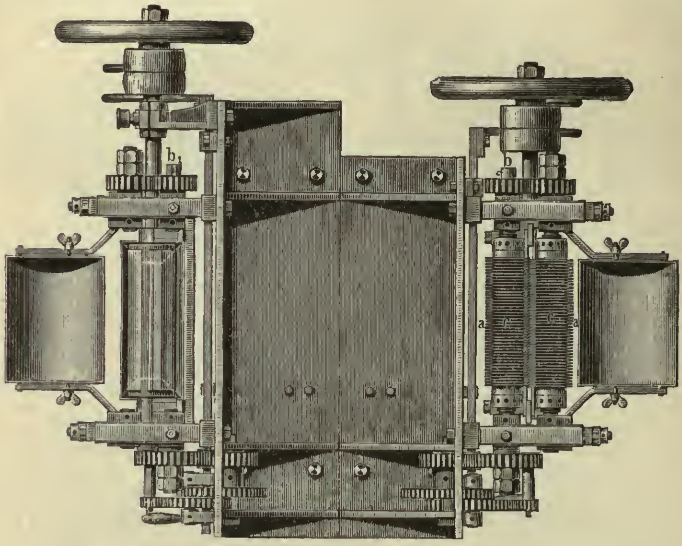


Fig. 228.—One-15th full size.

By the strong pressure in the final rolls all blisters are eliminated from the powder, and the heat of the warm rolls used in compression effects a further solution of the nitro-cellulose, so that the resulting thin sheets are perfectly transparent and homogeneous. The rolls are fixed according to the required thickness of the powder, but it is preferred to roll the sheets for all sizes of powder quite thin, and if the finished powder has to be of greater thickness, it is obtained by folding the sheets over and passing it again through rolls set to the proper distance apart. In this way the solvent is almost completely expelled from the powder, and the various thicknesses required are made by a kind of welding together of a certain number of thin sheets. The sheets thus built up are perfectly transparent, and on cutting them vertically no traces of joints can be seen; but if such a thick sheet or a grain be cut obliquely, the line of division between the sheets will be apparent. The rolling of the sheets must not be done too rapidly, but by gradually reducing the thickness by successive rollings, otherwise their surfaces will become rough and cracked.

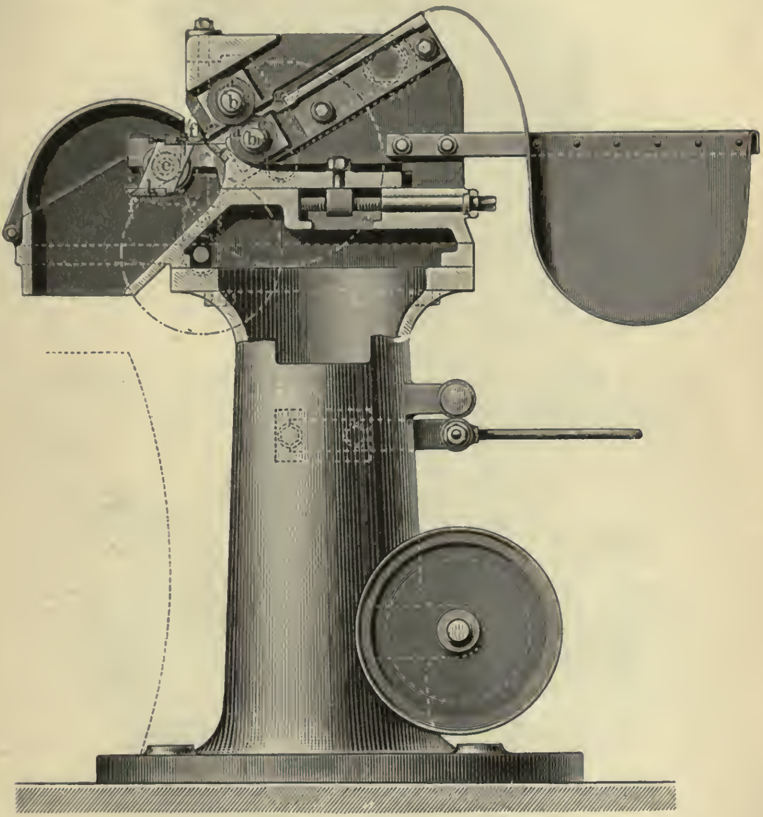


Fig. 229.—One-10th full size.

As previously mentioned, attempts have been made to recover the solvent driven off on rolling, and for this purpose hoods in connection with exhaustors have been tried, the exhausted vapours from which were carried into a condensing or absorbing arrangement. Since the solvent only gradually gets out of the powder, a great quantity of air was carried off with it, and it was found that the recovery of such a small quantity of solvent mixed with so large a quantity of air was hardly worth the expense.

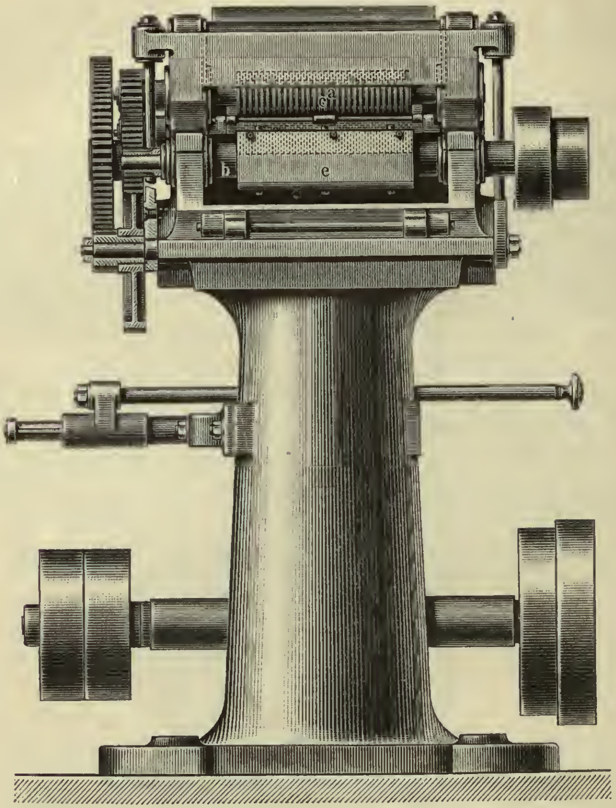


Fig. 230.—One-10th full size.

The sheets issuing from the rolls are of about the consistency of india-rubber. They are cut by means of cutting machines, first into strips, and then into small square plates. The most primitive form of a cutting machine is that in which a guillotine knife goes up and down vertically, whilst the sheet is moved forward in a similar manner to a tobacco-cutter.

The cutting machine now almost universally used has circular knives which overlap each other and cut the sheet of powder into longitudinal strips, whilst a horizontal knife rotating against a cutting edge chops the strips into cubes. In Fig. 227 a cutting machine made by Bolle and Jordan of Berlin is shown in section and in Fig. 228 in plan. This machine has been introduced in large numbers in the Royal Prussian Gunpowder Factory at Spandau. Each machine has on a frame two cutting gears, and the driving shaft on the one is a little longer than that of the other in order to let the belt pass. On the shafts (b and b_1) placed parallel to each other the cutters (b_{11}) are fixed. A number of disc-shaped knives (a) are put on and held at suitable distances apart by washers (c) of smaller diameter, and kept in place by end-nuts (c_1). The knives slightly overlap each other, and in front of them is a comb (g) which prevents the sheet of powder from being carried round by the knives, and at the same time serves as a guide for the strips. These then pass over the

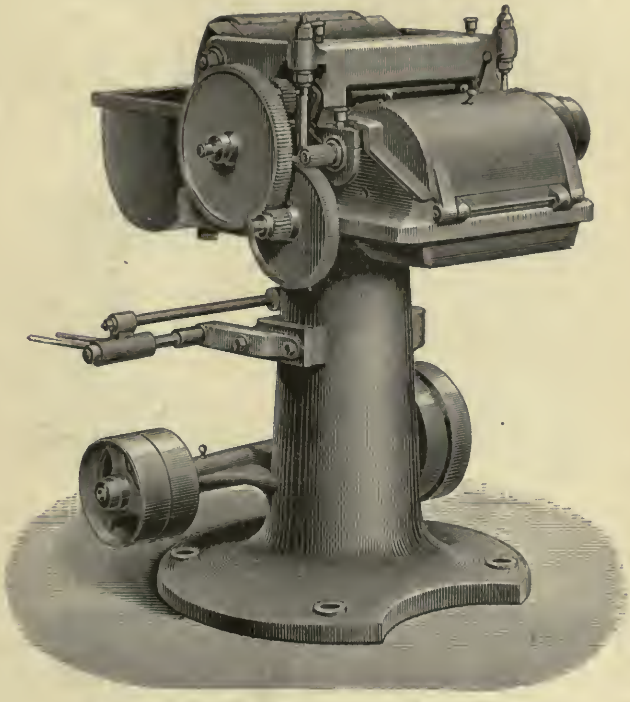


Fig. 231.

cutting edge, and meet a rotating knife shaft (e). On this there are four horizontal steel knives (f) fixed in a manner which is sufficiently intelligible from the drawing. The movement of the pulley is transmitted first to the knife shaft (e), and then by a system of cog-wheels to the shaft b, and then by another cog-wheel to the shaft b_1. In order to introduce the sheet of powder, a box is hung over the whole length of the rolls, and in it the sheet unrolls gradually. To prevent the powder from being thrown about, a brass casing with a discharge funnel is fixed over the knife shaft.

Another cutting machine which has recently come more to the front is made by Ernst Schiess, of Düsseldorf-Oberbilk, and illustrated in Fig. 229 in cross-section, in Fig. 230 in longitudinal section, and Fig. 231 in view. Here also there are circular knives (a) kept apart by washers (c) and fixed on two shafts (b and b_1). There is also the comb (g) and the cutting edge (d) provided. The shafts (b and b_1) are not at the same level, but one higher than the other. By this means the strips of powder run in an uninterrupted line towards the horizontal cutting knife, which in this case consists of a special casting on to which steel knives (f) are fixed by screws. By turning the screw (h) which gears with the brass nut (i), the cutting edge can be brought forward or backward. The knives (f) can also be adjusted by the screws (i and j) in case they should have been worn. The upper knife shaft revolves in a bearing which is fixed to the upper casting of the machine. This can be turned on a hinge (m), but when the machine is working is held fast by a vertical bar (n) and nut (o). The hole through which the bar passes is

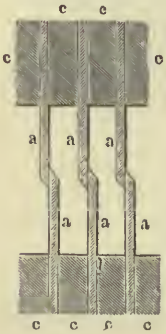


FIG. 232.

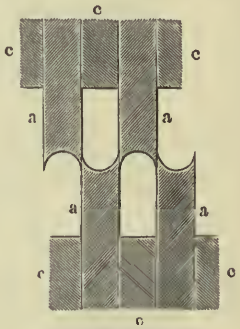


FIG. 233.

conical, while the tightening nut and the boss on the bar are spherical. If the nut (o) is tightened or loosened, the upper part will be lifted or lowered on its hinge. In this way the distance between the two knife shafts can be regulated according to the thickness of the powder sheet. The trough to take the sheet of powder has the same form as with the machine previously described. The horizontal knife is in a box over which a cover (p) is cast on the frame of the machine with a lid that can be turned round a hinge (q) in order to give access to the machine. The powder falls into a barrel placed under the outlet funnel.

Both machines work on the same principle, but that of Bolle and Jordan has two cutting arrangements, whilst the Schiess machine has only one. On the other hand, the machine of Schiess is more compact, more readily adjustable, and all parts are more accessible.

The circular knives were originally bevelled, as in Fig. 232, but it has been shown that this form gets clogged more easily by the powder. To prevent this the comb (g) was adopted, but later it was shown that the best form of knives is U-shaped, as illustrated in Fig. 233, which has been long in use in machines for cutting playing-cards. With this shape the strip of powder does not adhere to the knife, and is not pinched in sideways. The hollow groove also gives a good point of contact for the comb.

In making square flakes there is of course no waste. Round flakes are made very rarely, and are cut from cords. The flakes coming from the cutting machines still contain a small quantity of the solvent, and to completely eliminate this they are placed in drying-houses constructed in the usual way. The drying is done slowly at a temperature of about 104° F., and lasts from three to four days.

With some powders part of the solvent is left in them to reduce the rapidity of combustion. Sometimes this is also obtained by mixing some alcohol with the acetone. With well-made powders and careful selection of the nitro-cellulose, any remaining traces of the solvent are a disadvantage, because on prolonged storage some evaporates off, and the rapidity of combustion of the powders will increase. On the other hand, the rate of combustion can be reduced by a glazing of black-lead, which can be given very simply by glazing in a drum with a little fine black-lead. This gives the powder a silver grey to dark grey colour. Sometimes also the powders are coloured with aniline to increase their stability under the heat test, and also to obtain a more uniform coloration.

Flake powders made of gun-cotton only are used in various countries. For instance, the German, French, Austro-Hungarian, and Swiss rifle powders, and the "Wetteren powder" (L. 3, Librecht's powder), the Troisdorf powder, and Von Förster powder, are all pure gun-cotton powders in flakes.

(*b*) GRANULATED POWDER FROM WALSRODE.

In addition to flake powders, Wolff and Co. of Walsrode make granulated ones also, which are more especially adapted for sporting purposes. They are made by mixing the nitro-cellulose with acetic ether, and then treating them in a kneading machine for about an hour to effect complete solution. About a quarter volume of the mass of water at 140° F. is then added and the kneading continued whilst steam is introduced into the mixture. The combined effect of the kneading and steam is to break up the gelatine into fine grains within a few minutes, the grains are then transferred to a special vessel and treated with boiling water until the solvent is completely evaporated. The grains are freed from water by pressing, centrifugaling, and drying, and are finally sifted.

4. POWDERS MADE FROM NITRO-GLYCERIN AND NITRO-CELLULOSE.

Four such powders are at present made:—Nobel's ballistite, which has soluble gun-cotton as its base; cordite, which is made from insoluble gun-cotton; amberite, which contains a mixture of both kinds of gun-cotton; and Leonard powder, which contains also non-nitrated organic components.

(*a*) BALLISTITE.

Ballistite consists of about 40 per cent. of collodion cotton and 60 per cent. of nitro-glycerin, with the addition of about 1 per cent. of aniline or di-phenyl-amine. According to the patent specification of Mr. Alfred Nobel for this powder, 1 part of collodion cotton is put into a vessel, with 6 to 8 parts of nitro-glycerin, at a temperature of between 43° and 46° F. The air is exhausted from the vessel by means of an air-pump, in order to bring the nitro-glycerin and collodion cotton into intimate contact with each other. Part of the nitro-glycerin is removed in a press or centrifugal machine, so that equal quantities of nitro-glycerin and nitro-cellulose remain in the mixing vessel. The cake remaining from the compression is broken up and heated to a temperature of between 140° and 194° F., when the collodion cotton dissolves in the nitro-glycerin. The mass is then rolled out at the same temperature into thin sheets, and either cut into flakes, or built up into thicker plates by rolling several thin ones together and then cut. To ensure the chemical stability from 1 to 2 per cent. of di-phenyl-amine is previously added to the nitro-glycerin.

According to the English patent, the addition of camphor to the powder was also contemplated, but was subsequently omitted.

At present ballistite is made by an improved process patented by Messrs. Lundholm and Sayers (English patent, 10,376, dated June 26, 1889). According to this process the nitro-glycerin and collodion cotton are put into hot water and stirred up together in it by compressed air, but it is also sufficient to put the ingredients into water and to keep the mixture at a temperature of about 140° F. for some days; frequent stirring, however, quickens the gelatinization. The powder so mixed is freed from the bulk of the water by pressing, and is then put through steam-heated rolls, the temperature of which is kept at between 122° and 140° F. As the dough passes repeatedly through the rollers, complete solution of the nitro-cellulose in the nitro-glycerin is effected by the simultaneous pressing and heating, and at the same time a homogeneous sheet, free from blisters, is obtained, whilst the water is eliminated, partly mechanically and partly by evaporation.

On rolling ballistite and other smokeless powders small local explosions frequently take place. It is improbable that, as was originally suggested, they are due to the cracking of air blisters; on the contrary, they are probably due to the explosion of particles of undissolved collodion cotton, which at a temperature of 140° F. is very sensitive to friction and shock caused by the pressure of the rolls. This view is confirmed by the fact that on some places traces of burning are to be seen on the sheets, but such explosions are as a rule quite harmless. The finished sheets are cut into flakes in cutting machines of the usual type.

Sometimes, in Italy for instance, ballistite or other smokeless powders are made into cords or round flakes. The cord-shaped ballistite is known in Italy as "Filite." The cords can be made in the machines described further on in connection with cordite, and they can be cut into round flakes by a machine similar to the one illustrated in Fig. 234, which is made by Werner, Pfleiderer and

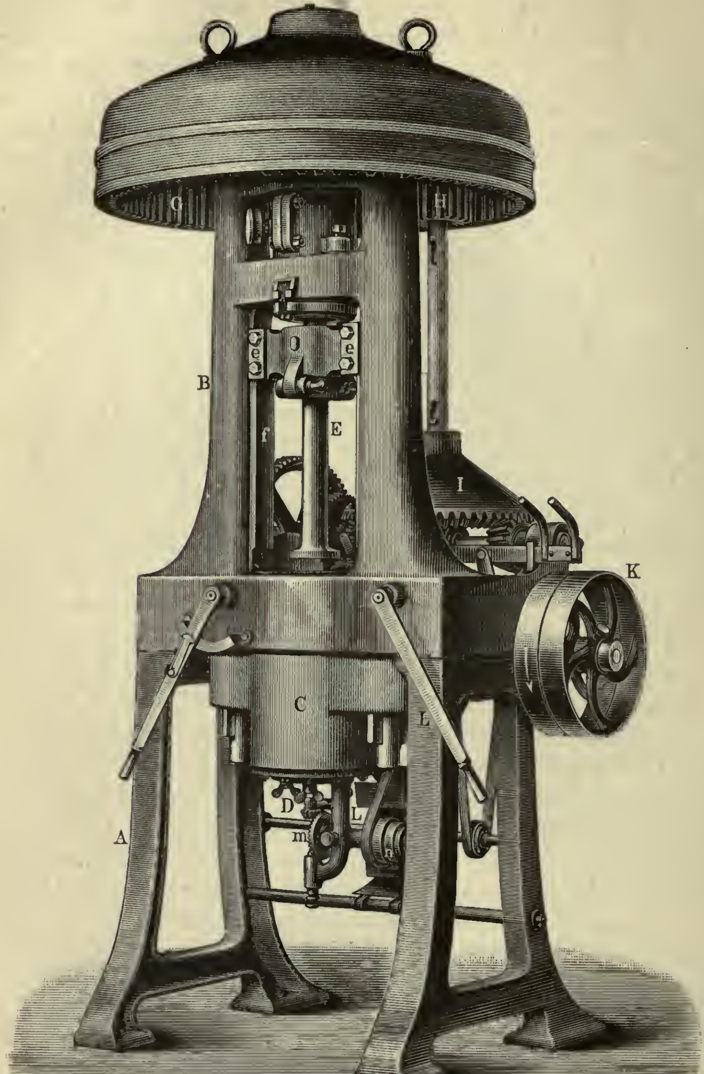


Fig. 234

Perkins, like those for making certain kinds of pastry. It consists of an upper frame (B) fixed to a strong base (A), below which the pressing mould (C) is placed. The bottom of this pressing mould is closed by a plate (D), in which there are a number of fine conical holes. A piston (E) works the mould, and is guided by means of slide blocks (e) and two bars (f) on the frame. The piston-rod terminates in a screw on the top which gears with a nut, and has attached to it the large cog-wheel (G), which also serves as a fly-wheel and which is driven by a small cog-wheel (H). This in turn is driven from a pulley (K) by a train of cog-wheels (I). The powder dough is put into the mould and pressed through the holes in the plate by means of the piston. On the plate there is a bracket (L) carrying a pair of cog-wheels (m and m_1). The larger one is driven by a coned pulley (n) from the main shaft, the smaller one has a flat two-armed knife attached to it, which is thus put in rapid motion by the cog-wheel, and thereby cuts the cord into fine flakes as it issues from the mould. The thickness of the flakes can be regulated by the coned pulley, since it is clear that the slower the knife rotates the thicker the cut, if the rate of pressing remain the same. The machine can also be driven by a reversing gear, as described on p. 242. As soon as the piston has got to its lowest position, it is stopped automatically by a striking gear on the machine.

By turning the lever (L) on the right, the piston is lifted, which is done more rapidly than the down-stroke. The piston has thus a quick return motion, and as soon as it arrives in its highest position, the machine is automatically stopped. In this position the piston can be swung horizontally round on a hinge and the mould filled. Another automatic arrangement prevents the machine from being put into motion before the piston has been again placed in its proper position over the mould. When the dough is rather soft, cutting it into flakes is not so simple an operation, and it is necessary to carefully regulate the consistency of the dough to prevent the flakes being crushed out of shape.

The colour of ballistite is from a light brown to a chocolate, and it has a density of about 1·6. It feels like hard rubber to the touch, and can easily be cut with a knife. In general it is not sensitive to moisture, and powder which has got damp can be easily dried. When heated over normal temperatures, *i. e.* to about 40° or 50° C., like all smokeless powders it gives higher velocities and greater gas pressures. On account of its great heat of combustion, ballistite affects the rifling of guns.

(b) CORDITE.

Whilst in Nobel's ballistite soluble nitro-cellulose is dissolved by heat and rolling, without the aid of a solvent, Sir Frederick Abel and Professor James Dewar patented, on behalf of the English Government, a smokeless powder (English patents, No. 5614 of April 2, 1889, and 11,664 of July 22, 1889) which contains insoluble nitro-cellulose, and thus consists of pure gun-cotton intimately mixed with nitro-glycerin made by aid of a solvent (acetone).

It can hardly be doubted, and in the patent action which Nobel's Explosives Company instituted against Dr. W. Anderson, the Director-General of Ordnance Factories, it was shown fairly conclusively, that the invention of cordite was to a great extent suggested by Nobel's ballistite patent. Amongst the many powders brought under the consideration of the Explosives Committee were the earlier forms of ballistite, which at that time still contained camphor. The unsatisfactory result obtained with it, and the objection of the War Office chemists to camphor as an ingredient, led Sir Frederick Abel and Professor Dewar to invent a powder which was free from these defects. In a certain sense the addition of nitro-glycerin to a solution of insoluble gun-cotton in acetone is to be employed in the explosive patented by Mr. Hiram Stevens Maxim (English patents, No. 18,663, December 20, 1888, and No. 4477, March 14th, 1889). According to the first it is intended to add from 2 to 10 per cent. of nitro-glycerin to a solution of gun-cotton in acetone, but in the complete specification it was left out altogether. According to the latter patent, oil, preferably castor-oil, should be added to "compounds of dissolved gun-cotton and nitro-glycerin."

The composition of cordite has, after many experiments, been fixed as follows:—

	PARTS.
Nitro-glycerin	58
Gun-cotton	37
Vaseline	5
	100

which are dissolved by 19·2 parts of acetone.

Cordite for blank ammunition does not contain any vaseline.

The nitro-glycerin to be used should not, when exposed in a desiccator over calcium chloride for 16 hours, lose more than 0·5 per cent. of moisture. 20 grammes of nitro-glycerin shaken with 50 cubic centimetres of water and 1 cubic centimetre of methyl-orange solution should not on titration with tenth-normal sulphuric acid show more than 0·1 per cent. of alkalinity, calculated as sodium carbonate. 50 grains of nitro-glycerin taken from 100 grammes that have been filtered through a neutral filter to eliminate any excess of moisture, should stand a heat test of 180° F. for 15 minutes. Further particulars on this subject will be found in the Appendix.

The gun-cotton used is the same as that made in the Royal Gunpowder Factory at Waltham Abbey, which contains as a rule from 10 to 12 per cent. of soluble gun-cotton. The presence of more than 0·6 per cent. of mineral compounds, or less than 12½ per cent. of nitrogen by the nitrometer test, is not allowed, and it should stand the heat test for 10 minutes.

The proportions of cordite were determined from the properties of the gun-cotton made at Waltham Abbey. It may, therefore, be assumed that the powder contains about 4 per cent. of soluble gun-cotton. If pure tri-nitro-cellulose were used, the proportions would have to be altered to allow for the more rapid action of pure gun-cotton.

In making cordite, the weighed quantity of nitro-glycerin is poured over the previously dried gun-cotton, and the whole carefully mixed by hand. The mixture is then put into a kneading machine surrounded by a water-jacket, and the acetone poured on to it. The mass is then kneaded for 3½ hours. To prevent evaporation of acetone, a glass or wooden cover is put over the machine. When the first kneading of 3½ hours is finished, the vaseline is added, and the kneading continued for another 3½ hours. During the kneading the acetone gradually penetrates the mixture of nitro-glycerin and gun-cotton, and dissolves them both.

Through the continued kneading a uniformly tough paste is formed from these three ingredients, which gradually assumes a buff colour. During the kneading the mass becomes heated, and therefore cold water is run through the jacket of the machine to prevent heating above the normal temperature and consequent evaporation of acetone.

The cordite paste is next formed into more or less thin cords similar to the pastry known as "Spaghetti." The action of the machine used for doing this is similar to the one used for making certain forms of pastry, the dough being pressed out by mechanical or hydraulic pressure through a fine orifice in a continuous thread.

The cordite paste is first treated in a preliminary press to make it as compact as possible in the mould and to free it as

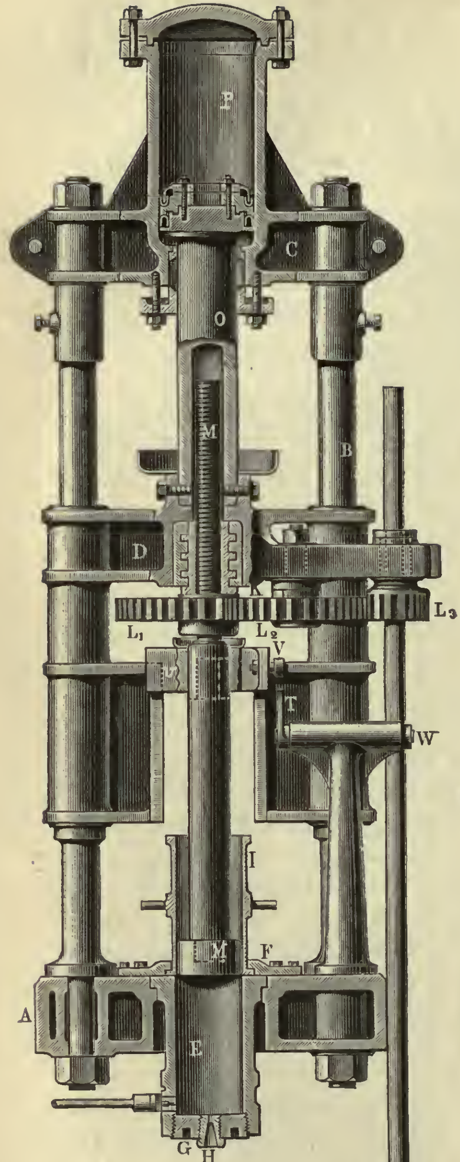


FIG. 235.

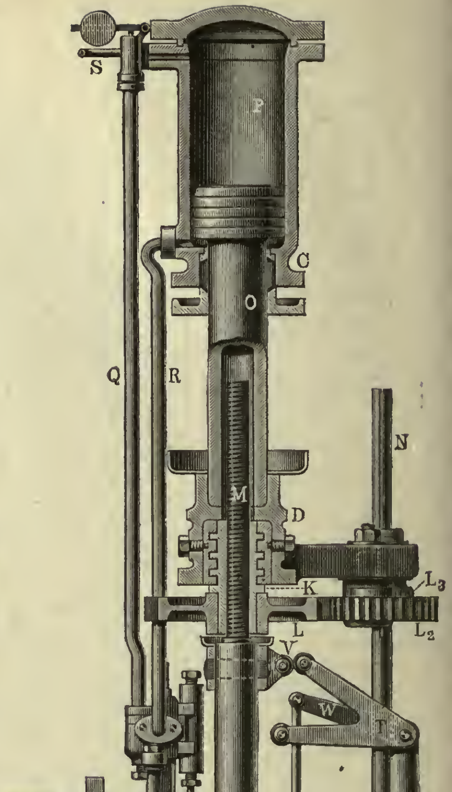


FIG. 236.

much as possible from air. The preliminary presses are worked either by hand or by hydraulic power.

For small quantities hand-presses are sufficient. The cordite is put into the mould in layers by means of a wooden tool, and then put under a piston which is drawn down by means of a long lever. Larger quantities are treated in a similar manner by hydraulic pressure.

The filled mould is at once placed in the cordite press proper, which is shown in Figs. 235 and 236 taken from the Patent Specification (No. 11,667 of July 22, 1889). The base plate (A) carries two columns (B—B) which are connected with each other by a cross-head (C), and form guides for the table (D). The base plate (A) contains the mould (E), which is held firmly by clamps (F). On the lower end of the mould there is a disc block (G) which screws in and carries a die (H) corresponding to the thickness of the cord to be made. A mould (I) which has been filled in the first press is put on the top of the pressing mould. The table carries the pressing gear, consisting of a nut (K) which is rotated by the cog-wheel (L) attached to it, the wheel (L_2), and the pinion (L_3). The latter can slide on a feather key on the shaft (N). Through N a screw spindle (M) connected with the pressing piston (M′) passes. It will be seen therefore that as the cog-wheels and the nut revolve, the screw forces the piston down into the mould, and presses out the mass from the die in the shape of a fine cord.

In order to prevent the pressure in the mould from becoming too great in case of the die getting blocked or from any other cause, a hydraulic buffer is provided. This consists of a hydraulic piston (O) attached to the table, and the screw passing up through the centre of it. This piston moves in a cylinder (P) cast in one piece with the cross-head. Hydraulic pressure is admitted simultaneously from the top through the pipe Q, and from the bottom through the pipe R. Since the free surface of the piston is smaller on the bottom than on the top, the piston is subjected to a pressure proportional to the difference between the two surfaces. If the pressure in the mould exceed that allowed, which can be regulated by a safety-valve (S) at the head of the pressing cylinder, the table rises instead of the piston being forced down. The press is fitted with an automatic reversing gear, which reverses the motion of the piston when all material put in the mould has been pressed out. This is done by the tappet (T). A tappet (V) fixed on the guide (U) strikes one arm of the double lever (T, W) as the piston gets to its lowest point and throws over the belt-striking gear, with which it is connected by the rod (X), so that one driving-belt is thrown on to the loose pulley and the other on to the fast pulley, which reverses the motion of the piston. It will be easily seen from the drawing that in the same way the machine can be brought to a standstill.

A machine on a similar principle is made for the manufacture of the thinner varieties of cordite. The cord issuing from the press is reeled on to drums made of sheet-metal, which are mounted in a reeling-machine similar to those used for yarn.

The cordite reeled on drums is taken to a drying-house to completely expel the acetone. During the pressing the powder has already lost part of its acetone, but the bulk is persistently retained by the tough mass.

In the drying-houses there are simple racks on which the drums are laid in rows. On the floor steam pipes are laid, and on the two gable ends of the house there are exit openings for the vapours.

The temperature of the drying-houses is about 100° F., and according to the thickness of the cord, the drying takes from three to eight days. With the thicker kinds of powder the cords are cut to the required lengths by a machine, and placed on trays in the drying-house. The thin cords after drying are sent to the ammunition factory, where they are reeled off the drums and cut into suitable lengths.

For blank cartridges, as has been previously mentioned, cordite made without vaseline is used in the form of chips instead of cords. These are made by cutting up cords. According to the colour of the vaseline used, cordite has a light brown to dark brown colour. Its consistency is that of hard rubber, the fractured surface is yellowish, and even after prolonged storage the cords are still sufficiently elastic to be bent and then recover their original form. It has a slight smell of acetone. If ignited, it burns with a strong flame, which can be extinguished by blowing strongly on to it.

(*c*) AMBERITE.

Curtis and André have patented a powder under this name, consisting of nitro-cellulose and nitro-glycerin. The nitro-cellulose used is intentionally a mixture of soluble and insoluble gun-cotton. As a suitable proportion, Curtis and André mention 44 parts of tri-nitro-cellulose, 12 parts of di-nitro-cellulose, and 40 parts of nitro-glycerin. They first of all form the nitro-cellulose into grains, which are then treated with a solvent that only dissolves the di-nitro-cellulose; thus, for instance, sulphuric ether with a little alcohol. The nitro-cellulose is made by adding a suitable quantity of soluble gun-cotton to the insoluble gun-cotton of commerce, which as a rule already contains about 12 per cent. of soluble gun-cotton.

By treating the grains with the solvent, a kind of cement is formed throughout the whole mass which firmly binds the gun-cotton fibres, in addition to hardening the surface. By this means the rate of combustion of the powder is considerably lowered.

Another variety of amberite is made which is said to consist of nitro-cellulose, barium nitrate, and solid paraffin.

(*d*) LEONARD POWDER.

M. E. Leonard, of Manchester, U.S.A., mixes 150 parts of glycerin, 50 parts of gun-cotton, 10 parts of lycopodium, and 4 parts of powdered urea crystals with acetone in tightly-closed pots, and allows the mixture to stand therein for 12 hours. The solvent is then evaporated, and the mass granulated in the usual manner.

5. POWDERS CONSISTING OF NITRO-CELLULOSE AND NITRO-DERIVATIVES OF AROMATIC HYDRO-CARBONS.

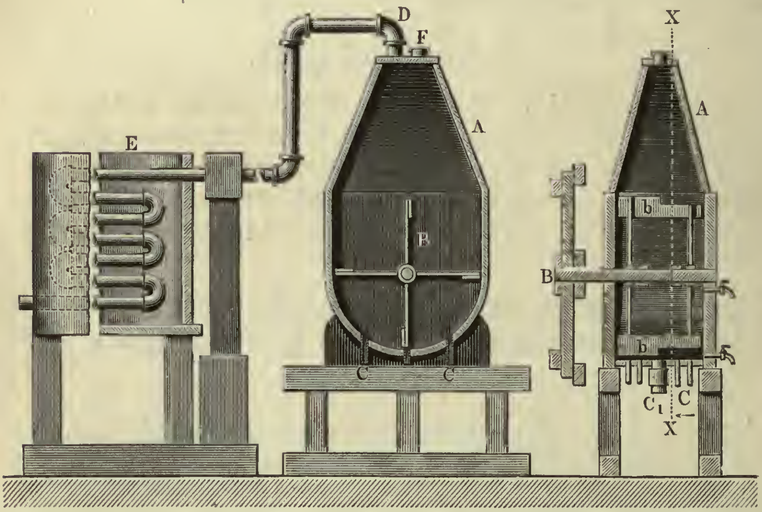
(*a*) INDURITE.

This powder, which consists of nitro-cellulose and nitro-benzene, was patented by Professor C. E. Munroe, of Newport, U.S.A. (English patent, No. 580, January 11, 1893). The inventor's chief aim is to obtain a gun-cotton perfectly free from soluble nitro-cellulose, and to this end he treated the gun-cotton first with methylic alcohol, using for the purpose an apparatus in which the methylic alcohol is repeatedly distilled off, and after condensing and cooling again runs on to the gun-cotton, until all soluble nitro-cellulose is eliminated. He dissolves the nitro-cellulose so made in nitro-benzene, forms it into sheets under rolls, and cuts it into grains. This dissolving in nitro-benzene he calls "colloidizing."

For each part of gun-cotton he uses about 0·9 to 1·8 parts of nitro-benzene. The powder so formed is then treated with hot water or steam, by which it is hardened or "indurated," whence its name. The hardening of this powder is probably only due to the excess of nitro-benzene being evaporated.

(*b*) Du Pont Powder.

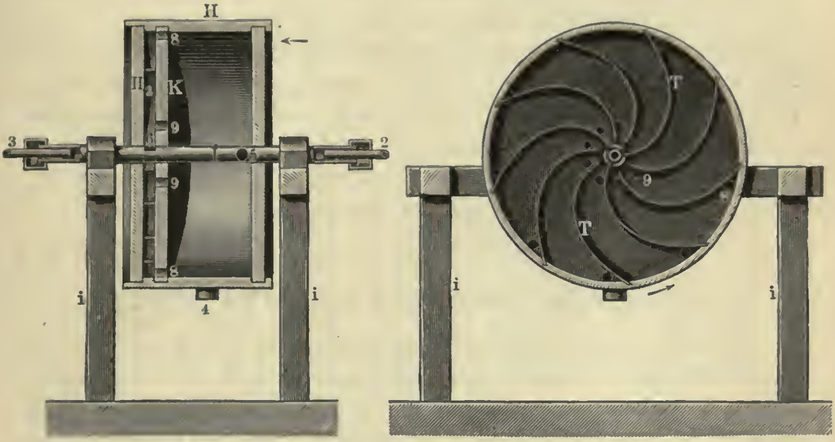
F. C. and P. S. Du Pont, of Wilmington, U.S.A., have patented a smokeless powder (English patent, No. 15,865 of August 22, 1893) consisting of nitro-cellulose and nitro-benzene, or another suitable solvent, which is granulated by a special process. They also, like Lundholm and Sayers, suspend the nitro-glycerin and the nitro-cellulose in a liquid, and use the greater affinity of the solvent for gun-cotton than water for effecting the solution. The powder is made as follows:—



Figs. 237, 238.

The apparatus used is shown at A in Figs. 237 and 238. It is fitted with stirring paddles (b), which rotate on the shaft (B). They are somewhat narrower on one side than on the other, in order to push the mass from one side of the apparatus to the other. Steam inlet pipes, with orifices of small diameter, and an exit pipe (D) going to the condensing arrangement (E) are also provided. The apparatus is filled through the opening (F), and 90 litres (20 gallons) of water are first put in. With certain kinds of powder the water has saltpetre dissolved in it. Six kilogrammes (13·2 lbs.) of finely-divided gun-cotton are then added, and stirred up by means of the paddles until the gun-cotton is evenly suspended in the liquid, when about 18 kilogrammes (39·6 lbs.) of nitro-benzene are run in. By the action of the solvent on the suspended gun-cotton, a plastic flocculent mass is formed, which with the assistance of slow stirring gradually becomes granular. On continued stirring, grains of decided form, but of a rather soft, paste-like consistency, are formed, which contain a good deal of water and an excess of the solvent. By introducing steam the solvent partly distils over into the condensing apparatus through the exit pipe (D). By this means the grains become harder and more solid. Samples can be drawn from the interior of the apparatus by means of small taps.

The apparatus is now emptied through the discharge opening (C_1) and the grains put into a rotating barrel, which is illustrated in Figs. 239 and 240. The barrel (H) has a tubular axis (I) supported on the frame (i) by bearings. Steam is introduced through the axis by means of the pipe 2, and passes out through the pipe 3. There is a false bottom (K) in the barrel near the end (H_2). In this two circular rows of holes are made—the outer one to separate the water from the contents of the barrel, and the inner one to take away the steam.



Figs. 239, 240.

Between the two ends (K and H_2) a series of cycloidal guiding blades (T) are arranged converging towards the axis. The grains are put into the rotating barrel, and revolved for some time, without the admission of steam, in order to make the grains more solid and round; the barrel is then partly filled with water and revolved in the direction in which the cycloidal scoops will not carry off the water. At the same time steam is introduced through the axle and through the hole (5) in it. The steam passes through the openings (8 and 9) in the peripheries into the space between the two ends (K and H_2), and from thence out through the opening (1) in the axle and the tube (3). At the same time the steam carries away with it any excess of solvent.

As soon as the operation has sufficiently advanced, the direction of movement of the barrel is reversed, whereby the water, and also the condensed water of the steam issuing through the openings on the outer periphery, is taken up by the scoops and carried out through the opening (6). The steam is still left on in order to further penetrate the powder and entirely drive out the excess of solvent. By revolving the powder in a space filled with steam, its density and solidity are materially increased.

If powder with a slow rate of combustion is to be made, from 2½ to 10 per cent. of a "taming" agent, such as nitro-resin or nitro-turpentine, can be dissolved in the solvent before it is added.

In another patent taken out on the same date Du Pont recommended converting the nitro-benzene into an emulsion before working it in the stirring apparatus.

(*c*) THE SMOKELESS POWDER COMPANY'S POWDERS.

The Smokeless Powder Company of Warwick sell various smokeless powders which are not patented, and the composition of which is kept secret, under the names of Rifleite, S. S., S. R., S. K., S. V., and S. B. Powder. From the definition given of these powders in H. M. Inspectors of Explosives annual report, and also from what has been made known by analysis of the powders and the communications at the meetings of the Company, they seem to consist solely of soluble wood nitro-cellulose mixed with saltpetre and nitro-benzene, and are probably granulated in a similar manner to the E. C. powder.

The powders are already used for sporting purposes.

(*d*) PLASTOMENITE.

Hermann Güttler of Reichenstein has found that nitro-cellulose dissolves completely in melted nitro-hydro-carbons—those made from benzene, phenol, toluol, naphthalene, etc., for instance—and he makes use of this property to produce a plastic mass which can be converted either into explosives, or, with the addition of suitable mineral colouring matter, into a stone-like mass which can be worked accordingly, and is applicable to a variety of purposes.

The smokeless powder of Güttler is a fused mass of di-nitro-toluene and wood nitro-cellulose. The di-nitro-toluene is melted in a water-bath, and about one-fifth part of nitro-cellulose gradually added. The mass is formed into fine grains. Its colour varies between light and dark brown, its gravimetric density is under 0·1, and the powder can be exposed to the heat test for a surprisingly long time without giving any signs of instability.

It has been used more especially for sporting purposes, and is much favoured in Germany. It seems that, like all granulated powders, it absorbs moisture a little more easily than flake powder, but there is not yet sufficient experience available. The shooting results with plastomenite are very regular and good, and at a competition in Bucharest, in 1893, it seems to have answered the conditions laid down by the Government very well.

6. OTHER KINDS OF SMOKELESS POWDERS.

(*a*) Nobel's Nitro-Starch Powder.

The Dynamit-Actien-Gesellschaft Nobel have patented a smokeless powder consisting of 70 to 99 parts of nitro-starch, and 30 to 1 part of di-nitro-benzene. It does not seem to have been practically used as yet.

(*b*) Kalliwoda von Falkenstein Powder.

A peculiar powder has been patented by Joseph Kalliwoda von Falkenstein, of Vinkovce (Croatia). He puts 2 to 4 parts of finely-divided cellulose into a solution of 1 part of potassium permanganate in 10 parts of water, after having cooled the solution to about 12° or 14° C. In this way amorphous cellulose is formed mixed with manganese di-oxide, which precipitates as a brown mass. The product has an alkaline reaction, and is washed until neutral, then mixed with 1 part of nitric acid of 1·3 specific gravity, and allowed to stand for 12 hours. After this the mixture is heated in a water-bath to 70° C. until all manganese di-oxide is converted into manganese nitrate, and the amorphous cellulose separated as a white precipitate.

This cellulose is formed into a cake by pressing, then pulped and washed, and then again pressed and rubbed through sieves and dried. The horn-like cellulose formed after drying is then ground into a fine powder. This is then nitrated with an acid mixture consisting of 1·5 to 2·1 parts of nitric acid of 1·40 to 1·55 specific gravity, and 4·5 to 6·3 parts of sulphuric acid of 1·85 specific gravity. The nitration is done with continuous stirring at between 5° and 8° C., and the temperature must on no account rise above 30° C. The nitration is finished in from 48 to 72 hours. After separating the acid mixture, the nitro-cellulose is washed and allowed to stand in a solution of from 1 to 2 parts of potassium permanganate in 10 parts of cold water, for 10 to 12 hours with frequent stirring. Then nitric acid up to 1 part of the weight of potassium permanganate is added, and the whole again allowed to rest for 6 hours, after which it is again heated on a water-bath to 70° C., until the development of gases ceases. Nitric acid of 1·45 specific gravity is again added, and the mixture once more heated in the water-bath to between 70° and 80° C., until all manganese di-oxide is converted into nitrate, and then the nitro-cellulose is washed first in hot and then in cold water, and finely ground. To this from 1 to 3 parts of gum tragacanth are added, and the whole worked up into a thick paste in a stamp-mill, after which the mass is pulverized between rolls and the grains sifted, to be finally glazed in a drum.

The real idea of the whole is not at all clear, and at any rate the process is so complicated that carrying it out on a large scale cannot be thought of.

(*c*) KOLFITE.

Hubert Kolf of Bonn nitrated vegetable waste, such as malt grains, malt waste, beet-root cuttings, etc., and sulphonated them with alkaline sulphates under a pressure of five atmospheres. He then mixed them with saltpetre and soaked them in a solution of di-nitro-benzene.

In a later patent Kolf also proposed making the nitrated materials into a gelatinous mass by the addition of nitro-molasses, nitro-sugar, or nitro-glycerin with the aid of a solvent (acetic acid, acetone, etc.), and then evaporating off the solvent, passing the mass through rolls, and cutting it into flakes.

(*d*) FRENCH POUDRE PYROXYLÉE.

The French Government issues for general use a smokeless sporting powder under the name of poudre pyroxylée (pyroxilated powder), which has the following composition approximately:—

	PARTS.
Soluble gun-cotton	28
Insoluble gun-cotton	37
Barium nitrate	29
Potassium nitrate	6
	100

10 kilogrammes (22 lbs.) of the materials are first mixed by hand, and then for 45 minutes in incorporating-mills, with the addition of 40 per cent. of water. After this the materials are pressed through a sieve of 2·5 millimetres ($\frac{1}{10}$ inch) mesh, and dried in a drying-house to under 1 per cent. moisture.

After drying 65 per cent. of ether is added to the mixture, and the paste obtained granulated on a sieve made of perforated sheet-zinc with holes of 1·8 millimetres diameter. The resulting mixture of grains and dust is treated for 45 minutes in a wooden drum 0·60 metre (2 feet) long and 0·40 metre (1 foot 4 inches) in diameter, at 27 revolutions per minute, then moistened with 40 per cent. of water, and brought back to the drying-house. After drying it is sorted on sieves into grains between 1·6 and 1 millimetre in size. The grains obtained are glazed with the addition of 15 per cent. of ether in quantities of 10 kilogrammes at a time in a copper drum 0·40 metre long and 0·60 metre diameter, at 10 revolutions per minute. The ether is added in the form of a fine spray through an atomizing apparatus. The powder is again rubbed through a sieve with 1·8 millimetres holes, dried and sorted into grains of between 1·6 and 1 millimetre diameter. In this way about 33 per cent. of the grains obtained are serviceable.

The waste is treated for 30 minutes in incorporating-mills and sifted through a zinc sieve with 2½ millimetres holes. The mixture of grains and dust so obtained is brought into the drying-house, and like the powder is treated with 50 per cent. of ether, sorted, and the grains glazed, again dried, and classified. The resulting dust is constantly re-worked until it is completely exhausted.

The finished powder, as sold, is a mixture of 1 part of grains of the original making and 2 parts of those made from working up the waste.

The poudre pyroxylée has a slightly yellowish colour, with regular and hard grains, of which from 2150 to 2300 go to 1 gramme. Its gravimetric density is between 0·480 and 0·530, and its real specific gravity at least 1·580. It stands the heat test for about 20 minutes. On being exposed in a room saturated with moisture for four days, the powder only absorbed a quite insignificant quantity of moisture (between 0·19 and 0·37 per cent.). The average muzzle velocity (V_{15}) it gives is 250 metres per second, and the pressure about 800 atmospheres.

(e) FRENCH "POUDRE J."

More recently the French Government made a sporting powder which was invented by M. Bruneau, which consists of 83 parts of gun-cotton and 17 parts of ammonium bi-chromate. Nothing has been published about its method of manufacture, but it is probably similar to that of the poudre pyroxylée. It is of brown colour, much harder than black powder, and has angular grains of irregular shape.

It is made in four sizes:—No. 0 for rifles and carbines, No. 1 and No. 2 for ordinary sporting purposes, and No. 3 for small-calibre pistols and revolvers. The following table shows the characteristics of the various powders:—

	No. 0.	1.	2.	3.
Number of grains per gramme	300	600	1250	2000
Real specific gravity ...	1·620	1·590	1·560	1·435
Gravimetric density ...	0·740	0·710	0·680	0·530

The average moisture in the powder as sold is 3 per cent. Before it is issued it is tested for muzzle velocity and gas pressure. The rifle used for this purpose has for the Nos. 0, 1, and 2 powders a calibre of 16 with cartridge cases 65 millimetres (2½ inches) length. The trials these powders have to pass are as follows:—

KIND OF POWDER.	No. 0.	No. 1.	No. 2.
Weight of charge—grammes ...	2·80	2·80	2·60
Velocity—metres	220 to 230	240 to 250	245 to 255
Maximum gas pressure—kilogrammes per square centimetre	350	500	500

The "Poudre J." gives a slightly dark smoke which disappears rapidly, and is less disagreeable than that of poudre pyroxylée.

The residue consists of a fine dark green dust which does not adhere to the barrel. The powder absorbs a good deal more moisture than the poudre pyroxylée, but made up in cartridges it is quite equal in this respect to the latter. After prolonged drying and on increasing the charge by 0·2 gramme, the powder did not give more than 600 kilogrammes pressure, and thus for rifles of 16 calibre it is still below the limit permitted.

7. PROPERTIES OF SMOKELESS POWDERS.

According to the purpose for which they are intended, smokeless powders will have somewhat varying properties, and since they are mostly made of highly explosive materials, means must be adopted to reduce the rapidity of combustion. Whilst for large guns a very slowly burning powder, which will only gradually develop its full force and reach its maximum just as the projectile leaves the barrel, is required, it is on the other hand desirable that sporting powders should have a rapid combustion in order to give the shot sufficient penetration and a good pattern.

Hand-in-hand with this, small gas pressure should be developed and as high a muzzle velocity as possible. For military purposes a powder should further develop considerable force per unit of weight, because this gives lighter cartridges, and the soldier can carry a greater number of them. With sporting powder it is again desirable that the charge should have a volume approaching that of black powder, since with the large calibre of sporting guns the cartridge case should be filled as nearly as possible to the same height as with black powder, and also because overcharging the cartridge could not involve such a degree of danger of bursting the barrel as would be the case with powder of high gravimetric density. For military rifles and guns, great straightness of flight is of importance, because thereby the soldier is in a position to shoot with safety at varying distances without it being necessary to constantly adjust the sights of the rifle.

Since it is not possible to obtain perfect absence of smoke on combustion of powder, as small a development of smoke as possible must be aimed at, but it is of the highest importance that all the products of combustion be innocuous.

The residue a powder will leave also requires special consideration when determining the composition. The stability of the powder at varying temperatures and during prolonged storage under varying climatic conditions, and hand-in-hand with this, small alteration of shooting results, and finally a certain measure of absence of danger during handling and with the varying accidents of warfare, are also of great importance. It will be seen, therefore, that the manufacture of a smokeless powder coming up to the high requirements of modern times is not at all an easy matter, although the composition of the powders themselves is not a complicated one.

(*a*) PHYSICAL PROPERTIES.

Smokeless powders are at present made either in the shape of grains, parallelopipedic flakes, cubes, or that of cords. The grains are as a rule very small—between $\frac{1}{25}$ and $\frac{1}{50}$ of an inch in diameter. The flakes are about $\frac{1}{16}$ of an inch square and about $\frac{1}{80}$ of an inch thick. For sporting powder the thickness is reduced to $\frac{1}{250}$ of an inch. Powder for large guns is made either in square flakes of $\frac{1}{8}$ inch sides and $\frac{1}{36}$ of an inch thick and upwards, or in cubes of 2, 5, 10, 15 and more millimetres ($\frac{4}{50}$, $\frac{1}{5}$, $\frac{2}{5}$, $\frac{3}{5}$ inch and more) size. Of the appearance and the shape of cordite everything necessary was mentioned in describing its manufacture.

The sizes used are :—

	INCH DIAMETER.
For the ·303 rifle	0·0375
For the 12 Pr. B. L. gun	0·05
For the 4·7-inch Q. F. gun	0·20
For the 6-inch Q. F. gun	0·30
For large navy guns	0·50

The colour of pure gun-cotton powders is, as a rule, a dirty grey or yellow, which is due to the solvent, and that of nitro-glycerin powders a light to dark brown. Sometimes, through the materials added or from being coated with black-lead, the powders are of a silvery grey, black, or reddish colour.

Smokeless powders are usually insoluble in water, but some which contain saltpetre lose the latter when wetted. The surface is mostly smooth and hard, and as they can only absorb moisture on the surface, they are practically unaffected by it. At Krupp's factory nine different kinds of ballistite in cubes of from $\frac{1}{25}$ to $\frac{3}{5}$ of an inch side were exposed in a room in quantities of 2 kilogrammes (4·4 lbs.) in china saucers, simply covered with paper and the saucers weighed every month. After a year seven kinds had less moisture, and two only more, than when the experiments were started. In one kind, which was coated with black-lead, the moisture had increased by 0·02 per cent.; in another one (not black-leaded) by 0·08 per cent. The maximum moisture was found in February and March, when it was between 0·07 and 0·23 per cent., and the average moisture of all nine kinds was in February 0·19 per cent. and in March 0·13 per cent.

The density of smokeless powders varies with their mode of manufacture. Most of them have a gravimetric density of between 0·25 and 0·40 if in flakes or grains.

(*b*) Chemical Properties.

The chemical stability of smokeless powders is very great, on account of the high requirements as to regularity of action. In general it can be assumed that they will stand the heat test much longer than 15 minutes. Krupp's factory stored 50 charges of 1 kilogramme and 0·75 kilogramme of ballistite of 3 millimetres ($\frac{1}{8}$ inch) cubes at 50° C.; the former were made up in brass cartridge cases, and the latter in ordinary bags. They were divided into batches of ten cartridges each, which were fired successively at once, after 42 hours, after four days, and after fifteen days, and at each experiment the gas pressure and velocity were determined. No considerable increase in either could be observed, and therefore the remaining ten charges were exposed for five days to a temperature of 60° C., and then fired. With this storage also, no exterior change could be noted, and the total increase of velocity was 20·1 and 23·5 metres, and that of the gas pressure 255 and 275 atmospheres respectively.

The analysis of smokeless powders can be readily made if due regard be paid to the indications given for the analysis of blasting gelatine and gun-cotton. On account of the greater hardness of smokeless powders, it is necessary to first grind them in a mill, for which purpose an ordinary coffee-mill with a steel grinding cone is sufficient. If it be feared that small particles of iron were broken off from the mill, they can be picked out by means of a strong magnet. The moisture and any adhering solvent can be determined by drying at 40° C., and exposing in a desiccator.

If one has to deal with a nitro-glycerin powder, the nitro-glycerin must first be extracted with ether. In the remaining nitro-cellulose the soluble nitro-cellulose is determined by extracting with ether alcohol, and the remaining insoluble part examined in the usual way for tri-nitro-cellulose and non-nitrated cellulose.

If one has to deal with a pure gun-cotton powder, it may be examined like gun-cotton.

For the determination of the percentage of nitrogen it is advantageous to use the syphon funnel designed by Professor Lunge for his nitrometer, since smokeless powder only dissolves slowly in sulphuric acid. It is best to put the weighed powder into the funnel of the nitrometer, close the latter with the syphon funnel, and then introduce the concentrated and, later on, the dilute sulphuric acid.

(*c*) MECHANICAL PROPERTIES.

Through the treatment with the solvent the ingredients of smokeless powders almost completely lose their explosiveness. According to experiments made at Woolwich with cordite, an explosion cannot be brought about even on igniting very considerable quantities.

The powder is also insensitive to shock, or to the passage of rifle bullets through it. In general smokeless powders are more difficult to ignite than black powder. In order to obtain the full effect from them in a gun, it is necessary to use stronger caps and special priming compositions, the explosion of which in the cap should develop as much heat as possible, and the charge of the cap must be increased in some cases to 0·6 grain.

The combustion is usually a complete one, the powder being converted into permanent gases (chiefly carbonic acid) and steam. At the same time compounds of nitrogen and oxygen are formed, giving a small amount of slight yellow-coloured smoke, with a pungent odour. Most smokeless powders do not leave any residue worth speaking of, and the barrels are perfectly clean after a shot, but after using they must be carefully cleaned, because traces of nitric compounds always remain which, combined with the action of the moisture in the air, will attack the barrel and the rifling.

Exhaustive experiments on the mode of combustion of smokeless powders have been made by only comparatively few investigators, and have not yet been published.

CHAPTER XX

CAPS AND DETONATORS

Soon after the discovery of fulminate, its property of being easily exploded by a shock or blow led to experiments in using it in percussion fuses. Percussion caps were first made by Joseph Egg, an English gun-maker, in 1815. Within a short time the manufacture of caps attained considerable importance, but many accidental explosions made it appear desirable to find suitable substitutes for the fulminate of mercury, although this has only been done to a very limited extent. At present almost all caps are made of a mixture of fulminate of mercury and other materials, which will easily explode under a blow.

Caps are small cylinders, closed at one end, the diameter of which is either equal to, or a multiple of, their length. The so-called detonators for blasting operations are an exception, as they have a diameter of between $\frac{1}{4}$ and $\frac{1}{3}$ inch, and a height of between $\frac{3}{5}$ and $1\frac{3}{4}$ inches in order to enable them to hold a comparatively large charge.

1. MANUFACTURE OF CAP CASES.

Caps are at present universally made of sheet-copper, which was formerly sometimes silvered. Nickel silver and sheet-iron have been tried, but with unfavourable results.

The copper used for caps must be as pure as possible and perfectly soft and pliable, so that it can be wrought into the desired shape by bending and pressing without a cracking or folding of the surface.

As a rule, the copper as bought is re-melted in the factories, carefully freed from slag and cast into bars or ingots. It is then formed into sheets of between $\frac{1}{100}$ and $\frac{1}{60}$ inch thickness by rolling-mills, with frequent annealing in a retort furnace, freed from the oxide formed, by washing first with dilute sulphuric acid, and then with water, and dried by rubbing with sawdust. After rolling, the sheet-copper must not only be of the proper dimensions and the required weight, but must also stand repeated sharp bending and working without breakage, whilst the surface must be smooth and free from cuts, folds, or cracks.

Annealing and rolling the copper is an operation requiring much practice. By over-heating copper can easily be burnt, which makes it hard and brittle, whilst if not heated sufficiently it will crack on being rolled.

The caps are made from the sheet-copper by cutting the sheet on rolls with circular knives into strips of previously determined width, and then forming these strips, either at one or several operations, into cylinders by a punch and die. The edge left from punching is cut off in a machine, or in some arrangements removed in the punching machine itself. The finished cap cases are cleaned with water, acidulated by sulphuric acid, or with soap-suds, and then revolved with sawdust in a drum which dries and polishes them.

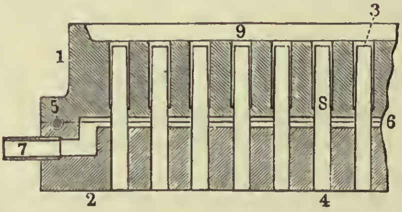


Fig. 241.

It is not necessary for the purposes of this book to describe the machines used for making caps. In principle, they do not materially differ from the apparatus in general use for similar work, such as making cartridge cases. Among the firms who make a speciality of these machines, Messrs. Greenwood and Batley, Limited, of Leeds, and Veuve Lachaussée of Liège, may be mentioned.

J. Paulus of Berlin suggested the use of an alloy of 70 parts of lead, 20 parts of tin, and 10 parts of antimony for making caps by casting them in the mould illustrated in Fig. 241. This consists of 2 plates (1 and 2). Plate 1 has a number of cylindrical holes (3), whilst in the lower plate there are a number of cores (4) corresponding to the holes in the upper one. The two plates are held together by a pin (5), and between them there is a channel (6) connecting the various holes, from which the air can be exhausted through the pipes (7). The space between the piston and the holes is of such size that air can enter it, but not melted metal. The melted metal is cast into all moulds at the same time through the groove (9) on the top; then the air being pumped out through the pipe, the melted metal runs into the space between the cores and the moulds without leaving flaws, being simply forced in by the atmospheric pressure. When cool the lower plate is taken off, and another one without the cores put in its place. The finished caps are then forced out by hydraulic pressure or similar means.

This idea for making caps is no doubt a very good one, but it is a question whether difficulties will not be met with in the process, especially with regard to making the ends of the caps of the proper thickness.

2. MIXTURES FOR CAP COMPOSITION.

About the year 1816, fulminate of mercury mixed with wax or an alcoholic solution of gum benzoë was first used as means of ignition. When the dangers attending the preparation of cap composition were better understood, makers ventured to make additions which, although they sometimes increased the danger, considerably strengthened the effect of the caps for certain purposes. For detonators that will give the strongest possible effect in the shortest possible time, and which will ignite by a strong flame, only pure white fulminate of mercury is as a rule used. For percussion caps the fulminate is mixed with other substances in order to ensure ignition of the powder charged by the blow struck on to the cap, and also to effect the ignition more slowly, and thus give a more prolonged action. For instance, for black powder, a slowly burning cap composition should be chosen, whilst for smokeless powder as strong as possible a shock effected in as short a time as possible is required in order to properly detonate the powder. Saltpetre, antimony sulphide, potassium chlorate, sulphur, meal powder, etc., are the ingredients usually added to the fulminate of mercury in making cap composition. Of these sulphur and saltpetre are now but rarely added.

According to Gottlieb, 4 parts of potassium chlorate and 8 parts of antimony sulphide made into a paste with a solution of gum arabic were used for the Prussian pin-fire rifle cartridges, and also a mixture of 5 parts of potassium chlorate and 8 parts of antimony sulphide with a little flowers of sulphur.

At present the following cap compositions are used:—

In Great Britain for the Martini-Henry ammunition: $2\frac{1}{4}$ parts of fulminate of mercury, $2\frac{1}{4}$ parts of potassium chlorate, and $1\frac{1}{2}$ parts of antimony sulphide; for gun primers, 3 parts of fulminate of mercury, $1\frac{1}{2}$ parts of potassium chlorate, and $1\frac{1}{2}$ parts of antimony sulphide.

Similar proportions are used for cap composition in nearly all countries where a dry process of mixing is adopted.

In Switzerland for metal-cased cartridges and revolvers:—

	GRAMMES.
Fulminate of mercury	600
Potassium chlorate	75
Ground glass	300

are taken and mixed with 45 grammes of a solution of gum arabic, consisting of 1 part of gum arabic and 2 parts of water. For gun primers, the composition consists of 60 grammes of fulminate of mercury, $7\frac{1}{2}$ grammes of potasium chlorate, 30 grammes of ground glass, and $7\frac{1}{2}$ grammes of gum arabic solution (1 part gum arabic to 1 part of water); for shell primers, 4 parts of potassium chlorate, 4 parts of antimony sulphide, and 1 part of ground glass are mixed with alcohol.

The priming composition in Great Britain for Nordenfeldt guns consists of 3 parts of fulminate of mercury, 3 parts of potassium chlorate, 2 parts of antimony sulphide, and 1 part of ground glass, which are mixed with a solution made of $\frac{1}{4}$ part of gum arabic and $\frac{1}{60}$ part of dragon's blood, each of which is dissolved separately in 5 parts of water.

3. MIXING THE CAP COMPOSITION.

Since fulminate of mercury is so very dangerous and sensitive, the operation of mixing is a very delicate one, and requires great caution. It is therefore either done in a dry state with special precautions, or a wet process is used.

In the wet process, the cap composition is as a rule mixed with a solution of gum arabic, or more rarely with one of shellac or gum benzoë in alcohol, and incorporated in a porcelain or wooden mortar with a wooden pestle for about 14 hours. The pestle is usually suspended from the ceiling. The composition is then put, still in a wet state, into the caps, or it is first freed from moisture in drying-houses. The drying-houses are heated to 104° F. at most, and the trays are made of wood covered with a string netting. The composition is put on to cloth or sometimes on to sheets of paper.

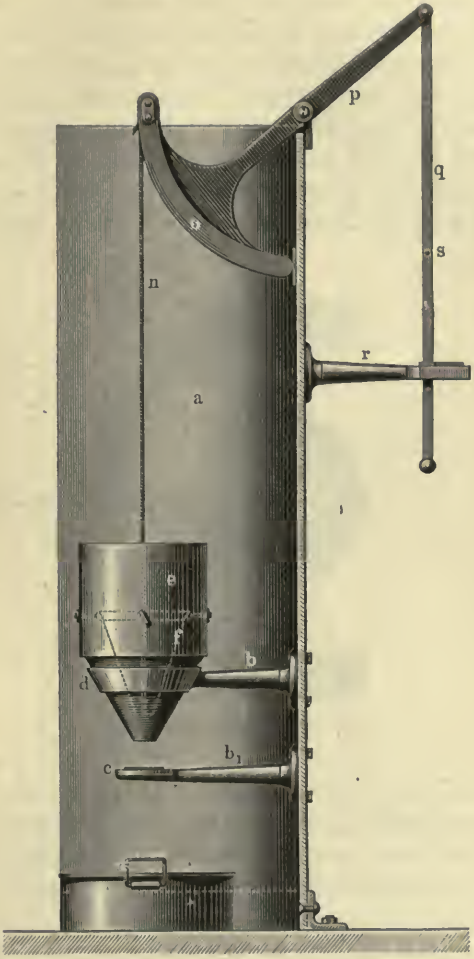


FIG. 242.—One-20th full size.

In some places dry mixing of the composition is effected by putting the ingredients a little at a time on to a sheet of paper and mixing them by means of a long goose or swan feather. The workman stands behind a protecting wall, and only his arms and eyes are left free. Another way of mixing consists in raising the two diagonally opposite ends of the paper, and thus make the composition run together. In both cases the composition is afterwards passed through a hair, or fine metal, sieve, by slowly inclining it, which effects a fairly good mixture.

There have been many explosions with both ways of mixing in spite of attempts being made to diminish the danger by covering the tables with felt, and placing the workman behind a screen, and also by reducing the quantity allowed in a room at a time to a minimum, the limit being as a rule 7 oz. Cap compositions, however, especially those containing ground glass or antimony sulphide, are so sensitive, and can so easily give rise to accidents, that even when using every precaution and when only the best trained and most experienced workmen are employed, accidents from this way of mixing will frequently occur.

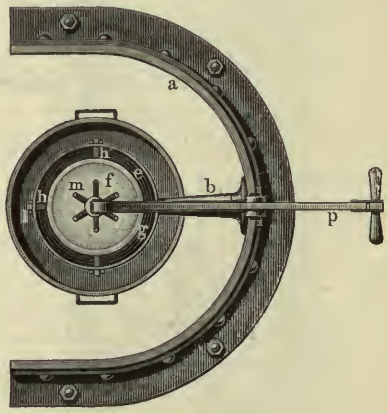


Fig. 243.—One-20th full size.

To avoid this, a machine for mixing cap compositions dry was introduced in the French factories, which has been subsequently adopted in England. It is illustrated in Fig. 242 in section and in plan in Fig. 243. It has proved to be perfectly safe during the time it has been in use, and although explosion of the composition can occur, only two explosions have happened with this apparatus in England, and no damage was done to the workman in either. The apparatus consists of a semi-circular wrought-iron screen (a) about the height of a man. Two bronze arms (b b_1) covered with leather are fixed on it. The arm b_1 supports a plate (c), which carries the vessel receiving the mixed composition. The other one carries a leather-lined conical ring (d) into which a box (e) made of sole leather is put. The upper part is cylindrical and the bottom conical, with its lower end open. In this box a bag (the so-called "jelly" bag) (f), which is illustrated on a larger scale in Fig. 244, is suspended. It is 10 inches wide at the top and 10½ inches high, and is made of white silk stitched over a gutta-percha ring (g). Four leather loops (h) are sewn on to the ring, and these pass through corresponding holes in the box and are fixed outside the leather pegs (l). Inside the bag there are

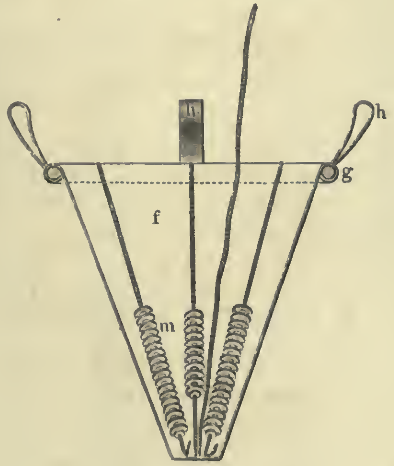


Fig. 244.—One-7th full size.

six cords (m) sewn, each of which has a number of india-rubber rings, alternately large and small, threaded on to it. The diameter of the large rings is $\frac{5}{8}$ inch, and that of the smaller ones $\frac{5}{16}$ inch. Four of the cords have 18 large and 18 small rings, and two of them only 11 large and 11 small ones. Sometimes, instead of the cords and washers, small india-rubber balls are put into the bags, but these give too much friction, and shocks are possible. A string is fixed to the point of the bag and passes upwards over a wooden segment (o). This is attached to a lever (p) which turns in a hinge on the screen, and can be moved up and down by means of a rod (q) fixed to the other end of the lever. The rod is guided by an arm (r) on the screen, and its down-stroke limited by a pin (s) put through the rod, the up-stroke being limited by the segment touching the screen. Flat rubber washers are fixed over both stops to take up the shock. The workman stands behind the screen and draws the bar up and down, whereby the bag is partly drawn upwards, and the composition falls between the rings, which as they rise and fall effect a thorough mixture. To empty the bag, it is drawn completely up, when the composition falls over the gutta-percha ring into the space between the bag and box, and runs into the collecting vessel through the conical bottom of the box.

As a rule, the antimony sulphide is first put into the bag, then a cardboard funnel put on, over which a sieve is laid. The potassium chlorate is rubbed through this by means of a brush, and the bag slightly shaken to make it enter completely. The fulminate of mercury is put in last. The mixing is then started and continued for three minutes by moving the bag up and down.

The finished composition is stored either in boxes of varnished cardboard or more usually in hard rubber ones. Composition made by the wet process is rubbed through hair sieves to granulate it, in which form it is more easily put into the caps. The sieve must be carefully cleaned after each operation.

4. CHARGING THE CAPS.

The older method of charging caps was a very dangerous operation for the workman engaged in it, because it was usually done by hand. Three instruments were used for this—first, the so-called "hand," which was a perforated copper plate holding the caps; the second, a funnel consisting of two copper plates sliding one over the other, and provided with holes for filling; and the third, the "pin-holder," provided with pins for pressing the composition into the caps.

Now-a-days the charging is always done by a machine, and it has been brought to such perfection that the workman is exposed to no danger, and the explosion of a cap rarely spreads to the others.

Josten constructed a charging machine in which he used the principle underlying the old method of hand-charging. He retained the "hand" and "funnel," and adapted his machine as much as possible to them. The complete machine and details of it are shown in Figs. 245 to 251. Fig. 245 is a side elevation, Fig. 246 a longitudinal section of the charging machine itself, Fig. 247

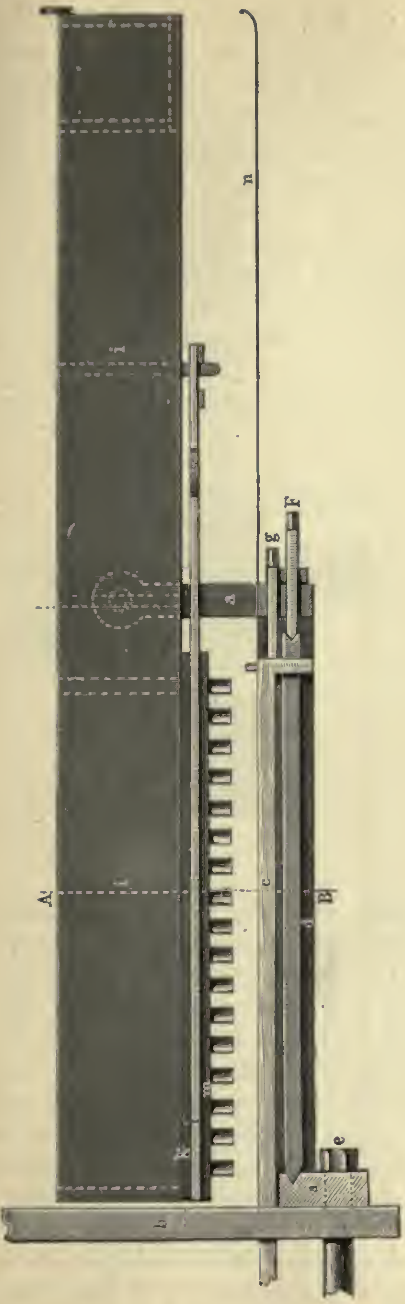


FIG. 245.

FIG. 246.

a front elevation of the screen (b), Fig. 248 a section on the line (A-B, Fig. 245), Fig. 249 is a plan, Fig. 250*a* a section of the charging slide (l) in front of its perforation, Fig. 250*b* the same behind the perforation, Fig. 251*a* a longitudinal section of the bottom of the charging machine in front of the perforation, and Fig. 251*b* the same behind the perforation. Figs. 250 and 251 are drawn full size, the others quarter size.

The machine is placed in the charging-room, and the workman stands behind the wrought-iron screen (b), which is $\frac{5}{8}$ inch thick, 5 feet 3 inches high, and 3 feet wide, and gives efficient protection. It is slightly bent (Fig. 249) and firmly connected with the frame of the machine (a) by means of the screws (e). The brass slide (c) moves through a suitable opening of the screen, and the charging mould (the hand), after being filled with empty caps, is placed on it. The slide is guided by the small cast-steel bar (d), and its path limited by the screw (g). In order to fix the charging mould, it has holes corresponding to the pins fixed on to the slide. The screening sheet (n, Figs. 247 and 248) is to prevent grains of the composition from falling out when the charging mould is changed.

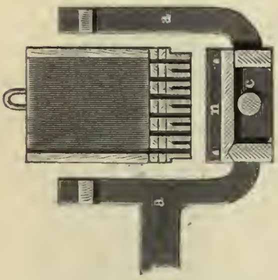


FIG. 247.

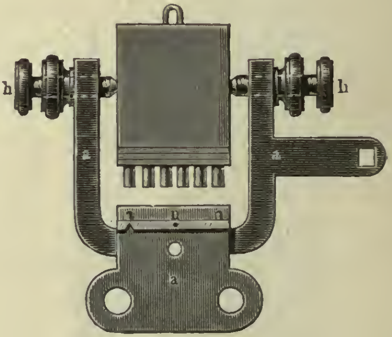


FIG. 248.

On the other end of the frame (a) are screws (h), Figs. 248 and 249, and on their points the charging machine itself can move up and down like a beam. The machine requires a very careful construction, and the lighter it is made without impairing its solidity, the less destructive are the effects of an explosion. The frame (i) is made of fine-grained wood and is divided into several compartments. The part towards the screen (b) is the filling trough, the bottom of which is a thin brass plate (k, Fig. 245 and

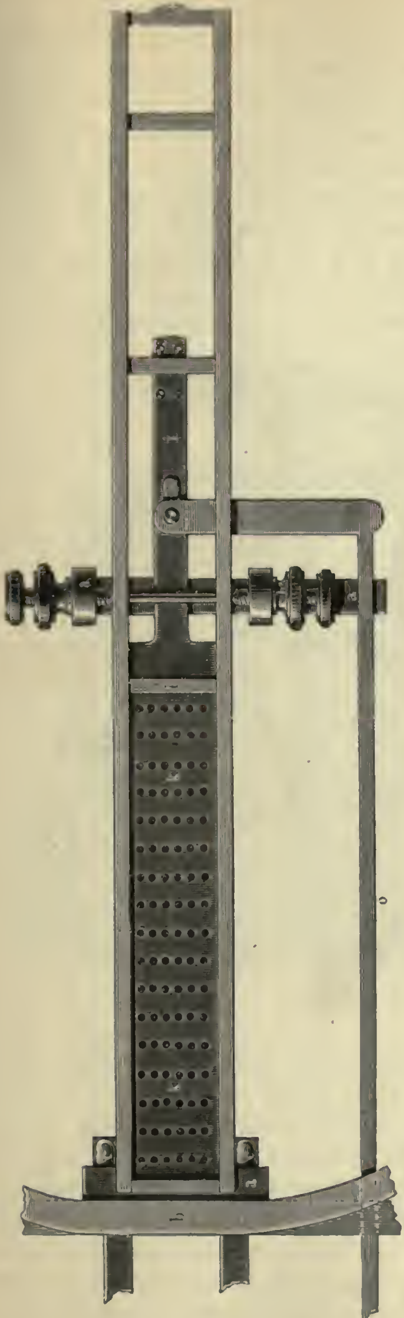


FIG. 249.

FIG. 250.

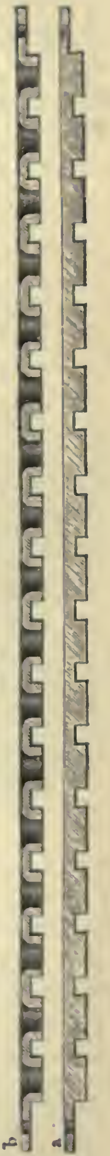


FIG. 251.

Fig. 249). Fig. 246 shows the bottom of the old machine, and Fig. 249*b* as improved by Josten. Below the bottom the charging slide (l) (Figs. 249 and 250*b*) is moved by means of the bar (o); its rubbing surfaces being reduced to a minimum. The bottom also is so constructed that the friction shall be very small. Below the charging slide is the tube-plate (m). The projections (p and q) fix the two chief positions of l. If q is on r, there is no communication with m, but if p is on r, the composition falls through the perforations into the caps.

A weight or india-rubber spring fixed on the other end of the frame (i) tends to lift off the charging mould. The workman can counteract this pressure by drawing a cord passing over a pulley, and thus he can press at will. On charging, the workman introduces the slide (d) and the charging mould with one hand, presses down with his foot the charging plate, and by moving the rod (o) with the other hand fills the caps.

Fig. 252.

The more modern machines do the charging in a comparatively simple manner. Such a machine made by Greenwood and Batley, Limited, of Leeds, is illustrated in Fig. 252. Here also the caps are put into a hand, which is put into the lower part of the charging machine. This consists of three brass plates (a) which are ground together and thus work perfectly smoothly and with very little friction. All the plates are perforated with a number of holes corresponding to the diameter of the caps. The perforations of the lower one are exactly opposite to the caps, while the upper one has its holes opposite to the plain part of the lower plate. The middle part is fixed on to a rod (b) guided at the sides and capable of being rotated by means of a screw in a column (c), some distance off. On filling, the holes of the upper plate are opposite the plain part of the middle plate. The composition is put into the holes of the upper plate, the middle one is pushed forward by turning the screw-bar, whereby the perforations of the middle and lower plates communicate with each other, and the caps are filled with a quantity of composition corresponding to the thickness of the middle plate. The operation is effected by gradually turning the handle (d), and since no more composition is put on to the apparatus than is required to fill the caps, an explosion could not assume large dimensions. No accidents, however, with such an apparatus have occurred so far.

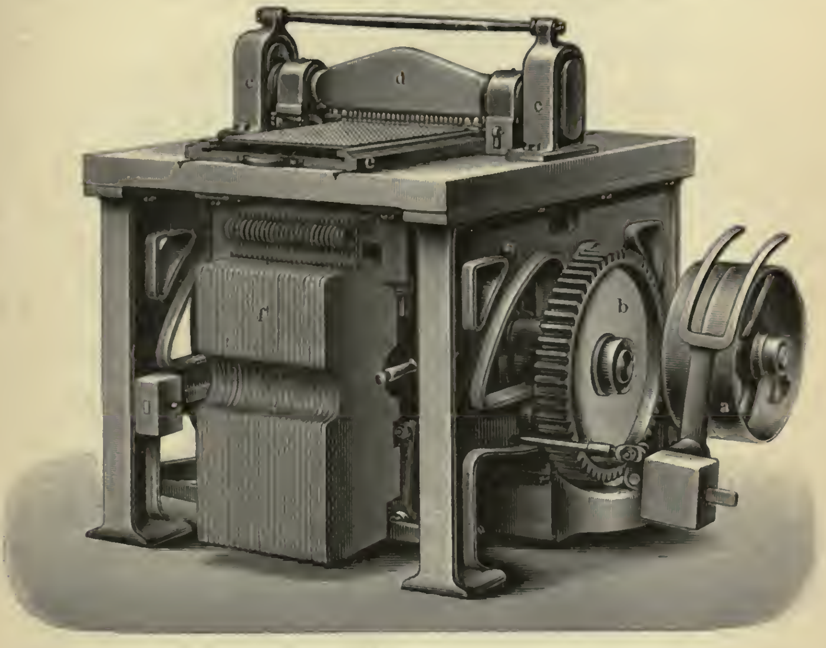


FIG. 253.

In order to press the composition into the caps, the machine illustrated in Fig. 253 is used. It consists of a cross-head (d) sliding between two guides (c), which is moved up and down by a belt and spur gearing (a—b), and at the same time the table (e) carrying the hand is pushed forward, so as to bring the next series of holes under the cross-head. The pistons are fixed to the cross-head. The row of caps being compressed rests on a number of movable bases kept up to their work by counter-weights (f), so that the pressure exerted on the caps can never exceed a certain limit depending on the weight of the plates (f). For rifle caps of $\frac{1}{8}$ inch diameter, the pressure is about 2 cwts. The construction of the machine will be easily understood from the illustrations.

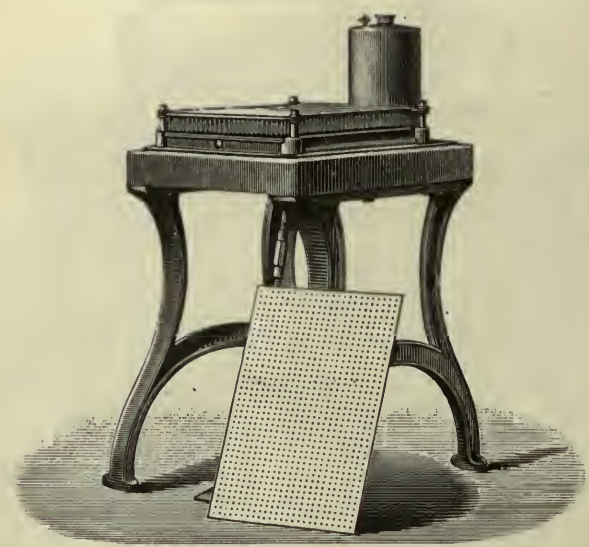


FIG. 254.

The surfaces of the compressed composition in the caps are covered either with shellac varnish or with a small piece of copper or German silver foil. The varnishing is done by putting a solution of shellac in alcohol on to a plate. A number of pistons go down to the plate, and thereby take up a thin layer of varnish; then the varnished plate is taken out, and a "hand" filled with caps is put in. The pistons are again brought down, and leave a thin coating of varnish on the surface of the composition. Fig. 254 illustrates such a machine, the construction of which is sufficiently clear to need no further description. The insertion of pieces of foil into the caps is done in a similar manner to the pressing of the composition by means of a machine illustrated in Fig. 255, where the hand is brought step by step under a number of pistons fixed in a cross-head. In the machines formerly used—those of Josten, for instance—the caps were carried on a disc, which brought them successively under a piston.

For larger caps, the detonators used for blasting operations, for instance, machines are used in which each one is treated singly, because the charge of such a detonator is sufficient by itself to cause considerable damage. In very large factories, however, as many as 100 detonators are sometimes compressed at a time, but then both the charging machine and the press are placed in perfectly isolated rooms, and are separated from the workmen by protecting walls with such arrangements that the opening through which the workman watches the process must be closed before pressing is started.

Fig. 255.

Presses working caps with such strong charges singly are as a rule enclosed in a strong wrought-iron casing, and the compressing is generally done by a quick-pitched screw. The detonator is put into a steel mould, and rests on a bottom-piece held up by a weighted lever. Such a mould, which is used at Woolwich, is illustrated in Figs. 256 and 257. It consists of an outer thick case (a) into which a slightly conical centre-piece (b) made in two parts is fitted by grinding in. The centre-piece is fixed against the outer case by the screwed stopper (e), the projecting part of which fits into a recess (f) of the weighted bottom part. The stopper and the centre-piece have an axial perforation (c) into which a pin (d), shaped according to the form of the cap bottom, is put.

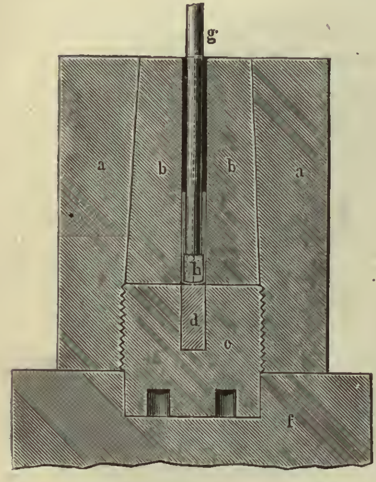


Fig. 256.

Fig. 257.

The detonator (h) when filled with the composition is put into the axial bore of the centre-piece, and is compressed by the upper piston (g). Should an explosion happen, only the centre-piece is as a rule destroyed, and thus can be easily exchanged. In this way the risk of broken pieces being thrown about is diminished, and it is also prevented by the casing of the press. With larger machines where several detonators are pressed at the same time, pressing is done by lifting the bottom part which carries the detonators up against the piston by means of a weighted lever.

The charge of rifle caps is on an average from 0·22 to 0·25 grain, according to the composition of the priming; for smokeless powders it is between 0·60 and 0·75 grain. Detonators are made in the following sizes and charges:—

	LENGTH IN MILLIMETRES.	DIAMETER IN MILLIMETRES.	CHARGE IN GRAMMES PER THOUSAND.
No. 1	16	5½	300
No. 2	22·	5½	400
No. 3	26	5½	540
No. 4	28	5½	650
No. 5	30	6	800
No. 6	35	6	1000
No. 7	40	6	1500
No. 8	45	6·8	2000

They are subjected to a pressure of 4000 lbs. per square inch, which gives the fulminate of mercury in the detonator a density of 2·800.

The finished caps are packed into cardboard or tin boxes. Detonators are put into tin boxes lined with felt, and the interstices filled with sawdust or cork waste. A number of such tin boxes are put into a paper roll, which is glued and pasted over at the ends with a paper label. About 50,000 detonators are packed in a wooden case, and this as a rule into a second outer case.

Experiments made by Lovel a long time ago proved that when only a few out of a large quantity of packed caps explode, the explosion is not communicated to the others. Out of 500 caps enclosed in a case, he detonated one by means of a punch passing through the cover, when three or four of the others at most went off. If, on the other hand, black powder were in the case either loose in grains or in cartridges, as a rule all the caps were exploded.

In the same way experiments made by the Birmingham Chamber of Commerce showed that the carriage of caps is not associated with any danger, even if whole packets catch fire. A case containing 50,000 caps burned away without explosion in a melting furnace. Packets containing 5000 caps were crushed by a weight of 1 cwt. falling from a height of 14 feet without a general explosion taking place. The chief danger is therefore in the making of the fulminate of mercury, and especially in some of the manipulations during the manufacture of the caps.

Similar experiments have been made by H. M. Inspectors of Explosives, which also proved that detonators are sufficiently safe from liability to explode *en masse*.

It frequently happens that the composition is not only introduced in a damp state, but even in the form of a paste, into the caps. This can be done either by making the composition into small pills which are put into the caps, or by smearing it on a perforated metal plate, the holes in which just hold the required amount. The holes in the metal plate are first closed by another plate, the composition is put into the holes, the lower plate taken away, and the mass is pressed into the caps placed below by a piston plate. If damp, or pasty, composition be put into the caps, it is allowed to dry in them. This process is especially favoured in the manufacture of sporting caps, and explosions with this method are almost impossible. The objection has been raised, however, that the ingredients partly effloresce, and that the metal of the caps is attacked.

Attempts have frequently been made to discover other explosives, or mixtures of such, which would give as good an effect in caps as fulminate of mercury. Mixtures of gun-cotton and potassium chlorate, nitro-mannite, lead picrate, as well as certain diazo- and many other compounds have been suggested, but the mixtures of fulminate of mercury have always held their ground.

CHAPTER XXI

FUSES

A LARGE number of different fuses are used for military purposes; for instance, the time fuses, which consist either of tubes or special heads screwed on to the projectiles, and which are filled with a composition with an exactly known rate of burning; also priming compositions for making various igniting arrangements, fuses for firing guns in rainy weather, etc.

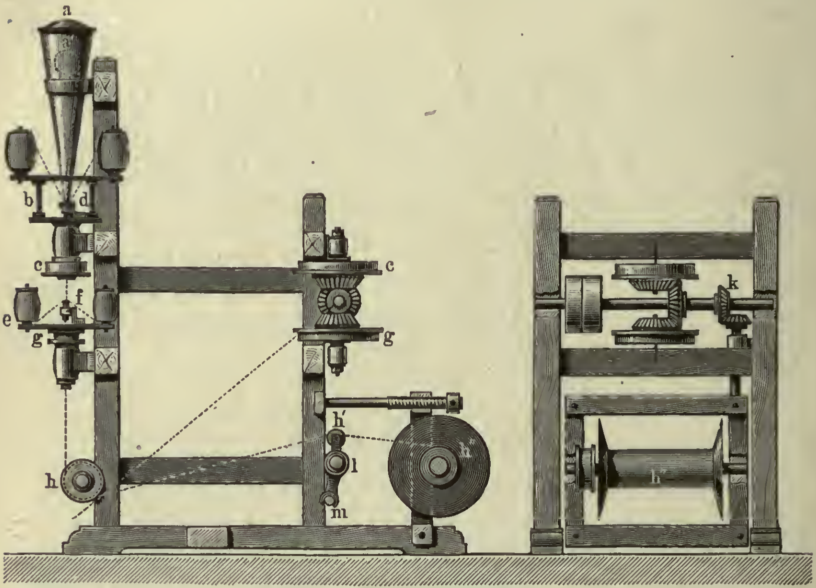
It is impossible to deal closely with them here, as the composition of most of them is kept secret, and also they are of no direct industrial importance. Only the most important fuses used in civil engineering will therefore be mentioned.

(*a*) SAFETY FUSES.

For firing blasting charges in bore-holes, and also for military purposes, the so-called safety fuses or Bickford's fuses are used on a large scale. They consist of a powder thread spun round with jute yarn and impregnated with compositions to make them waterproof. Since the core of powder is tightly, compressed in a thin continuous thread within the fuse, the fire only travels along it slowly, the rate of burning of a good fuse being about 30 seconds per foot.

Meal powder—that is to say, the very finest granular powder free from dust, which is of too small a grain for the ordinary uses of powder—is used for the manufacture of such fuses. There are various machines for making them. In those used by Messrs. Bickford and Co., a number of jute threads pass through an orifice whilst they are slowly rotating, and at the same time a fine current of powder falls into the tube thus formed, and is held as a fine core by the cord. A weight is attached to the cord, so that it is always tightly stretched. The cord formed is of quite loose texture, and is passed through a second machine similar to the first, which spins round it a second layer of jute yarn, but in the opposite direction.

In Karmarsch and Heeren's 'Technical Dictionary' (1892), B. Rabitz publishes the illustration of a machine for the manufacture of safety fuses, which is reproduced in Figs. 258, 259, and 260. In it is a funnel (a) in which there is placed a reel (a′) fitting loosely on a pin, which carries the cotton thread. The cotton thread serves a double purpose, namely, that it keeps the opening of the funnel always free, and at the same time is a trade-mark, each factory using a differently-coloured thread. The point of the funnel is illustrated in Fig. 261 to a scale of one-third. It has a shut-off tap, and an orifice which can be screwed on. The machine has two discs carrying wheels, the disc (b) for the filling reels, and the disc (c) for the "spinning-over" reels. The first has 10 vertical spindles, each of which carries a reel of jute yarn. All 10 threads of the filling reels enter an orifice (d), and a tube is formed, which has externally the appearance of a left-handed screw of 10 threads. At the same time powder runs from the funnel into the



Figs. 258, 259.

tube. This goes through a tightening nozzle (d_3), which compresses the loose tube. From here it passes through a steel nozzle (f), which has radial grooves on the top. A thread from the spinning-over reels passes through each groove, and as the disc is turned in opposite direction to the filling disc, by means of the belt and pulley (g), the six threads are wound round the hose and cover it with a right-handed twist of six threads. The cord passes several times over a small cone (h), then over a guide pulley (h′) on to the reel (h″). The cone is driven by the worm and wheel (i) and cog-wheels (k), and the reel (h″) by a belt and the pulleys (o). By using the belt and cone-gear, the reel, whether the quantity reeled on it be large or small, winds up the amount coming down from the cone. The turns of the cord are laid side by side on the reel by a guiding gear driven by the pulleys (i and i′). On the axle of it there is a right and a left-handed thread cut, the ends of which meet. This causes a nut to travel backwards and forwards, and so guides the cord carrying an arm and the guide pulley (h′). In this way the out-turn of a whole day, about 1800 yards, can be wound on to the reel.

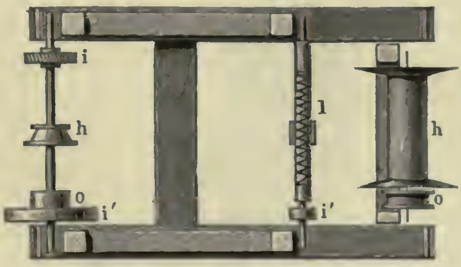


Fig. 260.

The finished fuse is drawn through a bath of tar, to which a little wood tar has been added. This coats externally and partly penetrates the cord. After tarring the fuse is drawn through fuller's earth or china clay, and is then wound on drums of such diameter that their circumference corresponds exactly to the length of a coil of fuse. As soon as the fuse is completely wound on to the drum, it is cut through parallel to the axis of the drum with a sharp knife, and the single length thus obtained wound into coils, which are then tied with strings and form the well-known coils in which safety fuse is sold. Twenty of such coils are made into a packet. The length of a coil is 8 metres in Germany, and 25 feet in England.

The tarring and dusting previously described is that used with the so-called "single black fuses," but other kinds of fuses are made for the various uses to which they are put. For ordinary blasting purposes, where no moisture is met with, the so-called "white fuses" are sufficient. These are simply coated with china clay made into a paste with a little glue size. Red fuses have also been made in the same manner by coating them with bolus powder. For coating both kinds, the paste is contained in steam-heated pans, and the fuse is passed over rolls either through steel dies or through weighted jaws on to the reel, which for the purpose of better drying can be heated by steam. For wet bore-holes the so-called "double" and "tape fuses" are used. The double fuse is first coated with tar, then again with a layer of yarn, and again tarred. The tape fuse is served with a covering with tape instead of with yarn, the tape being wound so as to overlap.

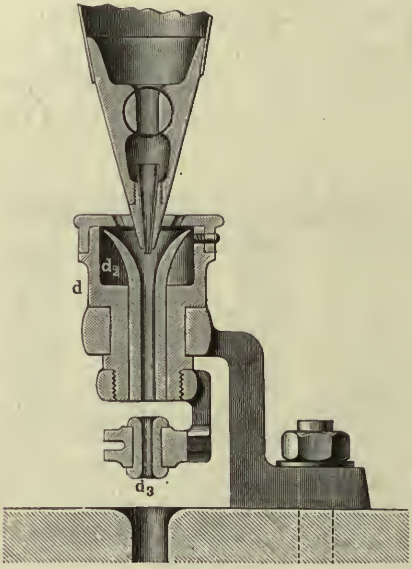


Fig. 261.—One-3rd full size.

For blasting under water gutta-percha-covered fuse is used. The fuse is first coated with sizing to give it a perfectly smooth surface. The size is made of a mixture of inferior gutta-percha with Stockholm pitch and linseed oil, or from Syrian asphalt, linseed oil, and gutta-percha. Its object is to perfectly fill up the pores of the uncoated fuse. Over this a thin coating of pure gutta-percha is put, which easily melts and quickly hardens, and on bending the fuse should not break, even after prolonged storage at low temperature.

For this purpose an apparatus, a description of which was also published by Rabitz, and which is illustrated in Fig. 262, is used, which is placed in a cold room or in a cellar. It consists of a pan (a), about 3 feet 4 inches long, heated by steam and divided into three compartments. The pan is fixed between two vertical columns, and in it the size and the gutta-percha are melted. Over it a rectangular steam-heated tube (b) is placed, which has in four recesses steel nozzles of different bore. In front of each of these recesses a guide pulley (c) is fixed in a suitable manner on the tube. The fuse is carried on a reel (d) and passes through the nozzle (e) on to the guide pulley (c), and from thence through tube b. A fan (f) blows cold air into a tube (g), and the cold air goes through orifices in it over the gutta-percha-coated fuse to prevent its sticking together. Small water boxes (h) are placed about 18 feet apart, and the fuse runs through them on wooden rollers, and then over guide pulleys (i) to the winding-drum (k). The latter is partly placed in a pit and rotates with about 20 feet per minute circumferential velocity. A guiding arrangement (l) worked from the main axle by means of a guide and two pairs of pulleys, prevents the fuses winding over each other on the drum. The work is, as a rule, done with two reels (d), one of which carries the uncoated fuse, and the other the sized one. Each of these fuses passes through a different orifice, and is drawn over the guide pulley, either through the sizing or the gutta-percha, which are put into recesses in the heated tube, from the pan. After having passed through the whole machine, the fuse goes back through the second and fourth orifice respectively, and finally on to the winding-drum (k), on one half of which the sized fuse is wound and the finished fuse on the other half. The finished fuse is then cut off, whilst the sized one is wound on to a reel to undergo the finishing treatment.

(*b*) Quick-burning and Detonating Fuses.

Contrary to the safety fuses, in which, as has been described, a fine thread of powder is made to burn slowly at an exactly

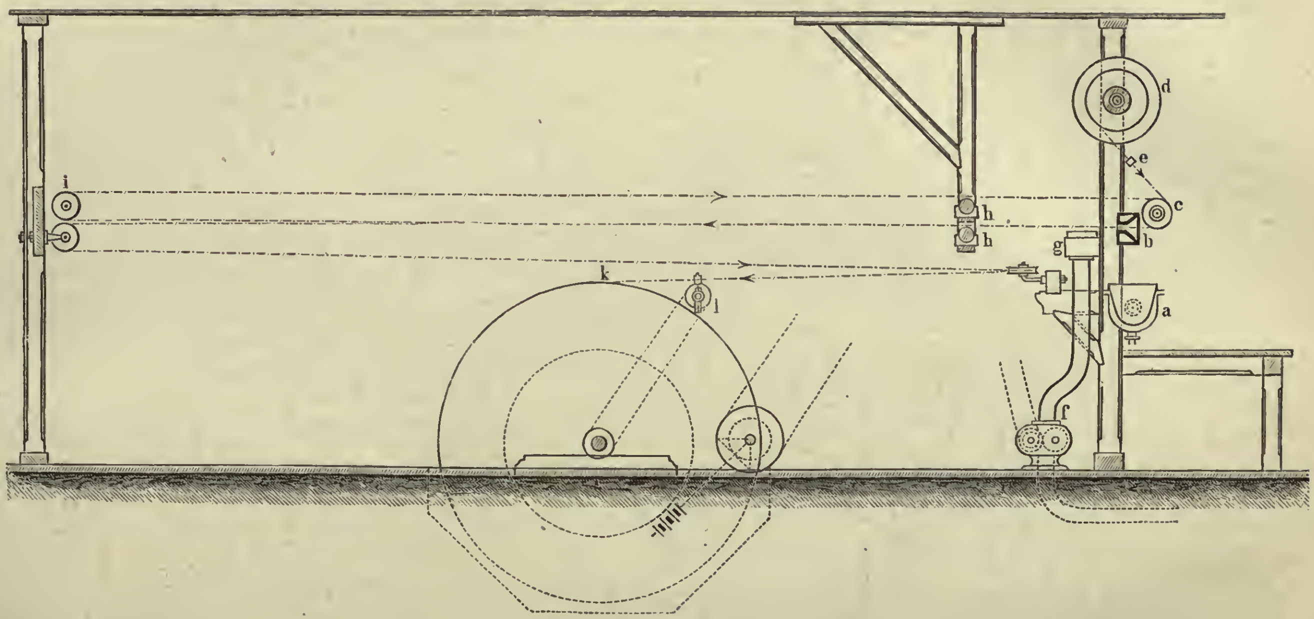


FIG. 262.

regulated rate of combustion by compressing it into a casing, it is sometimes desirable, especially to replace electric firing, to have fuses which burn instantaneously, or practically so, for the whole of their length, or even detonating fuses.

This object has been attained in the simplest way by the instantaneous fuse of Bickford, Smith and Co., of Tucking Mill, in which, instead of the powder core, there is a wick coated with meal powder paste and loosely spun over with yarn. In order to fire several shots at the same time, all fuses are put into a sheet-iron connector, in one end of which there is a disc of powder and a perforated wooden plug to insert a safety-fuse. The whole is then made tight by india-rubber cement. This instantaneous fuse burns at the rate of about 500 feet per second.

A quick-burning fuse invented by Gomez was formerly used in the Austrian army. It was made by drawing four cotton threads through a paste made by mixing equal parts of lead ferro-cyanide and potassium chlorate with alcohol. The fuse was protected by a cotton tape, with a thread serving spun over it. To make the fuse damp-proof, a lead pipe was drawn over it, or it was covered with india-rubber tape, then knitted over with yarn, and drawn through a mixture of bees'-wax and gutta-percha. The use of this fuse was not without danger, as it detonated by a blow of iron, or metal, on stone, and on cutting it, it had to be laid on to wood and cut carefully with a sharp knife. It burned at a rate of from 200 to 240 feet per second.

In the year 1879 detonating cords and tubes (cordeaux détonants, tubes détonants) were introduced in the French army. They are made by filling lead tubes with gun-cotton made from hydro-cellulose, and then drawing down the tubes. Lead tubes of 15·5 millimetres exterior and 12 millimetres interior diameter are cut into lengths of 5 metres. The end of the pipe is beaten flat with a hammer, and the pipe itself is suspended vertically, and filled by a man standing on a platform, who pours the previously dried nitro-hydro-cellulose powder, which runs easily, into the tube by means of a funnel; at the same time a second man standing on a ladder taps the pipe gently with an india-rubber tube of average hardness, in order to shake down the gun-cotton more tightly in the pipe: 200 grammes of gun-cotton are put into each of such pipes, giving a gravimetric density of 0·366. When filled, the other end of the tube is also beaten flat with a hammer, and these flattened ends form grips for the subsequent drawing down of the tubes. This is done by a draw-plate machine, consisting essentially of a disc perforated with a number of regularly decreasing holes and placed on a slide. On either side of it are two drums, on to which the tube is alternately wound, being drawn off one on to the other through the holes in the perforated disc successively, commencing with the largest. The perforations of the disc decrease by 0·5 millimetre from 15·5 to 5 millimetres, and then by 0·2 millimetre down to the smallest, which is 4 millimetres diameter. The cord is thus drawn down to 4 millimetres thickness, and in order to do this passes through the perforated disc 26 times. The reduction in diameter increases its length to between nine and ten times what it was to start with, and the density of the gun-cotton in the fuse is thereby brought to between 1·10 and 1·25. The total time occupied in drawing is three hours; that of charging 15 to 30 minutes. The warmer the gun-cotton powder is, the more easily can the tubes be charged with it. The finished tube weighs about 88 grammes per metre, and each metre contains about 5 grammes of gun-cotton.

More recently these fuses have been made by means of improved drawing machines, in tubes of tin. The finished cord is wound on reels like telegraph wire, and the ends are made tight either with varnish or liquid rubber. The lead tubes are covered with hemp like insulated wire. If this fuse be ignited, it only burns for a short length, and then goes out by itself, but if it be detonated by a cap, the explosion travels along it at an average rate of 4060 metres per second.

Starting from the principle underlying the French cordeaux détonants, Colonel Philipp Hess of the Austrian Military Committee invented a detonating fuse made of fulminate of mercury. In this a cotton thread is drawn through a paste of fulminate of mercury, and then spun over like Bickford's instantaneous fuse. The fulminate of mercury fuse has since been introduced into the Austrian army. It also is exploded by a detonator, and its rate of explosion is 5000 metres per second.

CHAPTER XXII

APPARATUS FOR THE EXAMINATION OF EXPLOSIVES

I. APPARATUS FOR MEASURING VELOCITY, RANGE, AND RECOIL.

A. GAUGES (EPROUVETTES).

1. ORDINARY FIRE-ARMS.

(a) *MORTARS.*

FIG. 263 shows a cast-iron or bronze mortar, which is cast in one piece with its base. The axis of the mortar is at an angle of 45° with the horizontal surface of the base, and the latter is fastened to a stout oak base by four bolts. At both ends the base is tied with iron straps, and there are four handles for moving the apparatus about. The ball thrown from this mortar (Fig. 264) is made of bronze, and its diameter is either 0·190 or 0·1895 metre. There is a socket in the ball with a female thread, into which a handle (Fig. 265) can be screwed for conveniently lifting the ball and putting it into the mortar. When the ball is laid on to the charge, the handle is screwed off and replaced by a bolt (Fig. 266). The mean weight of the ball is 29·3 kilogrammes, and the charge of powder used 92 grammes.

When testing with this mortar, 560 grammes of powder are taken from the same spot in a barrel, and the sample is put either into a glass bottle or into a tin. The bottle or tin is sealed and brought to the testing-ground in a case. The bore of the mortar and the ball are then exactly measured. The weight of the mortar, including the oak plank and the fittings, should be between 210 and 215 kilogrammes. The mortar is then set on an exactly horizontal base. This should be of closely-joined planks 0·16 metre high and 0·1 metre thick, strongly joined together by two stout cross-pieces and laid on a solid block of masonry. The planks are parallel to the direction of the line of shot. The ground where the ball will fall should be neither hard nor stony. In case of need, it must be dug out for one metre in depth and filled in with clay. The mortar is loaded with 92 grammes of powder, which are put into the chamber by means of a bent funnel. The ball is put on to the charge, so that the axis of the screw-hole is parallel to the axis of the mortar; then the handle is screwed off, and replaced by the screw-bolt. A match is put into the touch-hole and the shot fired. The first shot is not counted, since its object is to burn out the mortar. After each shot the bore and the chamber are wiped out, and the ball cleaned. This has to be well looked after, since the slime deposited on the sides of the bore and the impurities covering the ball diminish the clearance, and thus considerably influence the distance to which the ball is thrown. The distance should be at least 235 metres, but with a bronze mortar it may be 10 metres less, since, owing to the wear, mortars, especially bronze ones, alter with use, and by and by shorter ranges are obtained with them. To avoid the introduction of errors through this, the powder to be tested is compared with a standard powder which is selected from black powder of current manufacture, and carefully kept dry in hermetically sealed glass bottles or tin boxes. The ranges of this powder at the time of test, compared with its original range with the same mortar, show the

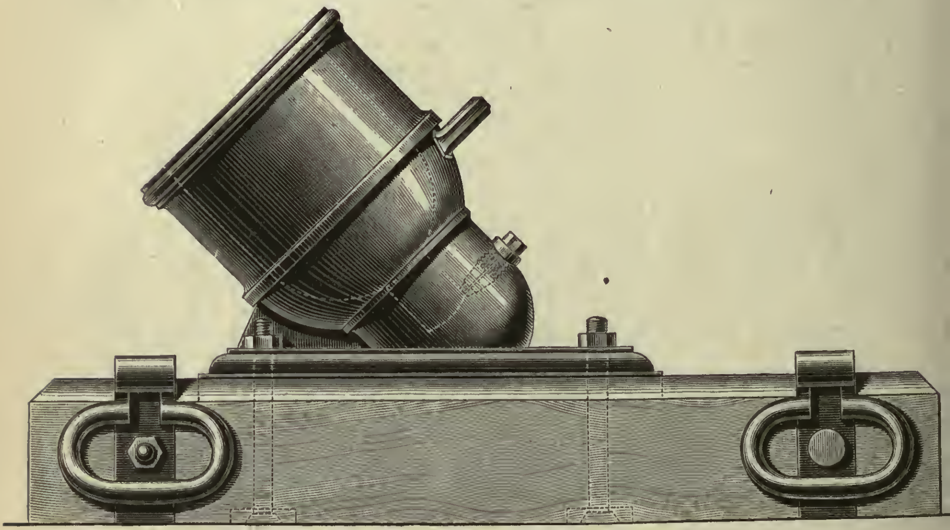


FIG. 263.

loss caused by the alterations in the instrument, and allow the necessary corrections to be made. The range of the standard powder is determined by firing six shots and taking the average of the last five. The operation must be repeated after each 25 shots fired from the mortar.

One of the chief defects of the mortar is the uncertainty due to the shot, which makes it impossible to exactly compare the results obtained at different times. This source of error can to some extent be avoided by always comparing with a standard powder as stated above, and reducing the ranges obtained to agreement with those of the standard powder obtained under the same circumstances. It has always to be considered that the clearance round the shot in the bore and the touch-hole exert considerable influence on the results obtained. The more shots are fired from the mortar, the more the touch-hole will be enlarged, and the more gas will escape without assisting in propelling the projectile. The fact that, according to the time of the day, different ranges are obtained with the same powder has also been urged against the use of mortars; but it is not at all proved that the mortar is really to blame for this, but rather the powder, since at noon, when there is a higher temperature than in the morning, the powder may also have a larger volume and also contain less moisture. The moisture of the air may also influence the flight of the ball.

Fig. 264.

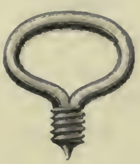


Fig. 265.

Fig. 266.

A similar mortar is used for testing high explosives, dynamite and such like, for instance, at the factories. It is either cast in one piece with its base like the mortar above described, or, as will be seen from Fig. 267, it consists of a heavy cast-iron cylinder (a) mounted in trunnions (b) and bearings (c) on a carrier (d). One of the trunnions has an index (e), and a graduated scale (f) is fixed to the carriage. This enables the mortar to be elevated to any desired angle. In order to be able to use the mortar after its chamber has been worked out, it is sometimes made with one at each end, so that when one is worn out it can simply be reversed and the other used. In this design the mortar is also better balanced. The projectile (g) is cylindrical with the edges of the ends slightly rounded off, and has an axial hole (h) of 6 millimetres in diameter passing right along the projectile. The object of this hole is to insert the fuse and caps for firing after the projectile has been put in place. Below the bore (i) of the mortar is the charging chamber (k), which is 30 millimetres deep and either 30 or preferably 60 millimetres in diameter. If the mortar has the smaller-sized charging chamber, the charge is inserted wrapped up in paraffined paper. With the large charging chamber, turned wooden cylinders are used with an axle-hole 20 millimetres in diameter about half-way through them into which the charge is put. The advantage of this is that the charge can be weighed directly into the wooden cylinders, and be carried to the mortar without loss, and also that the charging chamber is not so rapidly worn out by the destructive effect of the explosive, as at the moment of the explosion the wooden cylinder is first broken. The projectile and the bore of the mortar are machined all over exactly, and should be a good fit without being too tight. To prevent jamming, the projectile is slightly greased before being put in. The ordinary charge of dynamite for these mortars is 10

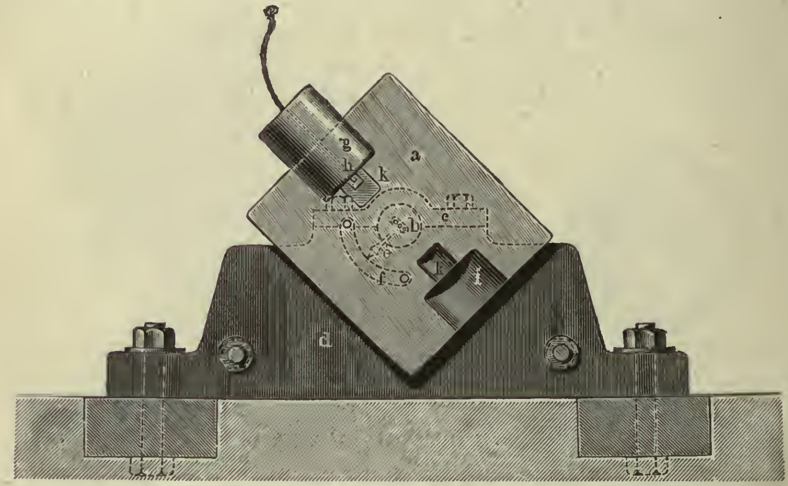


Fig. 267.—Six-100ths full size.

grammes; the projectile weighs 15 kilogrammes, and at an elevation of 45° good dynamite throws the projectile 132 metres, gelignite 165 metres, English gelatine dynamite 188 metres, and blasting gelatine 242 metres. The ranges obtained with such a mortar become slightly longer if, to compensate for the deviation from a true parabolic flight caused by the resistance of air, the elevation be 32° instead of 45°, but in both cases perfectly comparable results can be obtained if the precaution be taken of always firing a standard explosive as well.

With the mortar, as with most forms of testing apparatus, only explosives with similar properties can be compared with each other, and no conclusion can be drawn as to the comparative strength of say dynamite and blasting powder from mortar trials.

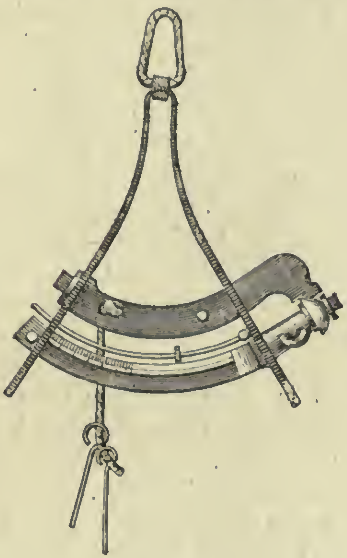


Fig. 268.

(b) *PENETRATION TEST.*

Sometimes the penetration of a powder is tested by placing a number of one-inch pine boards spaced at certain distances apart from each other, and taking as the figure of merit the number of boards through which the projectile has passed from a given range.

In a similar manner sporting powder is tested by placing together a number of sheets of brown paper or carefully-selected cardboard, and taking the penetration of a shot charge. As a rule 40 sheets of brown paper hung against an iron target are used. The number of sheets through which at least two shots have penetrated is taken as the measure of the penetration. Good black sporting powder fired from a distance of 40 yards penetrates about 26 sheets.

2. Spring Gauges.

Regnier's gauge (éprouvette à main de Regnier), Fig. 268, is used for testing sporting powder. It consists of a small gun suspended on a two-armed steel spring. The muzzle rests against the end of one arm, and the breech is attached to an iron cross-piece fixed on to the other arm. A graduated arc with 30 divisions is attached to the breech of the gun, and carries a curved rod on which an index disc of morocco leather slides. The gun is charged with one gramme of sporting powder, and in order to do this the two arms are brought together. Before firing the hand is placed exactly at zero—*i.e.* against the left arm. When the shot is fired the reading is taken from the leather index, which, as the spring recovers itself after the shot, retains the position it has been moved to and thus gives the strength of the powder. Ordinary sporting powder will give about 12 divisions and a fine one 14.

The instrument is more sensitive than the mortar, but as the resistance of the spring is liable to alter, it is always absolutely necessary to make two tests, one with the sample under examination, the other with a standard powder.

St. Remy's gauge was invented in 1697, and in shape resembles a pistol. In it there was a small vertical cylinder with a slightly rounded pawl, gearing at one end into a toothed wheel, which could turn on its axle placed about the centre of the pistol-barrel. At the end of the barrel there was a spring hooking on to the toothed wheel, which kept the pawl in the position in which it closes the cylinder. In order to charge the cylinder, the spring could be wound up by means of a screw. When the cylinder was charged the pawl was put on and the spring screwed up so that it could play freely on the wheel. The charge was then fired, and the number of teeth which the wheel turned back measured the force of the powder.

The instrument was not so good as the preceding one, and comparative trials with a standard powder were always required. In general, it must be noted that even if every single instrument is made to exactly the same pattern, each has its individual spring giving individual results. Also springs become weaker

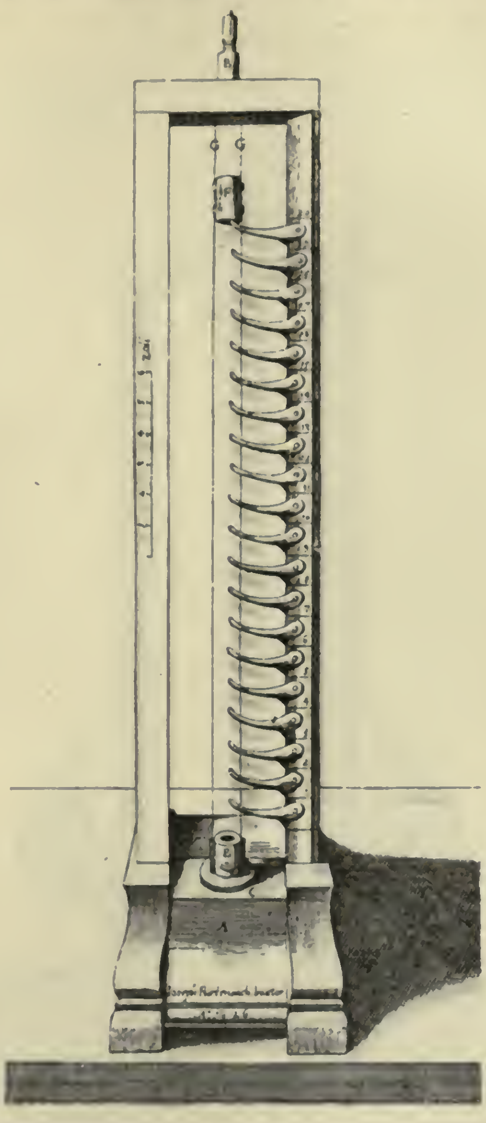


Fig. 269.

by use, and their strength depends very much on the temperature, so that they are liable to vary from day to day.

3. Weight Gauges.

(a) *RATCHET GAUGES.*

A gauge of this type was described as early as 1626 in Furttenbach's 'School of Gunnery' ('Büchsenmeistereischule').

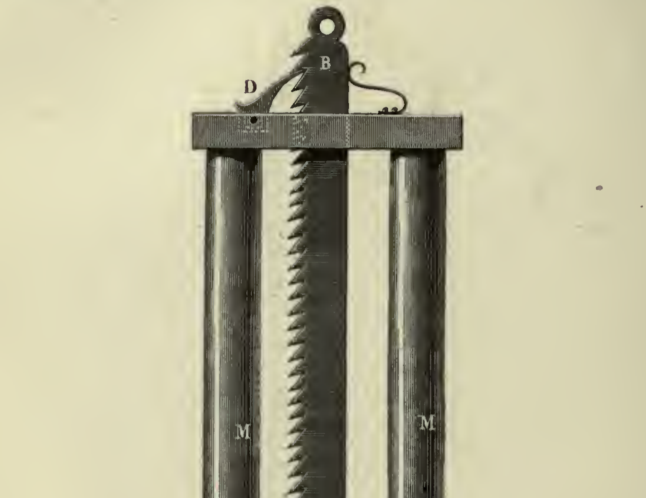


Fig. 270.

On account of its historical interest, it is illustrated here (Fig. 269). It consists of a small brass mortar (E) fixed on to a base (A) surmounted by two uprights and a cross-piece. On each side of the mortar is a brass wire (G) fixed at its lower end to the mortar and to the cross-piece at the top. These form guides for a weight (F), on the sides of which are rings through which the wires pass. The mortar is charged with the powder to be tested, and the weight let down on its upper surface; on firing, the weight is thrown up. As it moves upwards the weight easily lifts and passes a number of pawls hinged on the side of the frame, but is prevented by them from falling again, so that the height to which the weight is thrown registers the strength of the powder.

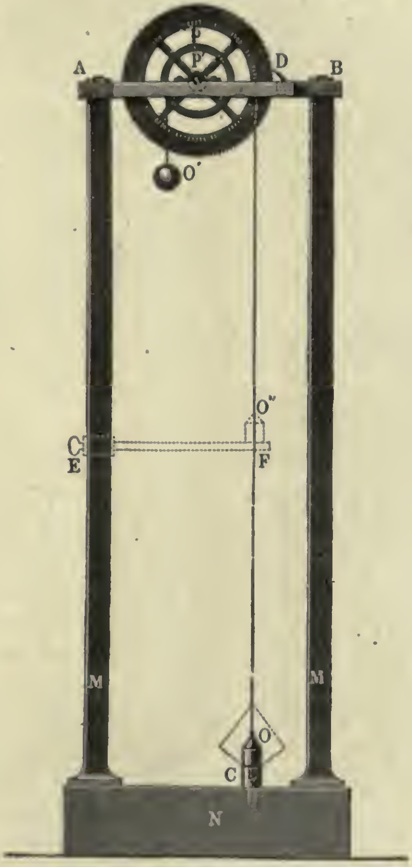


Fig. 271.

A modern form of ratchet gauge is illustrated in Fig. 270.

It consists of a small vertical mortar (C) fixed on an iron base plate, which is charged with 1·5 grammes of powder and closed by means of the block (P, P) weighing 2·5 kilogrammes. This block has a ratchet (A, B) attached to it, and can slide freely up and down the pillars (M, M). When the mortar is fired the weight is thrown up but prevented from falling down again by the pawl (D) engaging with the ratchet. The height of the throw is measured by the number of teeth it has lifted, and usually spoken of as so many degrees.

With this gauge blasting powder should lift the weight from 25 to 30, cannon powder 60 to 65, rifle powder 70 to 90, and target powder 120 to 150 degrees.

The results given by this apparatus correspond exactly with those obtained with the Regnier gauge. Its sources of error consist in the escape of gases through the touch-hole, in the friction of the weight (P, P) on the pillars (M, M), and in the variations in the resistance of the pawl.

(b) *COLSON'S GAUGE.*

This consists of a small mortar (C, Fig. 271), the chamber of which holds 2 grammes of powder. A weight (O) suspended by means of a silk cord is passed over and screwed to an easily rotating grooved wheel (p) 0·2292 metre in diameter, carried by the cross-head (A, B). On the same axle there is a second wheel (p′), the outer edge of which is toothed and its circumference graduated, and its diameter is exactly half that of the larger one. A cord with a counter-weight (O′) attached to one end passes over, and is fixed to p′ in the opposite direction to the one on p. The effect of the weight (O) is to turn the two rolls from right to left. The pawl (D) engaging into the teeth of the roll (p) prevents it from turning from left to right. The apparatus is used in the following manner:—The small mortar is charged with 2 grammes of the powder and the weight (O) lowered on to it, the small weight (O′) being thus lifted to its higher position. When the charge is fired, the weight (O) is thrown up vertically and the cord is wound on the pulley (p) by the weight (O′) descending. As soon as O reaches its highest point, it comes to rest, and then just commences to fall again, but is prevented by the pawl (D) engaging in a tooth on the large wheel, and so brought to rest. The amount this wheel has revolved can now be read off from the scale marked on it, and the force of the powder calculated.

The effect of the friction which is always present in this experiment can be eliminated by putting a board (F), which can be fixed at will by means of the screw (E), under the weight (O″) in the position marked (O″ = O) so that the weight rests on the board. The counter-weight (O′) is then lifted up to its highest position and allowed to fall down again. In this way the slack of the cord corresponding to the position of the weight (O″) is rolled up, but at the same time it obtains the momentum due to the fall of the weight (O′), and consequently the weight (O) will again be lifted a little. If this height be deducted from that obtained in the test, the true height which the weight would have reached by the force of the powder alone is obtained.

Colson designed a very ingenious arrangement for preventing loss of gas through the touch-hole. He used a wedge-shaped channel in front of the touch-hole, into which a hollow wedge exactly fitted. The latter was closed at its narrow end and had a hole on the side. The wedge was charged with powder and put into the funnel, so that its side hole came exactly opposite the touch-hole of the mortar. On firing, the flame passed through the side opening into the touch-hole, but since the recoil took place instantly, the wedge was driven further into the channel, and thus closed the touch-hole.

According to French reports this gauge will show the inflammability of the powder better than the mortar, and will even show the difference between powder made from red and black charcoal. In spite of all this, the gauge cannot be considered as perfect, since it gives the same results for a fine-grained powder as for a very inflammable one, whilst when large quantities are used in a mortar, differences due to the varying inflammability are evident.

(c) *DU PONT'S GAUGE.*

This gauge, which is largely used in America, consists of a small upright barrel, the powder chamber of which will take a rifle charge. The muzzle of the gun is closed by a weight of 2 kilogrammes which is fixed on to a lever arm. The movable end of the lever is attached to a graduated tape, which when the charge is fired is drawn up by the weight. As soon as the weight reaches its highest point, the tape is held fast by a slide. The strength of the powder is measured by the height to which the weight is thrown as shown by the tape.

The fault of this gauge is that weak, rapidly-igniting powders appear to equal stronger powders which ignite more slowly.

(d) *MEIER'S MORTAR.*

The construction of this mortar, which is used in Sweden, is in general the same as that of the ordinary mortars. The differences are that the axis of the mortar forms an angle of about 84° with the surface of the base. The bronze ball used with it weighs 14 kilogrammes, and has a hook on to which a silk tape 18 yards long and divided into inches is fixed. On firing the ball rises, and the tape runs over a glass cylinder against which it is held by a spring. By the side of the mortar there is a box filled with straw into which the ball falls. The charge used is $\frac{3}{4}$ oz. of powder. Before each test the mortar is burned out three times, then the complete charge without the ball is fired, and then the real test made. The charge is introduced by means of a funnel after the pricker has been put into the touch-hole. The pricker is then withdrawn, and meal powder put in by means of it. After each shot the mortar and the ball are wiped, first with a wet cloth and then with a dry one. One quarter of a pound out of each hundredweight of powder is tested, and if the powder throws the shot 93 per cent. of the height of the standard powder, it is accepted.

4. RECOIL GAUGES.

(a) *HOËR'S GAUGE.*

This consists of two bars connected with each other scissors-wise, and each having at one end a heavy ball, and at the other a small mortar. One mortar is charged and brought in contact with the other one, which thus forms a cover for it. On firing the distance the two balls are thrown apart is registered by a graduated arc and index.

The unequal friction in the joints, the variation in the length of arms with varying temperatures, and the small charge, all go to make this instrument very untrustworthy.

(b) *THE LEVER GAUGE.*

In Austria, a powder gauge similar in principle is used—the so-called Wagner gauge, which is illustrated in Fig. 272. On a base plate which can be made perfectly horizontal by means of levelling screws and a spirit level is fixed a strong lever (A). A double lever (B), on one arm of which there is a counterweight (E), is hinged to an arm projecting from the pillar. The arm which is not weighted carries a small mortar (D), and is fitted with a pawl (J) engaging into a graduated arc (F) attached to the pillar. When the powder explodes, the lever is thrown down by the recoil, and is held fast at the end of its path by the pawl. Since the charge the mortar will hold is only small and the touch-hole large in proportion to the charge, the results obtained with this apparatus are very unequal. Small errors in weighing also considerably affect the results obtained.

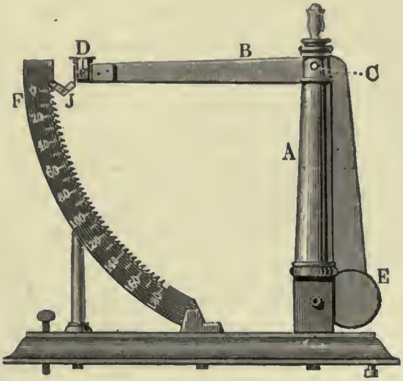


FIG. 272.

(c) *HUTTON'S GAUGE.*

This consists of a gun hung on an iron frame supported on knife edges of hardened steel, like the arms of a balance. The whole system is carried by massive stone pillars. A graduated arc, of brass or copper, measures the recoil of the gun. The arc is so graduated that seconds can be read off by means of a vernier which is moved by the recoil and retained at the end of the greatest recoil by means of a weak spring. When the gun is adjusted and at rest, its axis is horizontal, and the vernier on the zero point. The recoil of the gun measures the force of the powder, which is usually expressed in degrees.

The results obtained with this gauge are not trustworthy, since the differences between quite different kinds of powder are only slightly noticeable.

(d) *THE RIFLE PENDULUM AND THE BALLISTIC PENDULUM.*

A rifle (a, b, Fig. 273) is carried on the iron frame (o, m, n) which swings at o on knife edges. The rod (m, n), which can be fixed higher or lower by screws, carries a lead weight (p), which can be moved along it in order to bring the centre of gravity of the gun and frame vertically under the swinging point of the pendulum, whilst the axis of the rifle remains horizontal. At i there is a pin which moves a sliding index on the graduated arc (c, d). This index shows in degrees the recoil of the pendulum when the gun is fired—that is to say, the angle of swing.

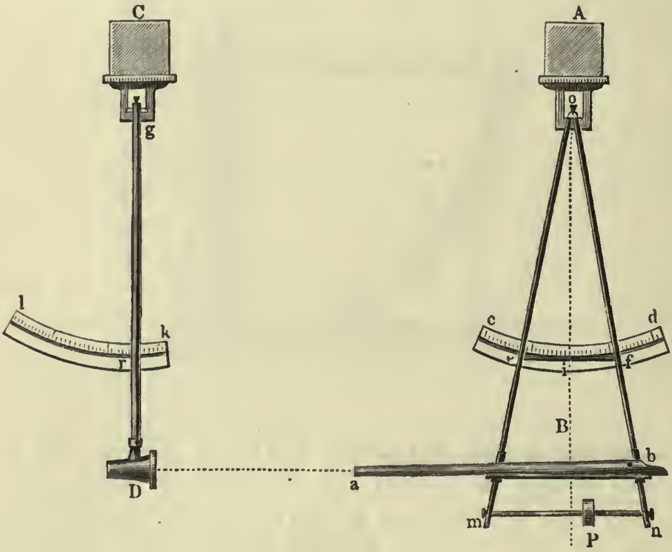


FIG. 273.

The ballistic pendulum (C, D) is supported at its upper end on two knife edges, and has a hollow cone of bronze filled with lead and fixed to its lower end, to the axis of which the swinging point of the pendulum lies normally. The lead is cast into the cone from a mould. The ball from the rifle penetrates into the lead and causes the pendulum to swing, the amount of the swing being read off from the graduated arc (l, k), and from this the force with which the ball struck the pendulum can be calculated.

The weight of each of the pendulums is 25 kilogrammes. When testing military powder, 10 grammes of powder are taken, but only 5 grammes for sporting powder. A fresh lead block is put into the pendulum for each test. The powder is put into the barrel by means of a funnel, the tube of which goes down to the powder chamber. The touch-hole of the barrel is closed by a plug, and the barrel held vertically whilst being charged. The ball is introduced by means of a rammer of 1·176 kilogrammes weight, after having had a rectangular paper weighing 0·7 gramme wrapped round it. The rammer is allowed to fall on to the charge from a height of 150 millimetres. The barrel is brought to a horizontal position, and adjusted by means of screws going through the sides of the frame. Then fine powder is put on to the touch-hole, and the shot is fired with a quick match without the barrel being touched in any way. The number of degrees marked by the index on each pendulum, and also the position of the point of penetration in relation to the axis of the gun, is noted. The muzzle velocity of the ball is calculated by the formula—

$$v = \frac{c\sqrt{(pdk + bi^2)(pd + bi)g}}{biR}$$

in which R is the radius of the axis of oscillation,

i, the distance of the axis of rotation from the centre of oscillation,

pd, the momentum of the pendulum about the axis of rotation (the product of the weight (p) of the pendulum, and the distance (d) of the centre of gravity from the axis of rotation),

pdk, the momentum of inertia of the ballistic pendulum,

g, the force of gravity,

b, the weight of the ball,

c, the chord of the recoil of the pendulum.

(e) *REGNIER'S HYDROSTATIC GAUGE.*

In this gauge the recoil of a small mortar, as registered on a float, measures the strength of the powder. The first suggestion for this apparatus was made by Bottée, who tried the effect of

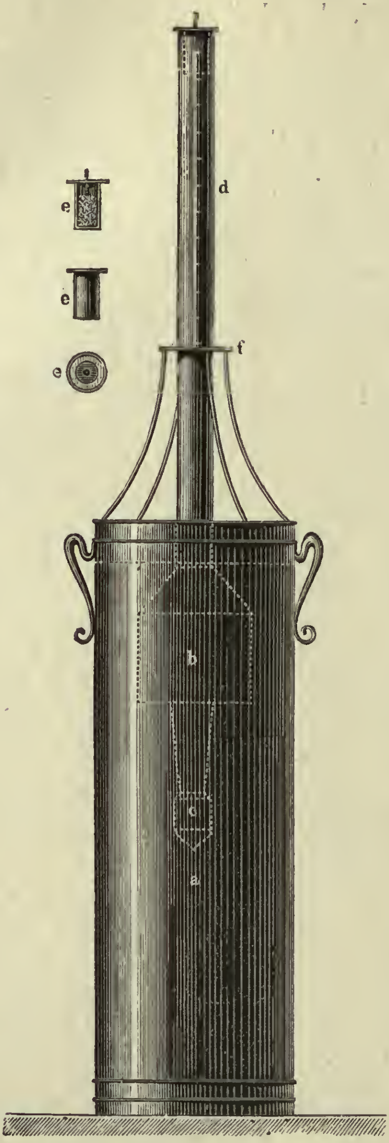


Fig. 274.

burning unconfined powder on a flat plate resting on a float. It consists of a float (b) contained in a sheet-iron vessel (a, Fig.

274). When being used b is loaded with the weight (c) in order to bring the centre of gravity of the float as low as possible. At the upper end of the float there is a polished and graduated brass tube (d), on to which a small mortar (e) is put. The mortar is provided with a cover with a small round opening, through which the charge and the fuse are introduced. The mortar will hold about 6 grammes of powder, but as a rule it is charged with 3 grammes only. The tube is guided at f by a flat brass ring carried by four legs.

In making a test, the vessel (a) is filled with water to about three-quarters of its height and kept at as constant a temperature as possible. The float with the scale and the charged mortar is placed in position, and more water poured into the vessel, until the zero point on the scale is exactly at f. Sawdust or lycopodium powder is thrown on to the surface of the water, and the charge in the mortar fired. The depth to which the float has been forced down into the water by the recoil is read off by the sawdust or lycopodium powder adhering to the scale.

A 4 grammes charge of ordinary sporting powder gives a reading of 110 scale divisions, and fine sporting powder 156.

This gauge gives exact results if the temperature of the water—that is, its density—be kept constant. The recoil may occasionally be a little smaller than it should be through some grains of powder being thrown out of the mortar without having contributed to the recoil.

(f) *MELSEN'S DYNAMETRIC GAUGE.*

The essential features of this apparatus are the same as Regnier's float gauge. The chief characteristic of this instrument is the position of the mortar, which is put on to the stem of the hydrometer, and is provided with either cylindrical or cylindro-conical perforations, on to which are fitted a number of nozzles (the so-called "lumières") with orifices corresponding to the perforations mentioned above to allow the gas to escape.

When using the instrument the hydrometer, weighing 4·950 kilogrammes, is put into a vessel of water at a temperature of exactly 15° C. A "lumière" is put on to the mortar, and a charge of 3 grammes of powder introduced. The fuse is inserted and the mortar screwed on to the stem of the hydrometer. The latter is then weighted by means of shot until the zero point of the scale coincides as nearly as possible with the surface of the water. Then a thin layer of lacquer containing powdered brick, powdered bolus, etc., is painted on to the scale of the float by means of a brush. The charge is then fired, and the number of divisions the instrument has been forced into the water by the explosion is noted.

The advantage of this gauge compared with Regnier's is that one need not depend on experiments made under one set of conditions alone, but can use different mortars and "lumières" in testing the powder, and thus get a knowledge of its properties which will enable one to estimate in advance its effect in the barrel of a gun.

B. ELECTRO-BALLISTIC APPARATUS.

The first idea of using electricity in ballistic experiments came from Prussia, where, in 1838, the testing commission of the artillery had an electro-ballistic apparatus constructed for measuring the muzzle velocity of a projectile. Since that time the number of different types of such apparatus has considerably increased.

In classifying the methods of measuring the velocity of projectiles, a distinction can be made between those in which the time is indicated directly by the apparatus (for instance, by means of a clock, a pin, or similar apparatus), and those in which the time of the phenomenon being investigated is calculated from the measured duration of another one occurring simultaneously.

1. APPARATUS IN WHICH THE TIME IS INDICATED DIRECTLY BY THE APPARATUS.

(a) *WHEATSTONE'S ELECTRO-MAGNETIC CHRONOSCOPE.*

This instrument was first described by Wheatstone in 1840, and has been subsequently improved by Hipp. It measures the time taken by the projectile of a rifle in passing over a certain known short space. It is so arranged that the projectile on issuing breaks a wire which is stretched across the barrel and connected in an electric circuit, and thereby interrupts the current. At the same moment the hands of a dial begin to revolve with a known velocity. As soon as the projectile arrives at its end, it hits a movable surface, presses it back a short way, and thereby closes the electric circuit and brings the hand to a standstill. The muzzle velocity can be calculated from the time required for the flight.

Ballistic experiments made with this apparatus gave relatively large differences in the results; for instance, under exactly similar circumstances the velocity per second varied between 38·5 and 88·9 metres. Hence the apparatus has never been used in practice. Kuhn tried to improve it by using the recoil of the gun to open the circuit and to release the hand, so that the wires stretched at the muzzle of the barrel were dispensed with, but nothing has been published on the serviceableness of the apparatus in this form.

(b) *MARTIN DE BRETTE'S ELECTRIC CHRONOGRAPH.*

Round a metal cylinder covered with chemically-prepared paper, a platinum pin is revolved by means of a centrifugal pendulum. As soon as the ball breaks the wire stretched at the muzzle of the gun the circuit of an induction coil is opened, and the spark formed passing from the cylinder to the platinum pin perforates the paper. This again occurs when the projectile has reached the target.

The whole apparatus is nothing but a modification of the well-known apparatus of Siemens, in which the cylinder rotates in front of the pin, and the sparks fly direct to the cylinder. Both apparatus have been out of use for a long time.

2. Apparatus where the Time is calculated from the known Duration of another Phenomenon.

(a) *POUILLET'S GALVANOMETER.*

This instrument was introduced in 1844. The muzzle velocity is calculated by it from the deviation of a magnetic needle by an electric current. The strength of the current is known, and the time that it has flowed, which is controlled by the passage of the projectile through a known distance, is calculated from the swing of the needle. To make a determination, the galvanometer is connected up in circuit in such a way that the projectile closes the circuit as it leaves the gun, and opens it again the moment it hits the target. If the time elapsing between the making and breaking of the current is very short, it is difficult to calculate the velocity from the very small deflections that will be obtained. For this reason Pouillet constructed a table from which the time of the duration of the current could be seen for various deflections of the magnetic needle. This table is not free from errors, inasmuch as it depends on the commutator disc having a perfectly uniform strength of current, which is not the case, and any secondary currents which may be set up are not considered at all. Helmholtz, therefore, slightly modified this method. To calculate by his method the duration of a current, which is exactly equal to the duration of a phenomenon to be investigated, it is necessary to know the deflection or half the angle of swing both before and after the measuring current has acted, the period of swing of the needles, and finally the deflection which the measuring current would produce if it were constant in value. Since all these elements can be measured exactly, and all disturbing influences allowed for, the method is a very exact one. The troublesome manipulation seems to have been the cause of its not being used for ballistic purposes.

(b) *NAVEZ'S ELECTRO-BALLISTIC PENDULUM.*

This consists of three parts—the pendulum, the clock, and the interruptor.

The main part of this apparatus is a pendulum with a lenticular brass "bob" and an index-pointer swinging on the same centre as the pendulum. They are arranged to swing through an angle of 150° in front of a circular scale divided into degrees and twentieths. The pendulum is held in its initial position by an electro-magnet, the actuating current of which flows through a wire crossing the muzzle of the rifle. The magnet acts directly on a piece of iron let into the pendulum bob, and the index-pointer can be stopped by a second electro-magnet without bringing the pendulum to a standstill. This second magnet acts direct on to an iron disc attached to the index-pointer.

The arrangement for closing the circuit consists of an electro-magnet moving along a vertical bar, the magnet being excited by a current going through a target frame placed at a known distance from the muzzle. A wire in circuit with the closing arrangements is stretched on this target, and is broken by the shot hitting the target. The electro-magnet has on its lower pole surface a small pear-shaped lead weight, which for this purpose has a steel point where it touches the pole surface. The current is interrupted when the projectile penetrates the target, and thereby the lead weight falls on to a small metal plate in the circuit. The metal plate is bent by the pressure of the lead weight, and thereby the current is closed, by which that electro-magnet is actuated which has to arrest the hand of the pendulum.

The interruptor is a small instrument with a key for determining the loss of time between the action of the two electro-magnets due to the falling of the lead weight on to the metal plate. The interruptor enables both currents to be simultaneously broken, and the angle or swing that takes place when this is done must be deducted from that obtained when the interruptions are made one after the other by the projectile on making the experiment. The time is calculated from the swing in degrees.

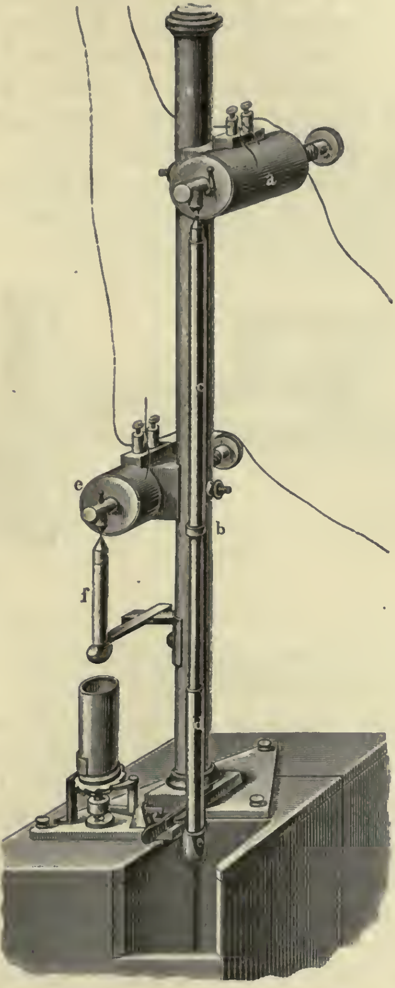


FIG. 275.

In this calculation the time required for the electro-magnet to release the piece of iron let into the bob of the pendulum, the time the lead weight takes to fall, and also the time required for the closing of the first circuit by the projectile, and for the disappearance of the magnetism in the electro-magnet of the closing arrangement, are allowed for. Small errors may occur in spite of everything, but they will be considerably larger if all the precautions prescribed by the inventor be not taken in making the experiment. These are mainly complete protection of the apparatus from air currents, dust, and moisture, prevention of any accidental vibration of the pendulum or the closing arrangement, and maintaining a definite temperature.

If the temperature in the experimenting-room sink below 12·5° C., the results are so irregular that no value can be attached to them.

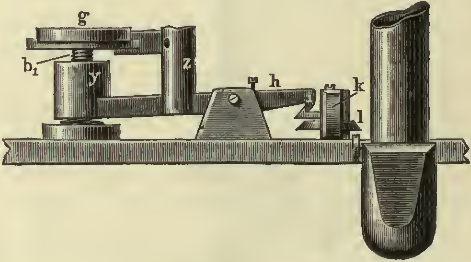


FIG. 276.

(c) *LE BOULENGE'S ELECTRO-BALLISTIC CHRONOGRAPH.*

At present Le Boulengé's chronograph is the apparatus most frequently used for determining the velocity of projectiles. It measures the time taken by a projectile in passing through a known distance in terms of the space passed through by a freely falling body in the same time. The projectile in its flight first cuts through two wire frames, or sometimes a wire stretched over the muzzle of the rifle as it leaves the barrel, and then hits a target at a certain distance, which replaces the second wire frame. The freely falling body is supported by an electro-magnet on the chronograph, and is released when the current flowing round the magnet is interrupted, the time of fall being shown by a mark cut into the free falling body by means of a knife put in action by the cutting of the wire on the second frame.

The apparatus is illustrated in Fig. 275. It consists of a column (b) with a horizontal base, which can be levelled by means of three adjusting screws, and two electro-magnets (a and e). The electro-magnet (a) carries the chronograph-bar (c), which is tipped with iron at the top, and has a slightly flattened head at the bottom. Over the bar either a zinc or a copper tube is slipped. The electro-magnet (e) also supports a rod, similar to the chronometer-bar, the so-called registrar (f), which also has a point at the top, and a flattened head at the bottom. On the base plate is situated the trigger (détente), which is illustrated on an enlarged scale in Fig. 276. It consists of a double lever (h) turning in two bearings; the shorter arm ends in a claw (i), and on the end of the longer there is a hollow cylinder (y). Between y and the bearing there is a vertical arm (z), from which projects a pawl (n) engaging with a toothed plate (g). This plate is connected with the hollow cylinder (y) by a screw (b_1). A spiral spring below the hollow cylinder tends to lift it, and thereby to press the claw (i) downwards. The claw at the end of h engages with a second one fixed to a long flat spring (k) attached to the foot of the column. A screw passing through this spring forms the arbor for a hardened steel disc (l). A hollow brass cylinder (*vide* Fig. 275) is placed over the plate (g) to catch the weight when it falls.

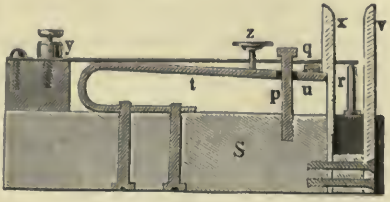


FIG. 277.

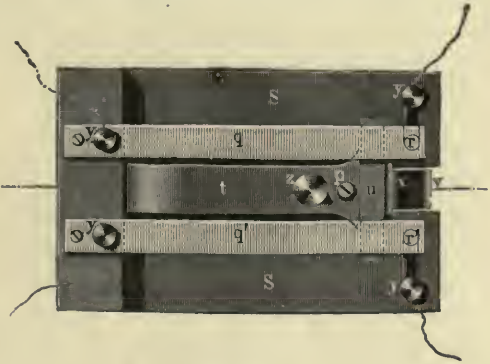


FIG. 278.

The interruptor (disjoncteur), which is shown in Figs. 277 and 278, is also an important adjunct. Its purpose is to simultaneously interrupt the two circuits. It has two pairs of terminals (y) fixed to a wooden base, one pair being connected with the electro-magnets of the chronograph, the other with the main circuits going to the wire frames, and each to its fellow by spring plates (q and q′). Between the two plates is a spring (t) guarded by a screw (p), which can be pressed down by a knob (z), when it is caught and held down by the vertical spring (x). In this position the plates (q and q′) are in contact with two pins connected electrically with the two right-hand terminals (y, y), and thus make connection between the two pairs of terminals and close the two circuits. On pressing back the spring (x), t springs back to its original position, and lifts q and q′ off the contacts (r and r′), thus interrupting the two circuits simultaneously.

The frames for holding the wires are made of wood, and the wire itself of copper, of as brittle a nature as possible. The wires are about $\frac{1}{50}$ inch thick, and are stretched zigzag fashion over the frame. When the projectile breaks the wire in the first frame, the first current is interrupted, the electro-magnet (a), on which the chronometer-bar is hung perfectly vertically, loses its magnetic power, and the chronometer-bar falls down. As soon as the other circuit is interrupted by the breaking of the wire in the second frame, the electro-magnet (e) also loses its magnetism, the registrar falls on to the plate (g), strikes down the arm of the lever, and thus releases k, which springs forwards and cuts a mark in the chronometer-bar, or rather into its zinc or copper sheath. If the point on the sheath which was opposite the knife when the apparatus was at rest be known, the velocity of the projectile can be calculated from the distance of this point from the cut made on the sheath when the second current was broken.

The manner of connecting up the frames and chronograph in circuit is clearly shown in Fig. 279. Here E is the upper electro-magnet, E_1 the lower electro-magnet, C the chronometer-bar, C_1 the registrar, D the interruptor, l and l_1 are the contacts, m and m_1 the terminals, B the battery for the first wire frame, (R), B_1 the battery for the second frame (R_1).

Before using the chronograph, the correction for the time the various parts require to come into action must be determined. These are the time required for the de-magnetization of e for the fall of the weight on to the plate, for the disconnection of the knife, and for the travel of the knife from the point where it is held to the chronometer-bar. On the other hand, the electro-magnet (a) also requires a certain time for de-magnetization, which would have to be deducted from the sum of the other corrections. If, then, both currents be interrupted simultaneously, and thereby a mark ("disjonction") is cut on to the lower part of the chronometer-bar, this will form a zero point for the apparatus, showing the necessary correction. This correction can be reduced in such a way that its value is always 110·37 millimetres, corresponding to a duration of 0·15 second.

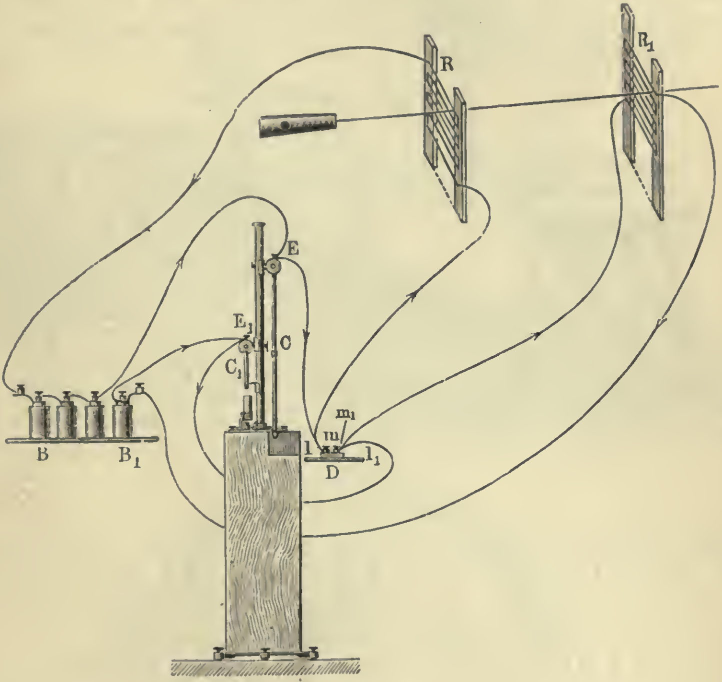


Fig. 279.

If the distance of the cut made by the shot passing through the second frame to the zero point be called h, then according to the laws of falling bodies, $h = \frac{1}{2} gt^2$, and $t = \sqrt{\frac{2h}{g}}$ Assuming the projectile has only a velocity of 500 metres per second, and the two frames are placed at 25 metres apart, it will have required $\frac{25}{500}$ or 0·05 second. The chronograph will, therefore, indicate $0{\cdot}15 + 0{\cdot}05 = 0{\cdot}20$ second. The distance between the cuts on the bar will therefore be $h = 0{\cdot}20^2 . \frac{9810}{2} = 196{\cdot}20$ millimetres. If, inversely, the velocity corresponding to certain distances between the two cuts be calculated from this formula in advance, and marked on a scale, the velocity of the projectile can at any time be read off at once by merely measuring the two cuts. For this purpose a scale is used, one end of which is put into a perforation in the flattened head of the chronometer-bar, and a vernier moved along the scale until it corresponds to the second cut, and the distance between the interruptor cut and the second one read off direct. Tables have been calculated by the manufacturers of chronographs for various distances between the wire frames or interruptions of current, and are supplied with the apparatus.

The velocity measured by the chronograph is the velocity of the projectile half-way between the two wire frames. It can be reduced to muzzle, or initial, velocity by the ballistic formula—

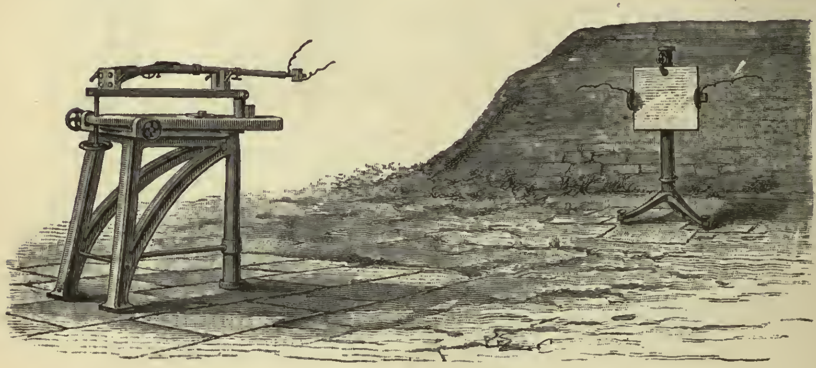


Fig. 280.

$$V_a = \frac{v}{\sqrt{1 - m\delta v^2 x}}$$

in which v is the mean velocity as measured by the chronograph,

x, the distance of the point half-way between the frames from the muzzle,

δ, the density of the air,

m, the ballistic co-efficient (about 1·2).

With cannon the two frames are placed 50 metres apart, the first frame being 25 metres in front of the cannon. With sporting guns the wire is stretched immediately in front of the muzzle, and the second frame replaced by a target with a contact plate. Such a target, together with a carrier for the rifle, as made by A. and R. Hahn of Cassel, is illustrated in Fig. 280.

(d) *LE BOULENGÉ'S ELECTRIC KLEPSYDRA.*

The Le Boulengé chronograph is only suitable for determining comparatively short durations of time, since the heights of fall increase exceedingly rapidly with the time. Le Boulengé tried to overcome this difficulty by employing as a chronometer the quantity of liquid running out from a vessel as determined by its weight during the interval of time to be measured. The outflow of the liquid being regulated by electro-magnetism, he called his apparatus "Electric Klepsydra," from the name of the ancient form of time-measurer, which marked the time by the running out of a determined quantity of water from a vessel.

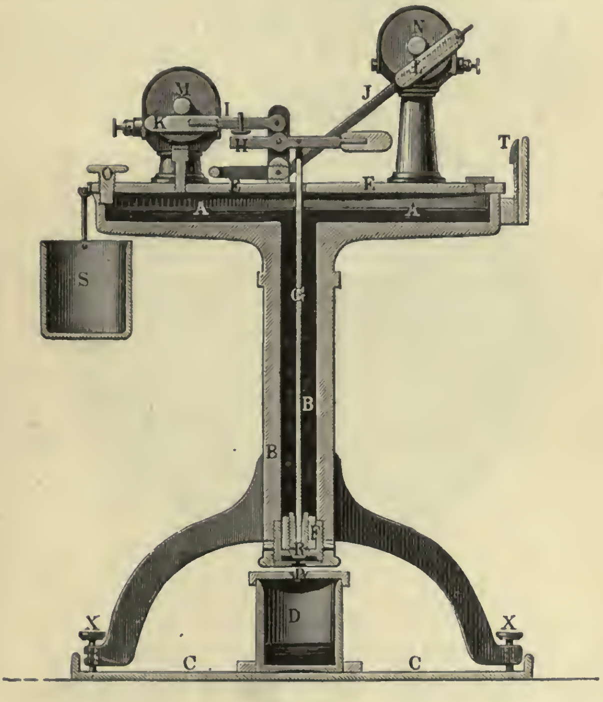


Fig. 281.

The apparatus, Figs. 281 and 282, consists of a round vessel (A) 20 centimetres in diameter and 3 centimetres high, which is carried by and is connected with a hollow central column (B) 20 centimetres high, terminating in a tripod with adjusting screws (X).

This vessel is of cast-iron, and stands on a round cast-iron plate (C), with a rim to take up any mercury which may accidentally have run out from the receiving vessel (D). A cast-iron disc (E) covers the vessel (A), and at the same time carries the mechanism of the apparatus. At the bottom of B there is an opening through a thin disc, which is closed by a conical valve to prevent the mercury from running out. The disc with the opening, the body of the valve (R), and the bearing (F) on which it rests, are made of steel. A rigid bar (G), which is connected with the valve at its lower end, passes through a central opening in the top cover (E). It is attached at its top end to a horizontal lever (H), the so-called valve lever. If the left-hand end of this lever be pressed down, the valve opens, and the mercury runs out, and when the pressure is withdrawn the valve closes, and the running out of the mercury is stopped.

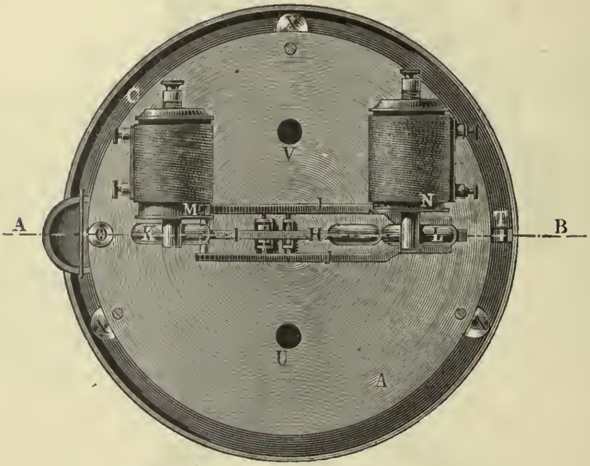


FIG. 282.

The opening and closing of the valve is done by two levers (I and J), which are fitted with soft wrought-iron weights (K and L). Normally they are held up by the electro-magnets (M and N) against their poles. The lever (J) closing the valve is made of two parallel pieces, which are connected on the one side by the wrought-iron fitting, and on the other side by a bolt by which the lever is held.

If the current working the electro-magnet (M) be interrupted, the opening lever (I) falls on to the valve lever, opens the valve, and the mercury runs into the receiver (D). If the second current which actuates the electro-magnet (N) be interrupted, the lever (J) drops down and lifts the opening lever (I) to its former position. By this means the valve lever becomes free, the valve falls back into its seat, and the mercury ceases to run out. A stop spring (T) prevents the closing lever (J) from vibrating after its fall.

On using the apparatus, the two currents are interrupted by the projectile in succession. The mercury begins to run out as soon as the projectile breaks the first wire frame, and is stopped when it touches the second one.

If we assume that the rate of outflow is constant, and call P_1 the weight of the mercury which runs into the receiver (D),

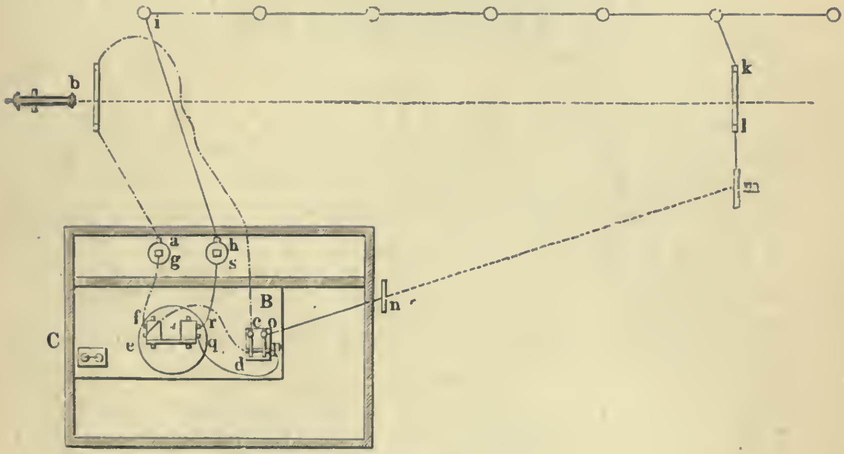


FIG. 283.

and P the quantity of mercury which passes per second through the opening with a continuous flow, then $\frac{P_1}{P}$ expresses the time which has elapsed between the moment of opening and the moment of closing the valve. The quotient $\frac{P_1}{P}$ would also indicate the time which elapsed between interrupting the two electric currents, if the valve opened and closed instantaneously as the corresponding current was interrupted. On interrupting the first current, however, some time is required to let the lever fall, and also some for the valve to lift completely; in a similar manner, time is also lost between breaking the current for closing the valve and the stopping of the flow.

To avoid determining these times independently, Le Boulengé used the method of simultaneous interruption of the current, a method already described in connection with his chronograph. If both currents be broken simultaneously, the first lever will open the valve a certain time sooner than the second one will close it. If the weight of the mercury which runs out when this simultaneous interruption is made be called p, the quotient $\frac{P_1 - p}{P}$ is the time which has elapsed between the interruption of the two currents.

The arrangement of the apparatus, as far as its use in connection with shot velocities is concerned, may be seen from Fig. 283. The opening circuit (a, b, c, d, e, f, g) contains the first frame (b), the switch, and the opening electro-magnet (a, g). It passes in front of the muzzle of the gun, either on to an ordinary frame placed 10 metres in front of the gun, or through a simple metal wire stretched in front of the muzzle.

The closing circuit (h, i, k, l, m, n, o, p, q, r, s) contains the second frame, the switch, and the closing electro-magnet (h, s). The current is brought through the circuit (i, k) to the frame (k, l) and goes through the metal plate (m) into the ground. A second metal plate (n), also placed in water or moist earth, completes the circuit to the apparatus.

After these connections are made, it must be seen whether the currents are strong enough to hold the levers of the klepsydra. The level of the mercury is then noted, and the simultaneous disconnection is made, three of these being made before each shot. Since the mercury always runs into the same vessel, its total weight divided by three is that of one disconnection. When this has been determined the test with the rifle can be made.

The weighing is done on a balance weighing to half a centigramme. This degree of exactitude is perfectly sufficient, since half a centigramme corresponds to a time of less than $\frac{1}{1200}$ of a second.

In order to be able to use the apparatus for the determination of the time of flight of shell fired from mortars also, Le Boulengé used the vibration of the ground set up in the neighbourhood of the point where the shell fell to interrupt the closing circuit of the electro-magnet (N, Fig. 281), as it is not easy to interrupt the second current on a frame on account of the great deviation in the flight of mortar shell.

For this purpose, a small box, which contained the apparatus illustrated in Figs. 284 and 285, was placed at the base of the bar at which aim was taken. It consists of an electro-magnet (A), held by a lever (B) with a wrought-iron weight (C) at one end. This electro-magnet is energized by a current (a, b, c, d). The closing circuit of the klepsydra passes to the pivot of the lever through the conductor (g), and to the core of the electro-magnet through the conductor (f), so that this current is interrupted as soon as the lever (B) falls away from the magnet. The lifting power of the magnet is regulated by a movable core in such a way that the lever is detached on the slightest shock, so that the vibration due to a shell falling in the neighbourhood of the box interrupts the current (f, g).

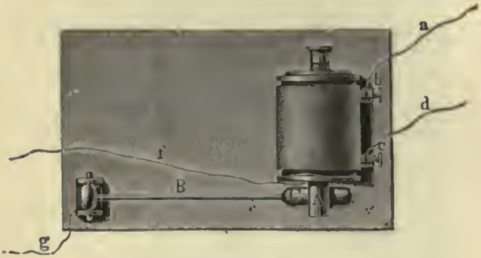


Fig. 284.

The times of flight of shells weighing 58·7 kilogrammes, with a charge of 1·240 kilogrammes, were measured by this method by means of the klepsydra. For a range of 1000 metres the average time of flight was 15·0388 seconds.

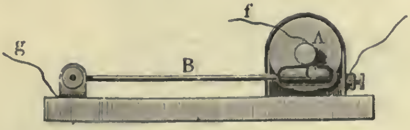


Fig. 285.

(e) *BASHFORTH'S CHRONOGRAPH.*

Navez's apparatus, and also that of Le Boulengé, only allow the velocity at one point of the flight to be determined, and the chronograph constructed by Mr. Bashforth in 1865, by means of which the velocity of a projectile can be determined at many points of its flight, was a considerable improvement. The apparatus reminds one in some respects of that of Martin de

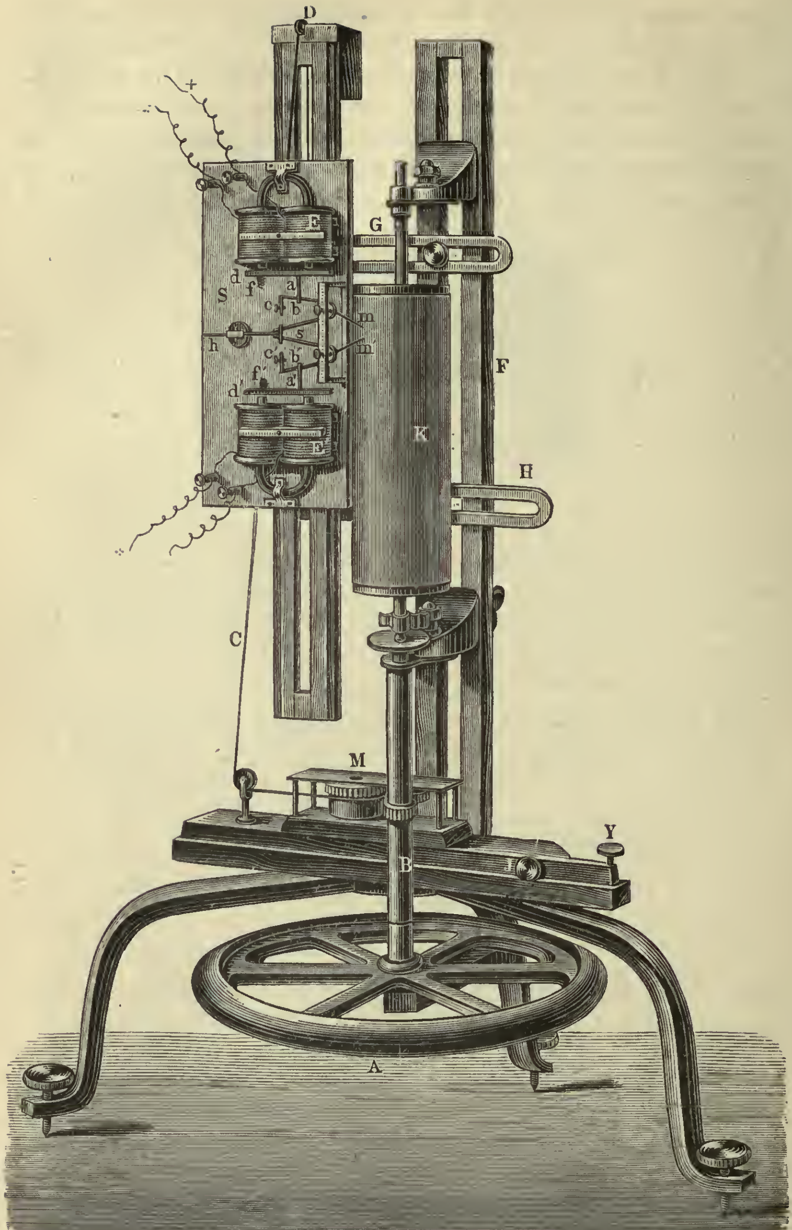


Fig. 286.

Brette, since a revolving cylinder and a marking pencil are the chief points in it.

Fig. 286 shows the arrangement of the Bashforth chronograph. K is a drum covered with specially prepared paper, on which the records are made. Connected with the axle of the drum is a shaft (B), carrying a fly-wheel (A). On the shaft (B) there is a pinion, which drives the cord-drum (M) by means of an intermediate wheel. A cord (C) passes upwards from M over a guide wheel to the wheel (D) at the top of the apparatus, and then downwards to an attachment on the sliding carriage (S). The frame (S) slides in, and is supported by two adjustable brackets (G and H) on the main standard. On S are carried the two marking-points

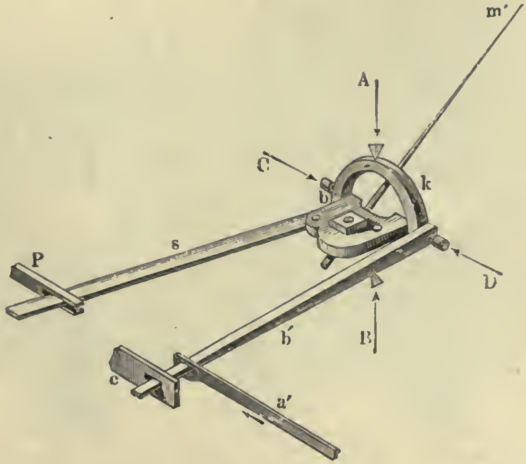


Fig. 287.

(m and m′), and the electro-magnets (E and E′) controlling them. E and E′ form parts of two independent electric circuits, one of which is interrupted by the projectile passing through a wire screen, as described above in connection with the Le Boulengé chronograph, and the other at regular intervals of time by a clock. When a series of measurements are to be taken, the fly-wheel (A) is set turning at about 90 revolutions per minute, the record-drum (K) rotates with it, and also the drum (M), the cord on which unwinds and allows the sliding-carriage (S) to descend. The marking-points, so long as their position is unaltered, will then describe a spiral line on the prepared paper on K. The position of these marking-points, however, depends on the position of the armatures (d and d′) of the electro-magnets (E and E′).

Fig. 287 shows the arrangement of the marking-points on an enlarged scale, and their action is as follows:—

When the current is interrupted, the armature (D) is pulled away from the magnet poles by the spring (f), and by means of the levers (a′ and b′, Fig. 287) the marking-point (m′) is suddenly moved to one side, which causes an abrupt vertical displacement in the spiral line it is drawing. When the circuit is again made, the reverse action occurs, and another vertical displacement of the spiral takes place—in the opposite direction, of course. The movement of m′ is limited by the support (k), which forms a gimbal mounting for it. k turns on the centres (A, B) and the lever (s) and the marking-point (m) about the line C, D, so that by means of h (Fig. 286) the points (m and m′) can be lifted off or put in contact with the paper on K. The time elapsing between the displacement is measured by a dividing engine. The upper marking-point (m) gives the breaking of the wires by the projectile, the lower end (m′) is moved by the current in E′, being interrupted at each double beat of a half-second pendulum, so that the spiral it draws gives the corresponding times at which the wires were broken, and thus the velocity of the projectile.

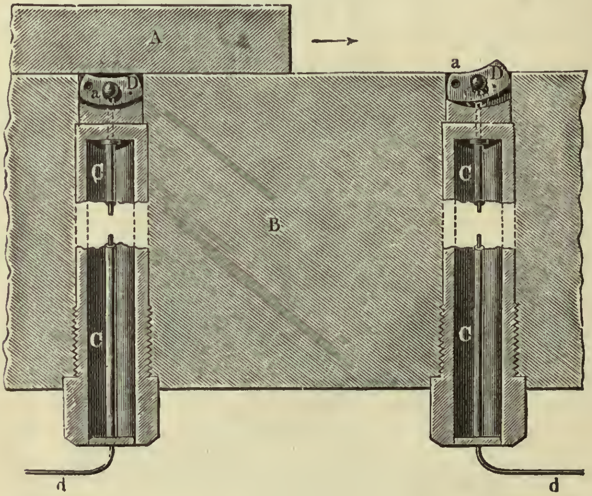


Fig. 288.

(f) *NOBLE'S CHRONOGRAPH.*

This chronograph differs from all the electric apparatus described above, in so far that it is designed to measure the velocity of the projectile not outside the barrel of the gun, but inside it. Sir Andrew Noble effects this object by registering by means of electric currents on discs rotating with large but uniform angular velocity the moment at which the projectile attains a series of definite positions in the barrel.

The apparatus consists principally of several thin metal discs, 3 feet in circumference, fixed on to a common insulated shaft. In order to rotate these discs rapidly, a heavy falling weight acting through a suitable quadruple gearing is used, and in order to increase the circumferential velocity of the discs, the action of the falling weight is assisted by a handle on the second counter-shaft, so that the discs revolve with a linear circumferential velocity of 1000 inches per second. On the third counter-shaft there is a clock-work, which can be clutched in and out at will, and which shows the time taken to make a certain number of revolutions exactly to one-tenth of a second. The peripheries of the metal discs are covered with paper strips coated with lamp-black, and are connected with one of the secondary wires of an induction

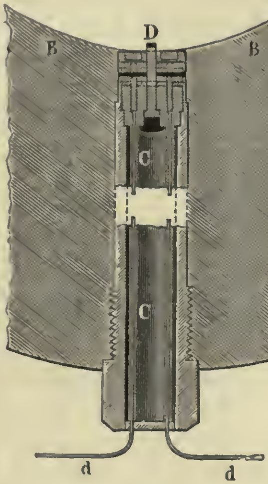


FIG. 289.

apparatus, whilst the other secondary wire is carefully insulated and connected with a discharger, which is placed opposite the periphery of the corresponding disc at a small distance from it.

Cylinders (C), shown in longitudinal section in Fig. 288 and cross section in Fig. 289, are screwed into the wall of the gun-barrel. Where the cylinders enter the bore of the gun they have a rocking-piece (D), through an opening in which (a, Fig. 288) the primary wire (d), which enters at one end of the cylinder, issues on the other side (Fig. 289). The two ends of this wire are connected up to the main conductors leading to the apparatus, as soon as the cylinders are screwed into the gun.

On a shot being fired, the projectile (A) presses down the rocker (D), whereby the wire (d) passing through it at (a) is cut and the primary circuit of the induction coil broken. This sets up an induction current in the secondary wire of the coil, and a spark instantly jumps across from the discharger to the rotating disc. This burns the lamp-black on the paper strip, and leaves a well-marked white spot. The same takes place when the rocker of the second cylinder is pressed down, and so on.

After the experiment is finished, the series of sparks on the metal disc are examined, the mark on the first disc being placed exactly opposite the point of the corresponding discharger by means of a micrometer screw. A vernier is fixed on to the end of the shaft and placed on to the zero point. The other discs are treated in a similar way. From the position of these points on the various discs, and by the circumferential velocity of the latter, the time which the projectile requires for travelling along the bore of the gun from one cylinder to the other can be determined.

To control the readings of the apparatus, an arrangement has been fitted so that one disc, one discharger, and one induction apparatus always form, as it were, a measuring instrument by themselves, which marks the place of the spark as soon as the primary wire has been cut through; thus all the white points on all the discs must lie in a straight line parallel to the shaft, if all flaps be pressed down at the same moment, whilst deviations from the straight line show the errors occurring.

In order to produce simultaneous cutting of the primary wires, all wires are fixed on to a small frame standing near the muzzle of the gun, and are cut simultaneously by means of a square-headed projectile.

Fault has been found with this chronograph, because of the manner in which the wires are broken by the projectile, which is not at all free from objection. The force required to press down the rocker is so considerable, and the impact of the projectile so great, that deep longitudinal grooves are frequently formed on the surface of the latter, which must have diminished the velocity. Also the manner of determining the number of revolutions of the metal discs is not exact, since the clock-work is not only started by the apparatus, but also stopped by it.

The table of experiments published by the English commission shows in general fair results, but also some which indicate that there is room for improvement in the apparatus. Thus with one shot the time between the third and fourth cylinder was 0·000496, and with another shot 0·000525 second; the calculated velocity in feet per second for the first shot is 1030·5, and that for the second 973, so that there is a difference between the two shots of 57·5 feet.

(g) *SCHULTZ'S CHRONOGRAPH (IMPROVED BY MARCEL DEPREZ).*

In 1864 Captain Schultz constructed a chronograph in which a series of sparks passed from a platinum point in circuit with an induction coil to a revolving blackened cylinder, mounted on a horizontal shaft and rotated by a falling weight. The sparks left traces which could be read with a microscope on the blackened surface. At the same time an electrically actuated tuning-fork drew a curve on the drum, and thus enabled the time which had elapsed between one spark and the other to be determined.

Although this chronograph gave very exact results, and had a maximum error of reading of only 0·00001 second, the actuation of the tuning-fork and maintaining it in regular motion was not easy, whilst the registration of the spark lines was difficult, and could not always be done with regularity.

Marcel Deprez subsequently brought this apparatus to great perfection. In the first instance, he so altered the tuning-fork that at the moment the current was interrupted, it started vibrating by itself. For this purpose he attached to the tuning-fork, as Mercadier had suggested, a fine wire of exactly such a length that, when it vibrated, the point in contact with the cylinder was at the centre of the vibration. In order to keep the tuning-fork—which, of course, was very delicate—in good order, and at the same time to make it work automatically, Deprez arranged two contact screws which could be so regulated that the tuning-fork had only a very limited play, and consequently was constantly repelled and then attracted at the interruption of the current. After some experiments, this regulation could be made with such accuracy that with a sufficiently powerful battery the tuning-fork could be actuated automatically, which was made evident by the uniform note given. Instead of registering by an induction spark, Deprez used a pen which glided along the blackened surface of the cylinder, but which at the moment the current was interrupted was slightly displaced sideways, so that a break (crochet) in the line traced by it occurred, the pen continuing to draw a straight line until the current was again closed, when a break in the opposite direction was formed.

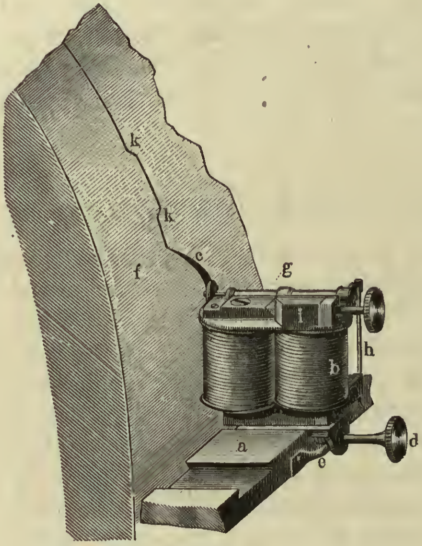


FIG. 290.

Details of this arrangement are shown in Fig. 290 and the complete arrangement in Fig. 291. On a straight bar (a) 10 electro-magnets (b), with the same number of pens (c), are fixed, and they can be so regulated by means of a screw (d) and spring (e) underneath them that they just touch the surface (f) of the drum. The armature of the electro-magnet carries the pen. It only weighs a few milligrammes and is fixed on a horizontal pivot (g), on one side of which the pen is fixed, and on the other a small lever held back by the india-rubber band (h). The proper tension is given to this by a small iron weight (i), which rests on the magnet. At the moment the current is interrupted, the armature is moved sideways, and the before-mentioned break in the curve (k) is formed, and another made in the opposite direction, when the armature is attracted to the magnet, on the circuit being again closed.

By means of this registering apparatus, Deprez has reduced the time elapsing between the interruption of the current and the action of the pen to $\frac{1}{3000}$ of a second, whilst with ordinary electro-magnets nearly $\frac{1}{100}$ second is lost between the time when the current is interrupted and the magnet energized and *vice-versâ.*

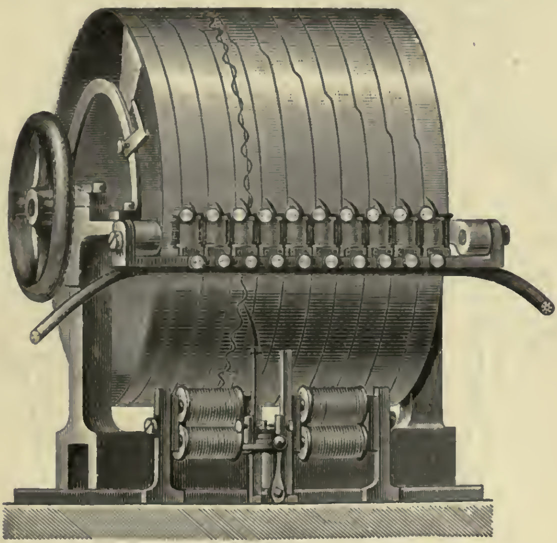


Fig. 291.

For still more exact measurement, Deprez has constructed other registering apparatus, in which the rubber cord is replaced by a spring, and which have various other mechanical advantages.

(h) *MAHIEU'S ELECTRO-BALLISTIC CHRONOGRAPH.*

An excellent solution of the problem of electro-ballistic chronographs was found by Lieutenant Henri Mahieu, of the Belgian artillery, the description of which is taken from Maudry's treatise on arms. Mahieu uses the tuning-fork, the instrument at present universally recognized as the best for such small measurements of time, to register the time elapsing during the flight of the projectile. He makes use of the method of disjunction whereby the errors, or retardations, of the apparatus can be eliminated, and besides this has arranged his apparatus to measure either a short or a long single time or several consecutive short periods during the flight of a projectile successively. The method of disjunction consists of interposing a circuit-breaking apparatus (the disjoncteur), similar to Le Boulengé's, which allows all currents to be simultaneously interrupted, when the chronograph gives the times by which its indications appear increased when an actual measurement is made. For multiple measurement of time, the simple chronograph is completed by a corresponding number of auxiliary chronometers.

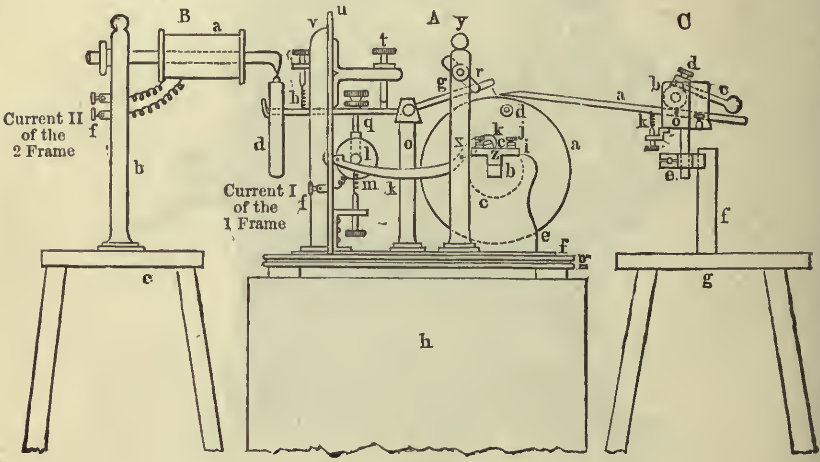


Fig. 292.

The chief parts of the single chronograph are the recorder (A) and the indicator (B) (Fig. 292). The registrations on the former are made on a drum (a), round the circumference of which a detachable sheet of blackened paper is fixed. A simultaneous rotating and longitudinal motion is given to the drum, the first by means of a clock spring, and the second by means of a guide screw (b). By means of arrangements, which need not be described in detail, the drum can be given either a very quick movement (lasting barely two seconds) or a gradually decreasing one, whereby the time of movement is prolonged. A two-armed lever (d) turning round (x) engages by means of a tooth (z) in the groove (k) on the boss (c) of the drum, whereby the latter is stopped when the spring is tight. The tooth only engages in the groove when the electro-magnet (l) attracts the end of the lever opposite to it. As soon as the current exciting the magnet is interrupted, the spring (f) draws the lever downwards, releases the tooth, and sets the drum rotating.

Above the drum is a tuning-fork (g), to one arm of which a bristle (i) is attached, which draws a time curve on the blackened surface of the paper. The tuning-fork can be turned round on the column (o), and is fixed by its stem on to a brass bar (s), fitted with a screw (q), which rests on the electro-magnet (l). In this position the tuning-fork is lifted away from the drum and remains held back by a tongue (r) until the electro-magnet is excited. As soon as the current is interrupted, the spring (h) lifts the brass arm, the tuning-fork is released from the tongue and moves, vibrating the while, towards the drum until the bristle touches the blackened surface. The upward movement of the brass arm is limited by the screw (t).

The plate (u) which is stiffened by the column (v), carries the electro-magnet and the other parts required for fixing the screws, etc. The indicator (B) consists of the electro-magnet (a) and the armature (d), and is carried by the column (b). The armature is rectangular, and is so hung to the magnet that as soon as the latter is excited, the end of the tuning-fork lever passes through the two long vertical sides of the armature.

In using this apparatus, the electric current (I) of the first frame is passed through the coil of the electro-magnet (l), and the current (II) of the second frame through the coil of the magnet (a). The spiral spring moving the drum is wound up, and the tuning-fork pressed into the tongue. Then the shot is fired. On interrupting the current (I) the action of the electro-magnet (l) ceases. The spring (f) draws the tooth (z) out of the groove, and the drum begins to rotate. At the same time the spring (h) lifts the back lever of the tuning-fork, the registering bristle comes in contact with the registering surface, and the time curve is drawn. On interrupting the current (II), the armature (d) falls on to the end of the tuning-fork lever, lifts off the registering bristle of the tuning-fork, and the time curve ceases. The time that has elapsed between the successive interruptions of the two currents can be determined from the number of vibrations of the tuning-fork registered on the drum.

If it is intended to measure the velocity at several points of the trajectory, or the times of flight for several parts of it, a corresponding number of secondary chronometers are inserted. Such a secondary chronometer (C) consists firstly of a lever (a) revolving about the centre (o) with a registering point, which presses lightly against the drum by the spring (k); secondly, there is an electro-magnet (b) and a lever (c), the upper end of which is held by the electro-magnet when it is excited, whilst on interrupting the current the fall of the lower lever arm is limited by the screw (d). In using the apparatus, the current (I) is passed through the first frame and the electro-magnet of the recorder, the current (II) through the second frame and the first secondary chronometer, and so on until the last current flows through the last frame and the indicator. As the projectile passes through the first frame, the drum begins to rotate, the tuning-fork to draw the time curve, and the lever of each secondary chronometer to draw its curve. As the current passing through the second frame is interrupted, the weight of the first auxiliary chronometer falls on to the registering lever (a), and thereby the line drawn by this chronometer is broken. The same takes place in the second chronometer on interrupting the first frame, and so on. At the last frame, the armature of the indicator falls down and lifts off the tuning-fork, so that the time curve is ended.

On simultaneously interrupting all the chronometers, lines are obtained on the drum which indicate the retardations due to the apparatus themselves. For this purpose a disjunctor is used, which is so arranged that the pressing on a spring is sufficient to break all the currents simultaneously.

II. PRESSURE GAUGES.

The phenomena occurring during the combustion of an explosive which is enclosed on all sides are for the most part not open to direct observation. Nevertheless, from various experiments which have been made both in bore-hole shots and with guns, it was possible to deduce that the total pressure produced by the sudden development of gases is always the same for explosives of equal strength, if they are exploded in perfectly confined spaces of equal volume, the ideal form being a spherical one. The degree of these pressures must therefore be subject to alteration from various circumstances which occur in using explosives in practice. Above all, the medium surrounding the explosive cools the gases, and thereby lowers their pressure. To a still greater extent the pressure is altered in guns and bore-holes in which the charge, at the moment of explosion, may be arranged as concentrically as possible, but where through the continued movement of the projectile along the barrel, and through the displacement of the rock in a bore-hole shot, a gradually increasing space has to be taken into account, the consequence of which is also a reduction of pressure. It follows from this that the pressure of the gases ought to reach their maximum at the moment of explosion, but in rigid media and with slowly burning charges, a series of undulatory movements will be produced in the direction of the longer dimension of the explosion chamber, which can give rise to local concentrations of pressure. Therefore, when determining the gas pressure given by an explosive, it is frequently necessary to be able to measure it on various points in the explosion chamber, and for this purpose various and sometimes very ingenious methods have been used.

According to a definition of Sébert, the pressure exercised by the gases of an explosive on a unit of surface is an actual force which can be expressed in kilogrammes, and which differs from the forces usually considered only in its magnitude and the extraordinary rapidity of its variation.

There are two methods of measuring forces.

(1) The static method, in which the force to be measured is equilibriated with a variable force, the magnitude of which is known. The typical apparatus for this method is the balance.

(2) The dynamic method, in which the force is allowed to act freely on a heavy body, and its value determined from the motion the force imparts to that heavy body.

A. THE STATIC METHOD.

1. RUMFORD'S APPARATUS.

In 1892 Rumford made experiments at the Munich arsenal with an apparatus which consisted essentially of a wrought-iron mortar with a smaller continuation at the bottom forming the firing vent. On the muzzle of the mortar weights of different sizes were put, and the charge was ignited by an incandescent iron ball which was put under the mortar by means of a long bar, and had a perforation which fitted over the firing vent of the mortar, and so closed it. If the gas pressure were small, the weights were not lifted, the gases remained enclosed, and no report was heard; but if the gas pressure were greater, the weights were lifted, and the gases could escape. The magnitude of the gas pressure could be approximately estimated from several experiments. Rumford calculated the pressure produced by black powder burning in its own volume to at least 55,004 atmospheres, an estimate which is decidedly too high.

2. Rodman's Cutter Gauge.

This apparatus, which was first suggested by Major Rodman of the U.S. army, in 1857, still enjoys favour at the present day, side by side with the crusher gauge invented by Captain (now Sir Andrew) Noble, which is described further on. The reason for this is that it can be applied in a very simple way, and gives perfectly comparable and fairly trustworthy results. It is illustrated in Fig. 293. The apparatus consists of a plug (a) screwed on the outside, through which a hole (b) of about $\frac{11}{16}$ inch diameter is bored, in which a piston (B) slides. The face of B, which is of steel, is formed into a cutter (C), and carefully tempered to an exact degree of hardness. Opposite to it there is a copper plate (D), which can be set up to the cutter by means of an adjusting screw (E). The edge of the cutter is not parallel to the copper plate, but pyramidal.

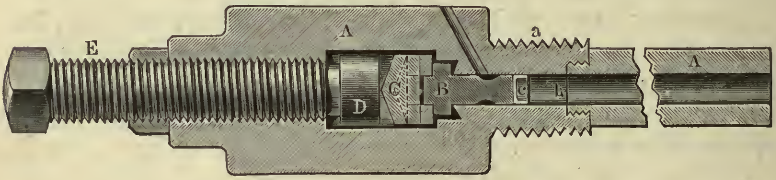


Fig. 293.

In order to use the apparatus, a hole of the same diameter as b is drilled through the wall of the gun, and its outer end enlarged and screwed to take the thread on the plug. The latter is then screwed in, a copper plate (D) inserted and set to just touch the steel cutter (C) by means of the screw (E). When the gun is fired, the gases drive the piston (B) forward and press the steel cutter (C) more or less deeply into the copper plate (D). On account of the construction of the cutter, a more or less long cut is made in the copper plate, and the length of the cut is a measure of the pressure which has been exerted. This length

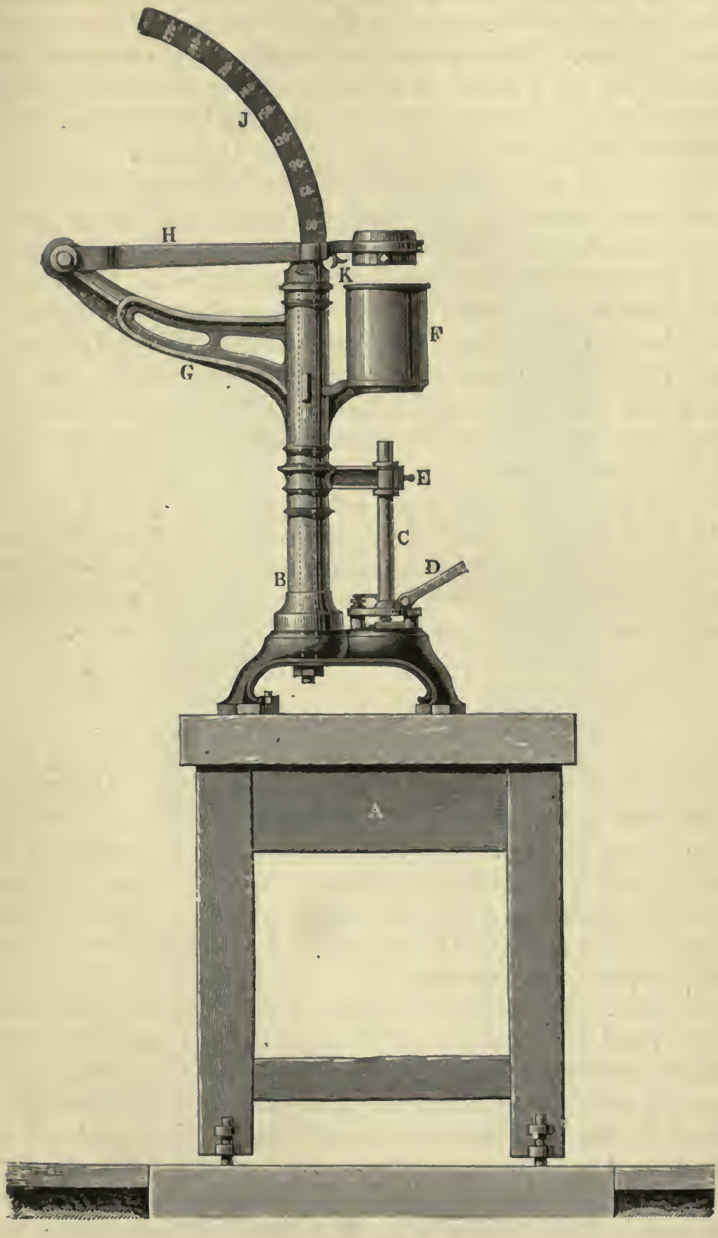


Fig. 294.

of cut is compared with a series of cuts made by a cutter of a similar form and hardness in a copper plate by means of a hydraulic press. Rodman published such a table wherein the length of cut for a shape of cutter exactly indicated by him is calculated for pressures between 45 and 2200 kilogrammes. The smallest length of cut is $\frac{1}{8}$ inch, the longest $1\frac{1}{8}$ inches.

Sébert has raised objections to this apparatus, which are chiefly based upon the fact that the steel cutter is gradually displaced by the gas pressure, although by a few millimetres only, which has some influence upon the results obtained. Much more would have to be allowed for the time in which the explosion takes place. This depends principally on the size and shape of grain of the powder, and also upon its inflammability. At any rate, it will have considerable influence upon the cut, because the quicker the cutter enters the copper plate, the more resistance will be offered, inasmuch as the particles of the copper plate struck by the cutter cannot get out of the way quickly enough, whilst they will behave differently if, as with the hydraulic press, the cutter be only pressed in at a much slower and more gradual rate. Rodman's apparatus will, therefore, not register the highest pressure, but only the mean of the sum of all the pressures.

3. Uchatius' Powder Gauge.

This gauge, which is based on the same principle as Rodman's, is illustrated in Fig. 294. A table (A) fixed to a stone foundation carries a firm base (B). This has an arm (E) and a clamp (D), by means of which a gun-barrel is put in vertically. On the other part of the apparatus, a lever (H) hinges on a cast-on arm (G), and swings along an arc (J). On the free end of the lever is the so-called receptor (r) and the pawl (K). On another arm a diaphragm (F) is placed. The receptor is made of cast steel and perforated conically, and there is also a hollow cover screwed on. The barrel itself has a cutter fixed at the bottom, similar to that in Rodman's apparatus, which rests directly on a copper plate. The barrel is charged in the ordinary manner with black powder and a lead projectile. After firing, the ball goes through the diaphragm into the conical perforation of the receptor, and draws the lever up to a point corresponding to the force of the shock, where it is held fast by the pawl. At the same time the cutter on the barrel makes a cut in the copper plate, as in Rodman's gauge. The edge of the Uchatius cutter is in the form of a circular arc, the advantage of which is that the length increases according to the pressure.

Now-a-days the apparatus is only used in Austria for classifying various kinds of powder.

4. Crusher Gauges.

The English Committee on Explosives sitting in 1868 adopted a new pressure gauge proposed by Sir Andrew Noble (then Captain Noble), which he called a "crusher gauge," and which he had already been experimenting with since 1860.

Fig. 295.

Fig. 296.

Fig. 297.

Fig. 298.

Noble's crusher gauge, Figs. 295 and 296, consists of a hollow steel screw closed at its lower end with a screw cap, so that small cylinders of copper (B) can be put into the chambers (C, D, E, F) as required. The upper surface of these cylinders rests on an anvil (A), whilst the piston (C′) acts on the lower surface. The piston is pressed against the cylinder by the spring (i), but is otherwise movable. A small watch spring (Fig. 297) serves to centre the cylinder in the chamber. The head of the piston is milled as shown in Fig. 298, and so is the anvil. Four long perforations at A′ and B′ are connected to a wide channel which goes axially through the upper part of the screw. The lower end of the piston (C′) is closed air-tight.

On using the apparatus it is screwed into the wall of the gun-barrel, and the cylinder (B) put in. As soon as the shot is fired, the gases press upon the lower surface of the piston, and crush the cylinder against the anvil, the degree of crushing giving a measure of the pressure of the gases. With the old 8-inch rifled muzzle-loader a base surface of the copper cylinder of 0·5 square inch, and a pressure surface of the piston of $\frac{1}{6}$ square inch, proved to be most advantageous. In order to estimate the pressure produced, similar copper cylinders are submitted to a series of experiments in which they are compressed by means of a press, and the results tabulated.

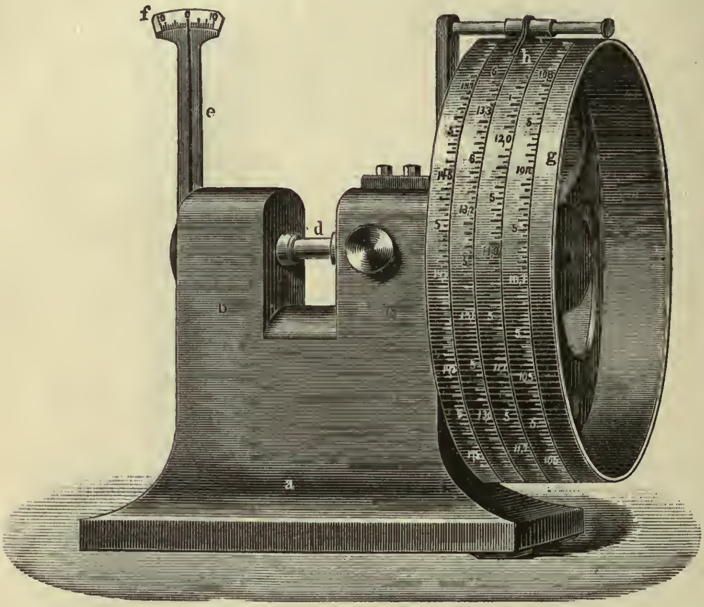


Fig. 299.

Comparative experiments made by the English Committee between Noble's and Rodman's gauges proved the superiority of Noble's apparatus. According to the opinion of the Committee, the reason for this is chiefly that on account of the size and shape of Rodman's copper plate and cutter, it is not possible to place them into the barrel itself; consequently the powder gases have to travel a not inconsiderable space between the bore of the gun and the apparatus, and the pressure must, therefore, reach a fairly high value before they can reach the piston, and drive the cutter into the copper plate. This excess of force is transmitted into the pressure gauge, whereby the latter indicates a higher pressure than is exercised in the bore of the gun.

The firm of A. and R. Hahn of Cassel make a micrometer for measuring the copper cylinders after they have been crushed. It consists of a cast-iron stand (a) (Fig. 299) with two projecting standards (b and c) at the top, one of which carries a cylindrical bar forming a measuring-bar. This passes right through the left-hand standard, bearing at the back against the long index-pointer (e), and is pushed forward by a spiral spring. The other bearing carries a micrometer screw, to the outer end of which is attached a large measuring-drum (g). The latter has a spiral groove turned in it, the edge of which is graduated, and in which the index (h) engages. The copper cylinder (b) is placed between the end of the left-hand measuring-bar and the inside end of the micrometer screw. The drum (g) is then turned until the index (e) stands at zero. As g is turned, the index (h) follows the groove on the drum, and enables the length of the copper cylinder to be read off exactly to 0·01 of a millimetre.

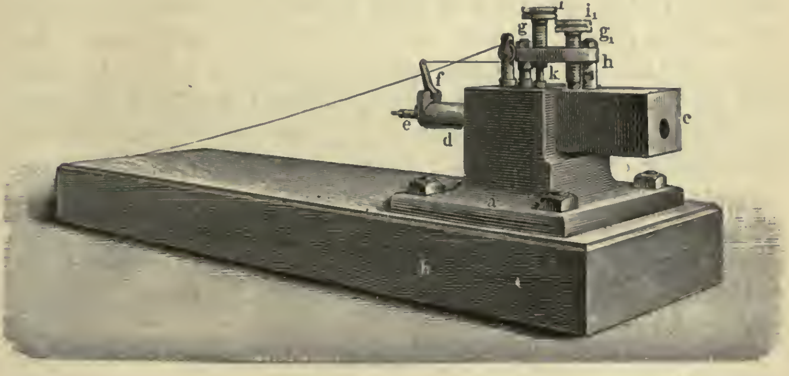


FIG. 300.

For small arms similar crusher gauges are used. For this purpose a rifle barrel is arranged in a wooden frame, and into it a suitably modified crusher gauge is screwed. For sporting guns it is frequently important to determine the gas pressure both near the cap and near the shot charge. This is done alternately at both places.

A suitable apparatus for this purpose, made by Cogswell and Harrison of London, is illustrated in Fig. 300. In it two lead cylinders, each of 0·324 inch diameter, and 0·5 inch high, are crushed. The apparatus is fixed to a wooden block (b), and consists of a carriage (a) with a T-shaped recess on the top, into which fits a rectangular steel block (c) with a firing chamber bored through it. A cylinder (d) carrying the firing bolt (e) screws into the base of c. The firing bolt is held back by a spring and trigger (f), and can be released by pulling the cord attached to it, which passes over a small grooved pulley on the frame. Two studs (g and g_1) on top of c carry a plate (h) through which the two screws (i and i_1), with fine threads and milled heads and accurately faced at the ends, pass. Two holes are drilled through the block into the firing chamber, one at 1 inch and the other at $2\frac{1}{2}$ inches from the breech end. These holes are 0·1595 inch in diameter or exactly $\frac{1}{5}$ inch in cross-sectional area. The screws (i and i_1) are concentric with these holes. A carefully-fitted plunger is provided for each of the holes, with a head 0·5 inch in diameter, on which the lead-crushing cylinder (k) is placed and held in position by lowering the corresponding screw until it is just held.

On using the apparatus, a hole of the diameter of the piston is drilled through the cartridge case in such a position that it will come opposite one of the plungers, according to whether it is intended to measure the pressure at the cap end or at the shot end; the cartridge is then put into the firing chamber, and the lead cylinder placed in position, whilst the piston which is not used is fixed by tightly screwing down its screw. The whole apparatus is then pushed forward into a hole in a block of brickwork, which partly takes the steel block, so that shot flying about cannot hurt the operator. After firing the cylinder is taken out, exactly measured by means of a micrometric gauge, and the value of the gas pressure obtained from a table calculated from previous experiments.

The German Metal Cartridge Factory of Carlsruhe have patented a pressure gauge for rifles and guns (German patent, No. 73015 of March 21, 1893), the most recent form of which is illustrated in Fig. 301. Here a copper cylinder is compressed, but as this is effected by the pressure of the gases coming from the full opening of the cartridge in the moment of their production, a more reliable measurement is possible than from a cartridge with simply a side hole drilled in it. The apparatus consists of a gun-barrel (a′), into which a cartridge case without bottom (l) is put in the ordinary way with a full charge and a projectile (l′). Round the end of the case a ring (m) is attached by which its position is determined when screwed up tight against the cone. The end of the latter has a V-shaped ridge on it which is forced into a corresponding groove in m. Both the cartridge case and the case are enclosed in a coned block (k), one end of which abuts against the barrel (a′) and the other against the flange (i), which in turn is supported by an internal flange on the block (a). The fixing of these parts together is done by screwing in the barrel (a′). h is the pressure piston which moves parallel to the centre line of the barrel, and is made in three parts and ground into the case (i), which has the same bore as the cartridge case (l). The gas-tight fit is assisted by a hard rubber ring (n) placed between the two front parts. In front of the pressure piston there is a detachable part (o) which enters the cartridge case to the place where the base of an ordinary service cartridge would be. The part (o) contains the firing anvil and holes, and a piston to take up the cap, as is shown in the drawing. There is a perforation in the pressure piston (h) through which passes the firing bolt (c_1). A copper cylinder, also perforated, is placed against the pressure piston and fixed in position by means of a screw (b). A tube (E) sliding gas-tight inside this screw carries the firing bolt (c_1), which is actuated by a spiral spring and screwed to a withdrawing bolt (c). The firing-pin is fixed in position by means of a screw (d). Below the withdrawing bolt is a trigger (f_1) which rests against the body of the firing-pin. When f is pulled, f_1 is

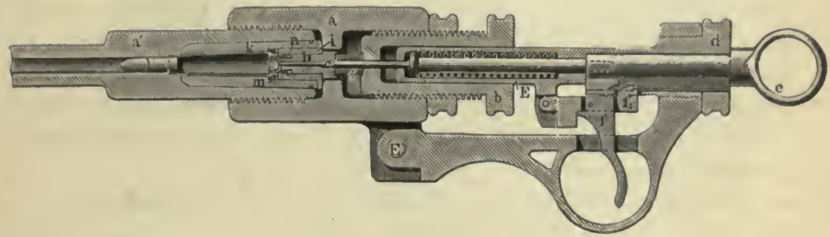


Fig. 301.

pressed downwards, and the pin flies forward. The apparatus can be put apart by removing the pin (E_1). When, by the flying forward of the firing-pin, the cap, and thereby the charge, has been fired, the gas pressure at the moment of its production can only move the pressing piston, which it drives back and crushes the cylinder (g), and from the amount by which it is shortened, the pressure can be calculated in the usual way.

5. Marcel Deprez's Manometric Balances.

As mentioned previously, the gas pressure given by an explosive, when it is fired in a confined space, varies considerably in extraordinarily short spaces of time. For these reasons ordinary pressure gauges cannot be used for measuring such pressures. To overcome this difficulty, Marcel Deprez has designed a gauge in which a pressure of a pre-determined value acts against the gas pressure on the piston, and he determined the moment at which the two pressures balance each other. By repeating the experiment and regulating the counter pressure according to the gas pressure obtained, the exact value of the gas pressure can be obtained with reasonable accuracy.

Deprez has constructed two kinds of such manometric balances. One with only a single piston, in which the exact measurement can only be obtained by repeated experiments, and the other with a number of pistons, which allows the maximum gas pressure to be obtained by one experiment, and which, at the same time, gives the exact sequence between the single pressures by means of a Schultz's chronograph.

The manometric balance with one piston (Fig. 302) consists of a case screwed on to the barrel of the gun in which a differential piston works. Its smaller surface, which has an area of 0·5 square centimetre, is exposed directly to the gas pressure; its larger surface, which is 400 times as large, is situated in a chamber formed by screwing a cap over it. Into this chamber air is compressed by means of an air-pump, and the pressure indicated by a gauge put on to the chamber. The surface of the large piston rests on a base plate by means of a rim, and in this position holds fast a slide. If equilibrium between the total pressures on the two pistons be established, the rail ceases to be held fast, and is withdrawn by an india-rubber spring. When this occurs, it is known that the pressure in the bore of the gun exceeds the corresponding pressure on the larger piston, and consequently the experiments have to be continued with constantly increasing pressures in the chamber until equilibrium is obtained. In order to avoid this rather cumbersome process, which is similar to weighing on a balance, Deprez constructed a multiple manometric balance which is illustrated in Fig. 303. This also has a differential piston, the larger end of which acts in a chamber filled with mercury. Into this the smaller ends of 10 secondary differential pistons enter, whose sizes vary in the proportions of 1, 2, 3, etc., up

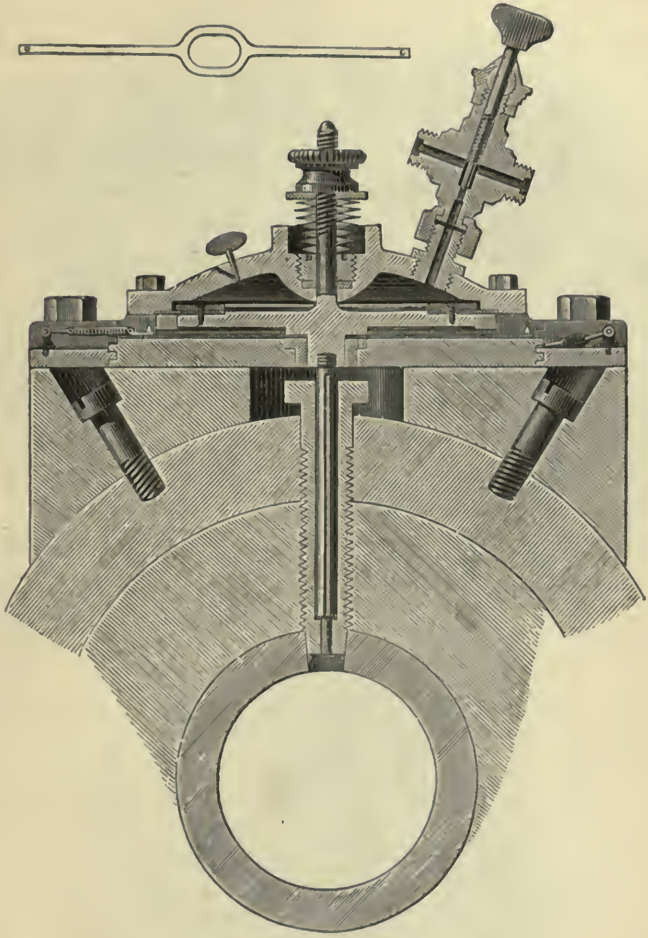


Fig. 302.

to 10. The other ends of these secondary pistons act in a common annular chamber filled with compressed air of known pressure, as in the case of the single manometric balance. Each secondary piston is fitted with a cylindrical sliding contact piece. These are arranged and coupled up, so that as each piston is forced outwards, the corresponding circuit is first made and then broken again. If,

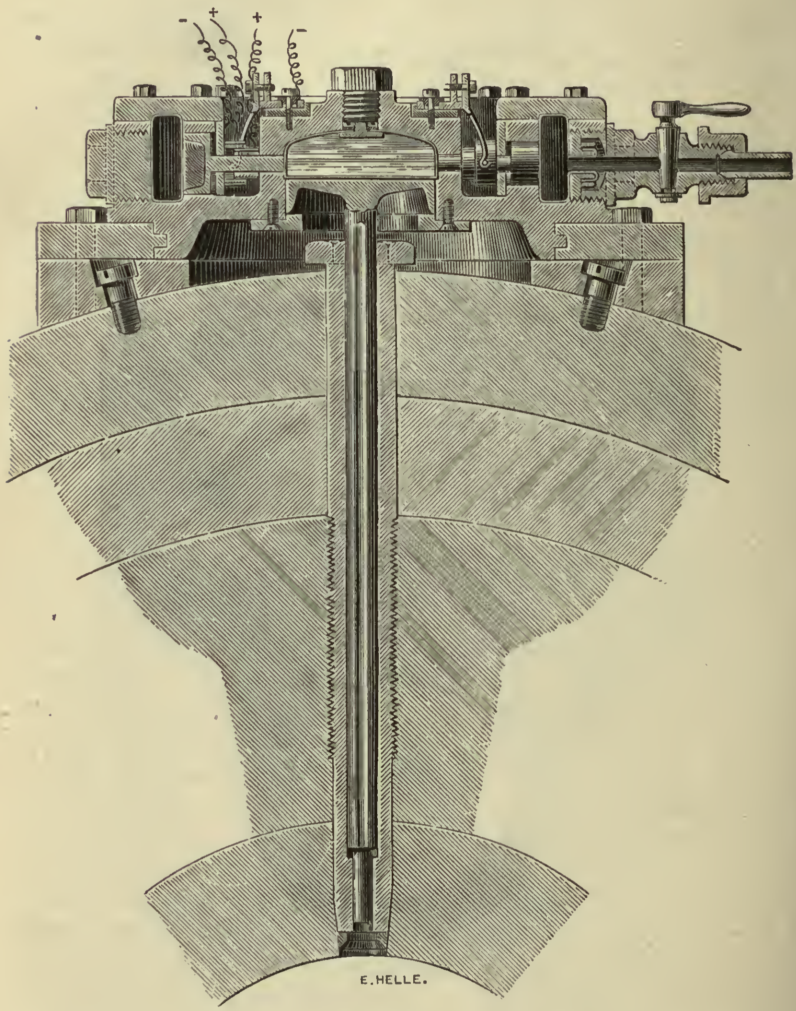


Fig. 303.

therefore, on the equilibrium being exceeded, a secondary differential piston moves forward, the galvanic current is first closed, and the registering apparatus of the chronograph connected with it is brought into action. After the explosion the piston goes back, the current is interrupted, and the registering apparatus put out of action.

Thus, by combining Deprez's multiple balance with Schultz's chronograph, as improved by Deprez, both the magnitude of pressure, and the interval of time in which it reaches a certain value, can be determined exactly.

Deprez has also constructed a manometrical balance in which the pressure is registered mechanically instead of electrically. It was exhibited at the Electrical Exhibition in Paris in 1881. Particulars of the results obtained with it have not been made known, but a description of the principles underlying it can be found in Col. Hess's report on that exhibition, in the 'Mittheilungen des k. k. technischen und administrativen Militär-Comité's' of 1881.

B. The Dynamic Method.

The dynamic method for the study of the ballistic phenomena was first used in 1760 by Chevalier d'Arcy, when he employed the ballistic pendulum for determining the velocities obtained with rifle barrels of different lengths.

In 1845 Col. Cavalli made a series of experiments by drilling holes in different places of a gun, and screwing into these holes rifle barrels throwing wrought-iron balls. For each experiment only one hole was left open, and opposite to the rifle barrel screwed therein a ballistic pendulum was placed. A committee which sat in 1851 under the chairmanship of General Neumann made a series of experiments by Cavalli's method. All these experiments, however, gave very irregular results. More detailed experiments were made in 1872 by the English Committee on Explosives by means of Noble's chronograph, as has already been mentioned, when they drilled holes in a gun in various positions along its bore, and put into each a Noble's chronograph cylinder. Noble and Abel continued these experiments in 1874.

Of the various other apparatus which have since been experimented upon, the accélérometers and accélérographs proposed by Marcel Deprez and Sébert especially deserve mention.

With the former a piston placed in a hole bored in a gun barrel is allowed to move along a certain path, and the velocity it attains registered by a special mechanism. With Sébert's apparatus a curve is drawn by a pencil on a special plate fixed on the piston. On the other hand, Sébert constructed apparatus, which he called "velocimeters," which, instead of measuring the pressure of the gases directly, allowed them to be estimated from the recoil of the gun, and thus avoided the necessity of drilling the gun. In these apparatus a flexible steel tape, with one surface blackened, is fixed on the gun, and a line curve drawn on the blackened surface by a Deprez's tuning-fork as the gun moves back under the influence of the shot, and thus the time required for recoil can be calculated.

Details of these apparatus, which are only important to military specialists, are to be found in the pamphlet published by Sébert.[1]

III. POWER GAUGES.

In general many of the gauges described in the preceding section can be converted into power gauges—that is to say, into apparatus which measure directly the work done by an explosive. Such power gauges are chiefly of use in civil engineering, for comparing the effects of different blasting materials.

It is unnecessary to describe all the many apparatus constructed for this purpose, and only the more important types which have been used practically will be mentioned.

A. The Timber Test.

In various armies the force of a normal blasting charge is tested by the breaking effect it has on a piece of soft timber. As a rule it is so selected that the standard charge of the standard explosive just breaks it. This simply shows whether the explosive being tested exerts a greater or a less force than the standard blasting material. The timber is supported at each end only at some height from the floor level. For ecrasite the present standard in Austria is a piece of timber 22 by 30 centimetres ($8\frac{11}{16}$ inches by $11\frac{13}{16}$ inches) thick, and 1·5 metres (4 feet 11 inches) long, placed on edge at a height of 80 centimetres ($31\frac{1}{2}$ inches) above the ground, with about 3 feet 3 inches clear between the supports.

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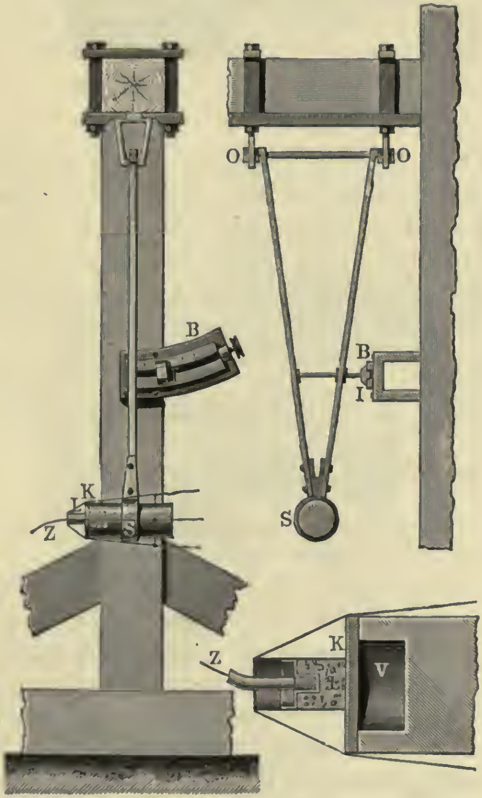
[1] Notices sur les nouveaux appareils ballistiques employés par le service de l'artillerie et de la marine, par H. Sébert. Paris, L. Baudoin and Co. 1881.

The sample of 1 kilogramme (2·2 lbs) of ecrasite is placed across the centre of the free part in such a way that about 1 centimetre of the wood should remain uncovered, and when fired by a detonator of 2 grammes charge, must break the timber with certainty.

B. Hess's Pendulum Power Gauge.

In 1873 Colonel Philipp Hess constructed the apparatus, which is based on the principle of the ballistic pendulum, and illustrated in front elevation in Fig. 304, and in side elevation in Fig. 305. It is used by the Austrian Technical and Administrative Military Committee for determining the admission of explosives as blasting materials. It consists of a pendulum hung on two knife edges (o),



Figs. 304, 305, 306.

with a cylindrical weight (S). There is a recess (V) in the weight (Fig. 306 on a larger scale), over which is placed a plate (K) made from rolled copper 5·5 millimetres thick. On to this is put the explosive (L), which is made up in a tin box and provided with a detonator and fuse (Z), and fixed in suitable manner by cords. The charges used are always 17 grammes in weight, and are fired by detonators with 1 gramme charges. When the explosion takes place, the pendulum swings to one side along the graduated arc (B), and pushes forward an index (I) by which the length of swing is recorded; at the same time the explosion indents or penetrates the copper plate. The indenture can be measured by introducing mercury, or by taking a cast. It was shown that it was difficult to get copper plates of uniform quality, and consequently the apparatus was later on modified by substituting for the large recess a quite small one in which were placed one or two lead cylinders, as illustrated in Figs. 307 and 308. A steel plate of 3·3 millimetres thickness was put between the lead cylinders and the charge, in order to prevent the former from being deformed too much. The amount the lead cylinders were shortened was then measured by means of a caliper gauge.

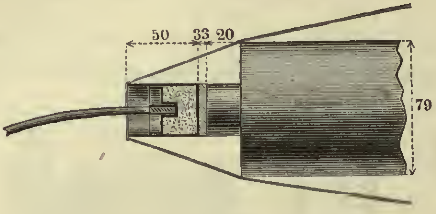


Fig. 307.

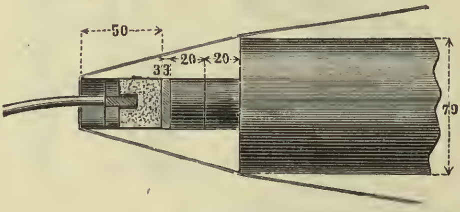


Fig. 308.

C. The Crushing Test.

(1) Austrian Apparatus.

In Austria-Hungary a method is used for determining the strength of explosives, which, in a similar manner to the pendulum power gauge, is based on the deformation of two lead cylinders by charges detonated in the open air above them. This method and those dealt with previously can only be used for indirectly exploding materials such as dynamite, gun-cotton, etc., free-lying untamped charges of which exercise on their base an effect corresponding to their strength, and also especially to their rapidity of explosion. This Austrian apparatus is illustrated in Fig. 309. It consists of a cast-iron base plate (P), into which four pins (D) are screwed, and which has a small recess at the centre. Into the latter two lead cylinders (Z), each 20 millimetres high and 31 millimetres in diameter, are placed, one on the top of the other. On to them a steel plate (S) 3·3 millimetres thick is placed, and then the charge, which is made up in a tin box and fixed by wires wound round the pins (D). After the explosion, the deformation of the lead cylinders is measured by means of a sliding gauge. The reason for using two lead cylinders is to give a softer base to the upper one which has to take up the blasting effect proper, so that it shall be protected from being crushed too much by the unyielding cast-iron base plate. As with all tests where lead cylinders are used as indicators, it is essential to employ cylinders

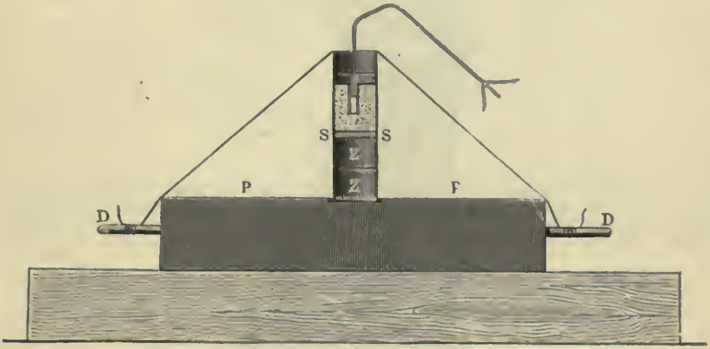


Fig. 309.

of perfectly uniform hardness. Cylinders cut from a drawn bar of lead are the best.

A cutter apparatus has been used in Austria in which by means of variable weights on a two-armed lever, an edged tool cuts a curve into a lead cylinder, from the length of which a conclusion is made as to the hardness of the cylinder.

For testing ecrasite for the Austrian engineer troops, it was necessary to modify this crusher slightly, because this explosive requires for complete detonation a very strong detonator (one with 2 grammes charge), and therefore the detonator would have a preponderating influence upon an inferior charge. For this reason 50 grammes of ecrasite are put in a cylinder of 40 millimetres outer diameter and 31 millimetres high outside, made of sheet-tin 0·5 millimetre thick. The lead cylinders are 30 millimetres high and have a diameter of 40 millimetres. Two round steel

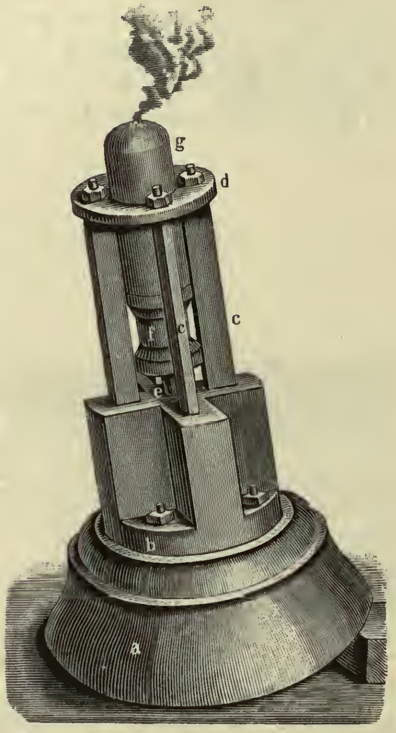


Fig. 310.

discs, each 40 millimetres in diameter and 4½ millimetres thick, are put between the upper lead cylinder and the charge. The shortening of the lower cylinder is, as a rule, between 24 and 27 millimetres, and that of the upper one 11 to 11·1 millimetres. The upper lead cylinder is always deformed to a mushroom shape.

(2) Quinan's Crusher Gauge.

W. R. Quinan, of San Francisco, designed a crusher gauge for measuring the force of explosives, which is much favoured in America. It is illustrated in Fig. 310, and consists of a heavy wooden block (a), on to which a cross-shaped cast-iron base plate (b) is fixed by means of screws. Four wrought-iron guide bars (c) are fitted to this, the inner edges of which lie round a 4-inch circle. They are held together at the top by a ring (d). A steel disc is let into the base plate, on to which a lead cylinder (e) 1 inch in diameter and 1 inch long is placed.

Fig. 311.

On to this is put a piston (f), measuring 4 inches at its largest diameter, and 5 inches high. This piston is turned away in the centre, so as to make it as light as possible, and moves freely between the guide bars. In the upper face of this piston there is a parabolic cavity in which the charge is put, and on to which a hardened steel projectile (g) 4 inches in diameter, 10 inches long, and weighing 34½ lbs. is put. The projectile is perforated axially to allow the fuse and the detonator to be inserted. When the charge is fired, the projectile is thrown up whilst the piston simultaneously crushes the lead cylinder. The latter is then carefully measured by means of a sliding gauge, and the corresponding pressure in foot-pounds is taken from a table calculated graphically by Mr. Quinan from certain theoretical considerations and practical experiments.

With this apparatus the explosive is more perfectly confined than in the Austrian apparatus, and the total force is found approximately, although part of the gases can escape through the firing vent in the projectile.

(3) KOSTERSITZ AND HESS'S POWER GAUGE.

A charge of 17 grammes of the explosive to be tested is put in a tin box (a), and this into a wrought-iron gas-tube $1\frac{1}{4}$ inches in bore and 1 foot 8 inches long (Fig. 311). Below this comes a steel plate (b) $1\frac{1}{4}$ inches thick, then a cylinder (c) of drawn lead, then a cylindrical punch (d) which is slightly conical towards the ends, and which is made from hardened steel, and finally a second lead cylinder (c). It is best to put the whole lot in a paper case and introduce them simultaneously to about the middle of the gas tube. The room (f) left free on each side is then tamped with sand and a clay stopper (g). The charge is, of course, provided with a detonator and fuse (s). According to Kostersitz and Hess, the tube should be broken in the middle or bulged to a cup shape and split, when the charge explodes. The steel punch should form regular conical hollows in the lead cylinders, and from their size an idea can be obtained of the work done by the explosive in a confined space. A long time ago the author made such an apparatus, following exactly the directions given by Kostersitz and Hess, but after careful experiments, came to the conclusion that no uniform results could be obtained with it. The steel punch frequently

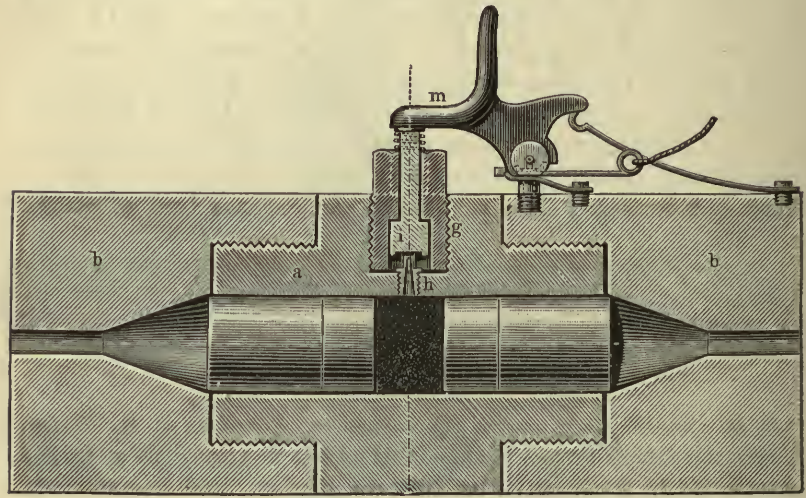


FIG. 312.—Three-8ths full size.

gets jammed, and the indentures made into the cylinders are of very different size when one and the same kind of dynamite is used for the experiments, and even with parts of one and the same cartridge. Apart from this, the preparation of the cartridge is very cumbersome, and the only safe conclusion which can sometimes be arrived at, is from the bursting of the gas-pipe, always supposing that uniform pipes, free from faults, can be obtained.

(4) GENERAL ABBOT'S RING APPARATUS.

General Abbot, of the U.S. army, used a wrought-iron ring of 4 feet diameter for measuring the force of explosives.

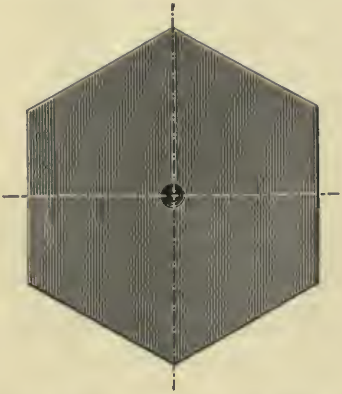


FIG. 313.—Three-8ths full size.

To this were fixed six crusher gauges with lead cylinders, arranged at 60° from each other. The charge was suspended by means of wires in the centre of the ring, and this was kept floating by a buoy in deep water. Abbot's experiments were made chiefly for the purpose of judging the effects of explosives for submarine mines, and in this respect they gave very valuable information. They could not give a comparative scale for judging the force of explosives, because, for one reason amongst others, a layer of water was between the charge in the crusher gauges, which led to the curious result that the strength of pure nitro-glycerin was only 81 per cent. of that of dynamite containing only 75 per cent. of nitro-glycerin.

(5) GUTTMANN'S POWER GAUGE.

Whilst in the preceding apparatus either the effect of the rapid explosion of the blasting material, or approximately the whole of the force produced in a confined space, was exerted as a pressure upon a yielding base, the author endeavoured to design an apparatus in which the charge of the explosive is detonated in a space closed on all sides, and has to do work which can be measured later on, so that the conditions under which it is exploded are as far as possible similar to those obtaining in practical work.

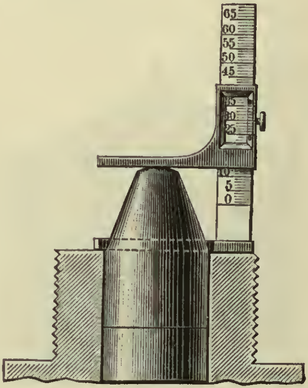


FIG. 314.—Three-8ths full size.

Although he constructed his apparatus in the first place for the measurement of directly exploding materials only (black powder, etc.), there is no doubt that by making it of suitably stronger construction it could also be used for indirectly exploding materials. This he never did, however, because Trauzl's lead test, which will be dealt with later on, gives approximately just as reliable results for indirectly exploding materials, and they can be obtained more readily.

Guttmann's apparatus (Figs. 312, 313, and 314) consists of a centre piece (a) and two end pieces (b, b), all three being made of hardened Bessemer steel. The end pieces fit over the screwed projections on (a). The centre piece has an axial cylindrical hole 35 millimetres (1·38 inches) in diameter bored through it. The hole in the end pieces is of the same diameter on the inside, but is tapered for a length of 35 millimetres, when it becomes cylindrical, and is 10 millimetres (0·39 inch) in diameter. At the top and on the centre line of (a) is the firing plug (g); this is screwed in to a depth of 25 millimetres (0·98 inch); at the lower end of this recess is the firing nipple (h), which takes the detonating cap; the firing plug (g) is perforated with a 6 millimetres (0·24 inch) hole, and is chambered out at its lowest end to fit over the nipple (h); g is fitted with a sliding piece (i) which serves the double purpose of transmitting the blow of the hammer into the cap on (h), and acting as a back pressure valve to prevent the escape of any gas after the explosion in the central chamber has taken place. The apparatus is charged by screwing off one end piece, and inserting first a cylinder of drawn (not cast) lead, 35 millimetres in diameter and 40 millimetres (1·51 inches) long; secondly a steel disc, 35 millimetres in diameter, the thickness of which depends on the gravimetric density of the powder, for the determination of which a special graduated vessel is supplied with the apparatus; thirdly a disc of cardboard ("glazing board") 1 millimetre thick, which fits tightly in the centre chamber of (a); fourthly, exactly 20 grammes of the powder to be tested; fifthly another cardboard disc; sixthly another steel disc, and lastly another lead cylinder. The end piece is then screwed on with a strong spanner, the outside of the apparatus being hexagonal (as shown in end elevation in Fig. 313). An ordinary cap is placed on the nipple (h) and the hammer raised. The latter is released by a cord, and the powder fired by means of the cap. When the explosion takes place, the valve (i) is automatically lifted and prevents the escape of any gases produced by the explosion. Consequently, the explosion takes place in a perfectly closed chamber, and *no report* is heard. The steel discs are forcibly driven outwards by the explosion, and force the lead cylinders into the conical holes in the two end pieces. The apparatus can be immediately opened by screwing off the end pieces, and the height of the lead cones projecting measured by a sliding gauge supplied with the apparatus (Fig. 314). This height is compared with that obtained by experimenting with some standard explosive, usually blasting powder, and gives the comparative strength. Less exact results would be obtained if the cavity produced were measured, as the original volume of the powder would have to be deducted, and this cannot be readily determined with exactness, on account of the screwing-on of the end compressing the powder somewhat.

This apparatus gives very exact results, as is shown by the fact that powders of the same composition, but having differently sized grains, which give the same results in bore-holes, also give the same height of cone in the author's apparatus, but give different results, one from the other, in any other form of apparatus.

Messrs. F. Krantz and Co., black-powder makers of Bautzen, have made extensive comparative experiments with this gauge which fully confirmed what has been said above. Since with some powders the charge was too large for the apparatus, and others had a very rapid effect, the lead cylinders were not only pressed into the conical part of the apparatus, but part of the lead was also forced into the narrow channel, whilst with very rapid powders, ball-shaped lead projectiles were thrown out of the channel. In order to avoid an error on account of these secondary influences, Messrs. Krantz and Co. preferred measuring the cavity produced after the explosion in the apparatus, instead of measuring the conical plugs formed. The results of their experiments are contained in the following table:—

KIND OF POWDER.	LENGTH OF CAVITY PRODUCED IN MILLIMETRES.
Sporting Powders—	
German sporting powder, round grain, made by Krantz and Co.[1]	69
Sporting powder ("Silver Dog" brand), finest grain, made by Krantz and Co.[1]	67
Patent sporting powder (in tin-foil), finest grain, made by Krantz and Co.[2]	65
Extra best sporting powder, finest grain, made by M. Buchholz of Lennep	65
Grain No. 4 (pink paper wrapping), middling grain, made at the Rottweil-Hamburg Factory[2]	65
Canister ("Silver Dog" brand), fine grain, made at the Rheinisch-Westphälische Factory	65
Extra best sporting and target powder, finest grain, made by W. Güttler of Reichenstein	65
Middle grain, Rheinisch-Westphälische Factory	65
American sporting powder, middle grain, made by Laflin and Rand[2]...	65
Linz grain, round grain, made by Novotny of Prague ...	63
Sporting powder, made by the Società Italiana, Milan ...	61
Extra best, in tin-foil, made by Kramer and Buchholz of Rübeland	59
Swiss carbine powder, round grain, made at the Chur Factory	59

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[1] Balls of lead thrown out from both cylinders.

[2] Balls of lead thrown out from one cylinder.

KIND OF POWDER.	LENGTH OF CAVITY PRODUCED IN MILLIMETRES.
Sporting Powders—	
Swiss sporting powder, middle grain, made at the Chur Factory	57
Diana powder, coarse grain, made by Kramer and Buchholz, Rübeland	57
Diana powder, coarse grain, made by Krantz and Co., Bautzen	55
Military Powders—	
English infantry powder, made by Curtis and Harvey, London	61
Russian infantry powder, coarse grain	51
French chassepot powder, coarse grain, from the war of 1870	51
Roumanian infantry powder, coarse grain, made by Krantz and Co.	51
German infantry powder, of 1880, model 1871, strong grain	59
Chinese infantry powder, coarse grain, made at the Rottweil-Hamburg Factory	53

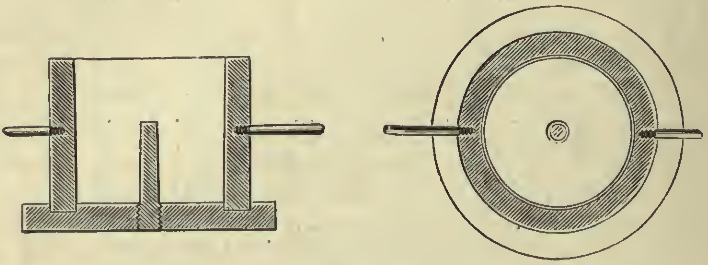
Similar experiments with Guttmann's apparatus were published by Mr. Max Georgi, manager of the mines at Zauckerode.

D. TRAUZL'S LEAD BLOCK TEST.

In 1877 Captain Dr. Beckerhinn had already tried detonating explosives in a perfectly uniform medium in order to obtain an idea both of the force of an explosion, and the shape of the cavity produced by it. For this purpose he compressed regularly piled paper sheets to a packet of 6 inches in height, in such a way that they formed a homogeneous medium, and drilled cavities in it to receive the charge and fuse. Beckerhinn also effected explosions in lead, plastic clay, and hard and soft wood, but obtained less favourable results with these materials.

Later Mr. Isidore Trauzl resuscitated this idea, and by using a suitable form of lead cylinders, and indicating an easy manner of charging, was able to place at the disposal of experimenters the very practical apparatus which is now generally used.

Lead blocks 200 millimetres (about 8 inches) diameter and of the same height are cast in an iron mould, the construction of which is shown in Figs. 315 and 316. Along the axis there is a cylindrical cavity 20 millimetres (about ¾ inch) in diameter and 110 millimetres (about 4⅜ inches) deep. The mould is made slightly conical, so that by lifting the top part the block falls out easily, and by overturning the bottom part it falls from the iron core piece for forming the central hole. Exactly 20 grammes of the explosive are placed in the cavity, a detonator and fuse are inserted, the bore filled with dry sand, and the charge fired. Theoretically, a spherical cavity ought to be produced by the explosion, but as some gases escape through the channel left by the fuse, and as the resistance of the tamping is less than that of the lead, the actual cavity produced is pear-shaped (shown in Fig. 317 by the dotted line). The volume of this cavity, which is measured by filling it with water from a graduated vessel, is a measure of the strength of the explosive. As the hardness of lead varies, and is increased by re-melting, it is advisable to keep in stock a number of lead blocks cast at one melting, and when trying an explosive to make a trial with the standard one it is to be compared with—usually kieselguhr dynamite containing 75 per cent. of nitro-glycerin. If this be done, very reliable results will be obtained.



FIGS. 315, 316.—One-10th full size.

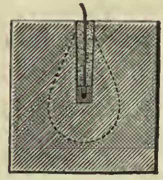


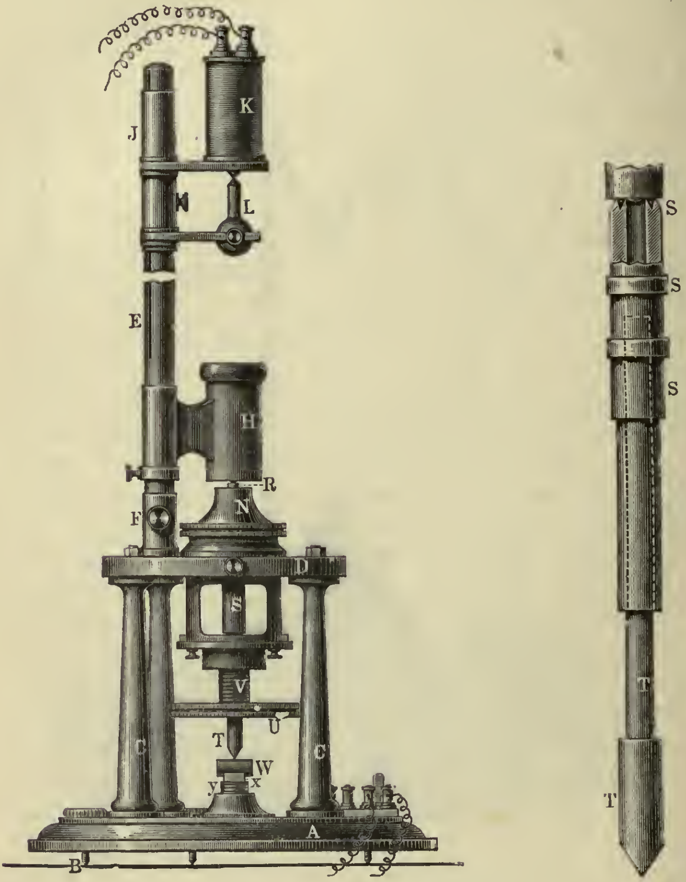
FIG. 317.—One-10th full size.

Although this apparatus and Guttmann's give very valuable assistance in judging explosives, the reader is warned against relying upon them entirely for drawing *absolute* conclusions as to the suitability of various explosives for any specific purpose. The circumstances attending blasting operations in rock vary so much, and the requirements are so various in different places, that a decisive opinion can only be formed by a trial extending over several weeks, made in precisely equal workings, and after careful calculation and consideration of all the advantages and disadvantages.

Fig. 318.

E. Power Gauges for Caps.

One of the oldest and most frequently used tests for measuring the power of caps, consists in exploding them on a lead or iron plate resting on a hollow iron ring, and estimating their strength from the deformation or the penetration of the plate. For larger detonators of between $\frac{1}{2}$ gramme and 1 gramme charge as used for bore-hole shots the plate would have to be of suitably greater thickness, and for such detonators an apparatus suitably modified from the Trauzl lead-block test is preferable. In such an apparatus the detonator is put into the hole of the lead cylinder, tamped with sand, and the cavity produced by the explosion measured by means of water, in the usual manner. Various apparatus for testing caps have been constructed, chiefly on the lines used for testing apparatus for explosives, and having, as a rule, crusher gauges to take up the force exercised.



Figs. 319, 320.

A suitable apparatus, made by Cogswell and Harrison of London, is illustrated in Fig. 318. It consists of a crusher gauge (b) screwed into the bottom of a frame (a). The cartridge case is put on to a piston in the apparatus with its opening downwards, so that the cap is completely enclosed, and the opposite end of the piston crushes a lead cylinder. A weight (c) held back by a spring is put on to the apparatus, and a cylinder (d), which is shown on the side of the illustration, is put over it to prevent splinters flying about. The firing is done by means of a falling weight (e) which slides up and down the bar (f), and which can be fixed at a suitable height. The strength is estimated by reference to tables calculated from an empirical formula.

W. D. Borland designed another apparatus for testing caps, which is a combination of the crusher gauge with the electrical disconnector of the chronograph. A strong base plate (A, Fig. 319), which can be fixed horizontally by means of the adjusting screws (B), carries the columns (C), which are tied together by a plate (D), and support the column (E), which can swing round the hinge (F). A sliding clamp (J) carries the electro-magnet (K) on which the weight (L) is suspended. The tube (H) receives the weight after it has fallen on to the firing arrangement (R). The cartridge case is held in position by a cover (N), whilst the firing arrangement (R) rests on the cap. Below the cross-head a plate is fixed by means of short pillars through which a hollow screw (V) with a milled head (U) passes. By it a tube (S), shown on a larger scale in Fig. 320, is pushed up and rests tightly against the cartridge case. A piston (T) passes through the screw (V) and the tube (S), fitting tightly, and has at its lower end a cutter. The weight is 2 ounces, and is allowed to fall from gradually increasing heights until good detonations are obtained regularly, without misfires. To conduct an experiment, a lead cylinder (W) is put under the cutter, and exactly fixed by means of the screws (X, Y). As soon as the current is interrupted, the weight falls down, strikes the firing arrangement, and the piston is driven into the lead cylinder by the explosion of the cap. Instead of the cutter, a flat steel head may be used, which compresses the lead cylinders instead of making a cut into them. Borland demonstrated with this apparatus that a cap for smokeless sporting powder is sufficiently strong if it is completely detonated by a weight of 2 ounces falling from a height of 12 inches, which makes a cut 0·10 to 0·12 inch deep in a lead cylinder of ½ inch length and ⅜ inch diameter; the piston weighing 4 ounces, and the faces of the cutter making an angle of 60°.

CHAPTER XXIII

STORAGE AND CARRIAGE OF EXPLOSIVES

VERY widely differing regulations for the construction of buildings for storing explosives in, both for military and civil purposes, obtain in different countries. In Austria-Hungary, for instance, the magazines must be made of light material, preferably wood, so that in case of an explosion the débris shall only be projected for small distances. In this country the greatest importance is attached to constructing the magazines so that they shall be burglar-proof, and to the storage of only explosives of trustworthy quality in them, so that the probability of an explosion shall be small. In the present state of the explosives industry, sufficiently stringent regulations for testing explosives can be insisted on, so that while their safety under all ordinary circumstances is guaranteed, the makers are not unduly hampered. There is, therefore, no reason to assume the possibility of accidental explosions in magazines from the bad quality of the material stored. It is true that explosions may be produced by lightning and by incendiarism, but it is much better to guard against the effects of such cases by placing the magazines at suitable distances from inhabited houses or frequented roads, and by not allowing the quantity stored in a magazine to exceed a certain maximum, depending on these distances. In this country magazines are required to have walls at least 18 inches thick, made of concrete or built with large stones, or good bricks, laid in cement. The roofs or ceilings must be made either of strong wooden beams, at least 6 inches thick, and not more than 6 inches apart, or of strong wire netting, or entirely of iron, etc., so that burglary is practically impossible. Magazines built entirely of angle-bars and corrugated iron are also permitted so long as they are quite burglar-proof. Double doors must be provided, both opening outwards, and the outer one must be either made of iron, or be lined with sheet-iron. The outer door must have either two locks, or one treble-bolt lock. Protecting mounds round magazines are only insisted on when the distance from neighbouring structures to be protected is not sufficient; in general, the distance from such a structure is allowed to be reduced to one-half, if earth mounds be provided. In such magazines explosives of all kinds can be stored, subject, of course, to their being on the list of authorized explosives. Detonators are an exception and must be stored in separate magazines, or, if they have to be stored in the same building, must be in a separate room, and there must be a free air space of at least 3 feet between this room and the one in which the explosives are stored. Windows are not allowed and no work is permitted in the magazines, but if cases or barrels have to be opened or closed, a special partitioned-off part must be provided for doing it in. No one is allowed to enter a magazine except in leather boots made without nails, or in felt slippers. Artificial lighting is not permitted.

An important question in connection with magazines for storing explosives in is their protection against lightning. In nearly all countries the provision of a lightning-conductor attached to the building is insisted on, and where no such regulation exists, it is, as a rule, assumed as a matter of course. There are different, sometimes considerably varying regulations for placing and connecting such lightning-conductors. Formerly, for instance, the conducting wires were carried down the building through insulating rings of glass. A committee which sat in England decided that this was dangerous, and that the clamps for the conductors should be made of the same metal as the conductors themselves. The different regulations contradict each other materially as regards the height of the lightning-rod and its material and shape. In general, it is agreed that the earth plate should be as large as possible, and that it should be put either into water, or into permanently moist soil, or, if neither of these be available, into a pit filled with coke. There are also different regulations as to how lightning-conductors should be tested from time to time, by means of galvanometers, etc., to ensure that they are in working order.

Unfortunately, it has happened that even magazines which had been tested only a short time before a thunder-storm, and found in perfect order, have been struck by lightning. Such cases have happened frequently within the last few years, and have greatly troubled the interested persons. Professor Oliver

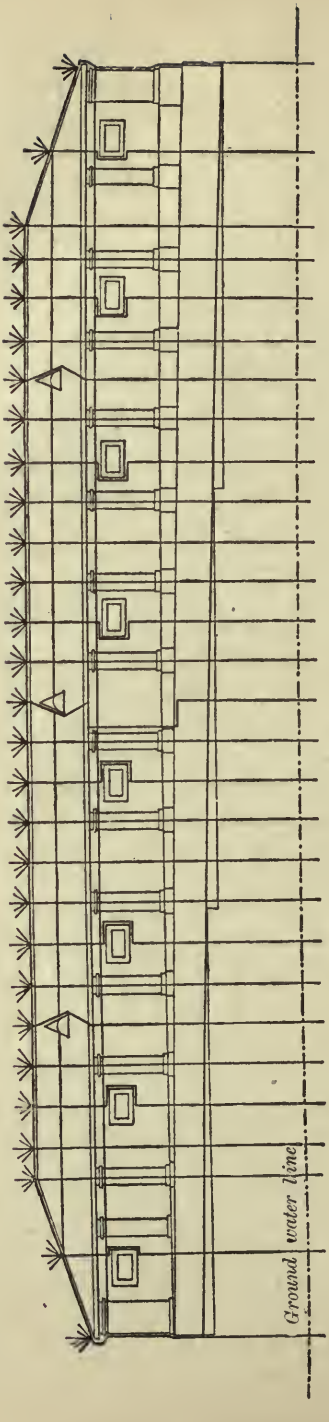


Fig. 321.

Lodge has shown that the ordinary way of trying to protect buildings by means of lightning-conductors can only be imperfect, and the author himself has repeatedly pointed out in 'Dingler's Polytechnisches Journal,' and in other publications, that with magazines for explosives the presence of a lightning-conductor may, under favourable circumstances, be a direct danger in itself instead of giving protection. Opinions are still divided as to the best method for giving protection from lightning, but it seems that an idea originally due to Melsens, and developed later on by Professor Lodge, comes nearest to the solution. It consists in surrounding the building either completely with a network of wires, or in conducting along the roof edges and corners of the building barbed wires which are earthed at several places.

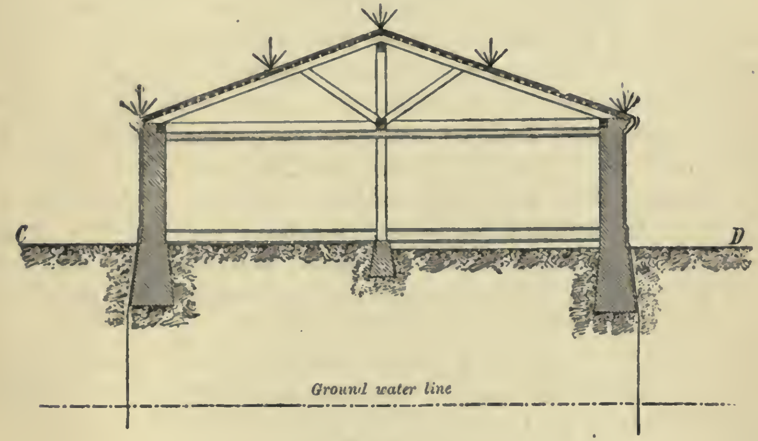


Fig. 322.

Such a magazine, which was built in Russia, and the construction of which is evident, is illustrated in Figs. 321 and 322. Instead of the many high lightning-rods only short bundles of wires are provided.

The best protection is probably given by making the building entirely of metal, and placing the foundations either wholly in moist soil or connecting them with it at various places; several magazines have, in fact, been built entirely of sheet-iron over iron frames.

It is absolutely necessary that magazines in which nitro-compounds, or other explosives liable to decomposition under favourable circumstances, are to be stored, shall be well ventilated, and, at the same time, sand and dust must be prevented from entering, and wilful incendiarism also made impossible. The best ventilators for this purpose are earthenware or metal pipes of syphon shape.

The barrels or cases for explosives should never rest against the wall, and should also be kept away from the floor by being placed on planks or battens. They should also be stored in such a manner that the labels or other distinguishing marks on the packing cases can be distinctly seen, and so that each case can be conveniently taken out and handled if required.

The regulations as to the carriage of explosives also differ greatly in the various countries, but they cannot be more than just referred to here. The chief points insisted on, amongst many others, are to have no protruding metal parts in the carriages; that the single packing cases shall be packed safely and firmly to prevent shaking; that suitable arrangements for the safety of the neighbourhood shall be made, in case a stoppage has to be made at a railway station or over night in carts, etc.; also that the carriages shall be kept at suitable distances from inhabited houses or much-frequented railway stations; that the carts shall be recognizable from a distance by signal flags, etc.; and that the men in charge of the carts shall abstain from lighting any fire, match, or such like.

In general, it may be said that accidents during the storage or carrying of explosives are of very rare occurrence when suitable regulations have been made, and when the common sense of people keeps them from foolhardy or rash actions.

CHAPTER XXIV

ON THE CONSTRUCTION OF EXPLOSIVES FACTORIES IN GENERAL

UNDER the present conditions obtaining in the industry in general, where machine work has to a great extent supplanted hand work, and consequently competition is not only easier, but has considerably increased in every branch of industry, the building of explosives factories is no longer so simple as in former times, but requires a far greater amount of technical knowledge and capital. Small explosives factories can only successfully exist under special local circumstances; but even very large factories, especially those making nitro-compounds, can only work with success when the whole of the products resulting as waste during the manufacture can be re-utilized with advantage, and when the majority of the prime materials required are made by the factory itself. Very frequently the profit resulting from such by-manufactures is an essential condition for the successful existence of the factory.

Now-a-days the building of an explosives factory is everywhere subject to a license which is given by the Government authorities with due consideration to all conditions required for safety. These are, in the first instance, a suitable distance of the buildings from each other, and from neighbouring inhabited houses and frequented roads and paths. In most countries also the political and individual responsibility of the licensee is considered, and it is insisted that the management of the factory shall be in the hands of competent chemists, or engineers, with experience in the special branch of manufacture carried on. In Great Britain and its colonies, and also in America, the latter condition is not essential, since in these countries the personal and monetary responsibility of the owner of the factory to his workmen, and with regard to damaging other people's property, is so great that it is to the interest of the manufacturer to use the greatest precautions and employ the best people.

Neither way of licensing is without fault, but it has been shown so far, that the system prevailing in this country, which is controlled by frequent visits from the Government Inspectors and by the imposition of severe penalties for illegal acts, is not only perfectly sufficient, but also interferes least with the manufacturers, and at the same time gives such a high degree of safety, that the English explosives factories are at present, without any doubt, generally looked up to as models.

Since the regulations for the building of explosives factories are sometimes left to the will of a single Government official, and even vary in some districts of one and the same country, only two sets of regulations which were made by law, and both of which act in a perfect way, will be dealt with here. These are the English and the Austro-Hungarian regulations.

In Austria-Hungary the buildings of an explosives factory are divided into several groups. The first comprises rooms for the manufacture of the explosives compounds, the second those for the manufacture of the finished explosives from these compounds, the third those for making cartridges and packing, the fourth the magazines for storing the finished explosives, and the fifth the dwelling-houses. Buildings belonging to any of the first three groups must be at least 50 metres from a building of another group. The two latter groups must be 200 metres from each other, and also at least 200 metres from buildings belonging to any other group.

Magazines holding 2000 kilogrammes must be placed at least 100 metres from each other, and those with a maximum capacity of 10,000 kilogrammes at least 200 metres from each other. The buildings of the first four groups must be placed apart from each other and surrounded with mounds of earth at least 1 metre wide on the top, and as high as the ridge of the roof. All parts of the factory where there are explosives at any time must be at least 500 metres from any buildings outside the factory area, when consent in writing has been obtained from the owner, and the same distance from unfrequented paths and roads. From inhabited houses, when written consent has not been obtained, and also from railways, water-ways, country-, county-, and district-roads, and from much-frequented footpaths they must be at least 1000 metres off.

In Great Britain the arrangement of the individual buildings is left to the manufacturer, subject to special objections being raised against it by the Home Office. The distance of the buildings from each other is regulated by the maximum quantity of explosives allowed in them. The following table shows these distances.

TABLE SHOWING DISTANCES FROM PROTECTED WORKS FOR MAGAZINES AND OTHER DANGER BUILDINGS.

In any case where the protected work is effectively screened from the Magazine or Danger Building, either by the natural features of the ground or by good and substantial artificial mounds of earth or mine refuse, of such height that a line drawn from any part of the Magazine or Danger Building to any part of the Protected Work in question will pass through the intervening ground or mound, the distance from that Protected Work (except for quantities of 1000 lbs. and under) will be reduced one-half.

Provided that when a natural hill so intervenes as to afford a degree of protection which in the opinion of a Government Inspector justifies a further reduction, the distance shown in the table will be reduced to one-quarter.

In no case, however, is the distance from her Majesty's Palaces to be less than two miles.

The figures printed in small type are the distances to be observed when gunpowder only is to be stored, or other explosives up to the equivalent of 5000 lbs. of gunpowder, every half-pound of such other explosive (with certain exceptions) being reckoned as 1 lb. of gunpowder.

AMOUNT OF EXPLOSIVE TO BE ALLOWED IN THE MAGAZINE OR DANGER BUILDING.		DISTANCES TO BE KEPT CLEAR FROM							
tons	lbs.	No. 1 in yards.	No. 2 in yards.	No. 3 in yards.	No. 4 in yards.	No. 5 in yards.	No. 6 in yards.	No. 7 in yards.	No. 8 in miles.
	500	50_{35}	—	100_{65}	—	50_{35}	100_{65}	880_{585}	2
	1000	50_{35}	75_{50}	150_{100}	—	75_{50}	150_{100}	1320_{880}	2
1	2000	50	100_{75}	200_{150}	—	100_{75}	200_{150}	1760_{1320}	2
	3000	51	101_{90}	200_{175}	210	110_{90}	240_{175}	1780_{1540}	2⅛
2	4000	52	102	200	215_{200}	120_{100}	280_{200}	1805_{1760}	2¼
	5000	52	104	200	225_{210}	130_{110}	320_{255}	1825_{1790}	2¼
3	6000	53	105	200	235_{220}	140_{125}	365_{310}	1850_{1820}	2⅜
	7000	53	106	200	240_{230}	145_{140}	405_{360}	1870_{1850}	2½
4	8000	54	108	200	250_{245}	155_{150}	445_{415}	1890_{1880}	2½
	9000	54	109	200	255	165_{160}	485_{470}	1910_{1905}	2⅝
5	10,000	55	110	200	265	175	525	1930	2¾
	11,000	55	111	205	270	180	560	1950	2¾
	12,000	56	112	210	280	190	590	1970	3
	13,000	56	113	215	285	200	625	1990	3
	14,000	57	114	220	290	205	655	2005	3
	15,000	57	115	225	300	215	690	2025	3¼
	16,000	58	116	230	305	220	720	2040	3¼
	17,000	58	117	235	310	230	750	2060	3¼
	18,000	59	118	240	315	235	785	2075	3½
	19,000	59	119	245	325	245	815	2095	3½
10	20,000	60	120	250	330	250	850	2110	3½
	22,000	61	122	265	345	265	920	2145	3¾
	24,000	62	124	280	355	280	990	2180	3¾
	26,000	63	126	295	370	295	1060	2215	4
	28,000	64	128	310	380	310	1130	2255	4¼

Amount of Explosive to be Allowed in the Magazine or Danger Building.		Distances to be Kept Clear from							
tons	lbs.	No. 1 in yards.	No. 2 in yards.	No. 3 in yards.	No. 4 in yards.	No. 5 in yards.	No. 6 in yards.	No. 7 in yards.	No. 8 in miles.
15	30,000	65	130	325	395	325	1200	2290	4¼
	32,000	66	132	340	410	340	1265	2325	4½
	34,000	67	134	355	420	355	1330	2360	4¾
	36,000	68	136	370	435	370	1395	2395	4¾
	38,000	69	138	385	445	385	1460	2430	5
20	40,000	70	140	400	460	400	1525	2465	5¼
	42,000	71	142	415	475	415	1590	2500	5¼
	44,000	72	144	430	485	430	1655	2535	5½
	46,000	73	146	445	500	445	1720	2570	5¾
	48,000	74	148	460	510	460	1785	2605	5¾
25	50,000	75	150	475	525	475	1850	2640	6
	52,000	76	152	490	540	490	1915	2675	6¼
	54,000	77	154	505	550	505	1980	2710	6¼
	56,000	78	156	520	565	520	2045	2745	6½
	58,000	79	158	535	575	535	2110	2780	6½
30	60,000	80	160	550	590	550	2175	2815	6¾
	62,000	81	162	565	605	565	2240	2850	7
	64,000	82	164	580	615	580	2305	2885	7
	66,000	83	166	595	630	595	2370	2920	7¼
	68,000	84	168	610	640	610	2435	2955	7½
35	70,000	85	170	625	655	625	2500	2990	7½
	72,000	86	172	640	670	640	2570	3025	7¾
	74,000	87	174	655	680	655	2640	3060	8
	76,000	88	176	670	695	670	2710	3100	8
	78,000	89	178	685	705	685	2780	3135	8¼
40	80,000	90	180	700	720	700	2850	3170	8½
	82,000	91	182	715	735	715	2915	3205	8½
	84,000	92	184	730	745	730	2980	3240	8¾
	86,000	93	186	745	760	745	3045	3275	8¾
	88,000	94	188	760	770	760	3110	3310	9
45	90,000	95	190	775	785	775	3175	3345	9¼
	92,000	96	192	790	800	790	3240	3380	9¼
	94,000	97	194	805	810	805	3305	3415	9½
	96,000	98	196	820	825	820	3370	3450	9¾
	98,000	99	198	835	835	835	3435	3485	9¾
50	100,000	100	200	850	850	850	3500	3520	10

In the above table—

No. 1 means: Room used in connection with the magazine, in pursuance of section 46 of the Act.

No. 2 means: (*a*) Workshop used in connection with the magazine, in pursuance of section 47 of the Act [see note (*b*)].

(*b*) Mineral or private railway, whether worked by steam or otherwise.
(*c*) Highway or public footpath.
(*d*) Promenade or open place of resort for the public, or for persons carrying on any trade or business.
(*e*) Canal or navigable water.
(*f*) Dock.
(*g*) River-wall or sea-water.
(*h*) Pier or jetty.
(*i*) Reservoir.

No. 3 means: (*a*) Room or workshop in connection with another magazine, store, or registered premises.

(*b*) Any other room or workshop or any shop.
(*c*) Magazine for explosive.
(*d*) Store for explosive.
(*e*) Furnace, kiln, or fire for the use of any boiler, engine, or machine, or for any manufacturing purpose.

No. 4 means: Public railway.

No. 5 means: Dwelling-house, *with* the consent in writing of the occupier.

No. 6 means: (*a*) Dwelling-house, without such consent.

(*b*) Factory not belonging to Government.
(*c*) Church or chapel.
(*d*) University, college, or school.
(*e*) Hospital or public institution.
(*f*) Town-hall or court of justice.
(*g*) Covered market.
(*h*) Theatre or buildings wherein persons are accustomed to assemble.
(*i*) Public building in charge of the Commissioners of her Majesty's Works and Public Buildings.
(*j*) Factory or magazine occupied by the Secretary of State, the Commissioners of the Admiralty, or other Department of the Government, *with* the consent, in writing, of the Secretary of State, Commissioners, or Department.

No. 7 means: Do. without such consent.

No. 8 means: Palace or house of residence of her Majesty, her Heirs and Successors.

NOTES.

(*a*) This Table furnishes the basis on which applications for licenses will be considered, but it is susceptible to modification under special circumstances.

(*b*) Buildings and magazines *inside* the factory will be required to preserve the distances from each other specified in No. 2. The same rule will apply to two or more magazines, *not* in a factory, belonging to the same occupier, or by mutual consent of the respective occupiers.

(*c*) Detonators may be kept in an annex near to or adjoining a magazine under the following conditions:—

(1) The amount of explosive contained in the detonators must not exceed 100 lbs., reckoned in the proportion of not less than 2½ lbs. of explosive per thousand detonators. (This proportion represents roughly about 44,000 detonators of "sextuple" strength; with detonators of greater strength, the number would of course be less.)

(2) The detonator annex must be so constructed that not less than two feet of masonry and three feet of air space shall intervene between any detonators in such annex and the interior of the main magazine.

All these distances can be reduced to one-half, if the building is surrounded with an earth mound reaching to the eaves of the roof, or if the natural formation of the ground practically forms a mound, even when such natural features are at some distance from the building.

In Austria-Hungary a detail-plan of each building and of each machine to be used has to be submitted with the application for license, and alterations from them can only be made after special permission has been granted.

In Great Britain the interior arrangement is left entirely to the manufacturer, and also the choice of such machinery as will ensure the process of manufacture being conducted so as not to infringe the legal regulations, but the inspectors of explosives have the right of objecting to any details involving evident irregularities or danger.

In both countries the buildings have to be constructed entirely of light wood, and with light roofs. No exposed metal is allowed inside the building, and the floors must be made without using stone or metal. With liquid explosives, the covering of the floors with kieselguhr, or with india-rubber or gutta-percha rugs, is insisted upon in Austria, in which country the heating of the building must be done by means of warm water. In Great Britain steam and air heating are both permitted.

Whilst in Great Britain the attachment of a lightning-conductor to each building is unconditionally demanded, in Austria-Hungary it is only required when local circumstances make it desirable.

In Great Britain the internal walls of each building have to be papered or varnished.

The above are the broad outlines of the Government regulations for the construction of an explosives factory; but in reality the precautions taken by the manufacturers in their own interest go far beyond them; many of these have been repeatedly mentioned in this book.

It is sometimes open to doubt whether it is of advantage to isolate each building completely from the other, and to place them at a certain distance apart. In the older continental factories whole rows of buildings, for instance, cartridge huts in dynamite factories, are frequently found built into the slope of a hill, and separated from each other by a mound measuring only a few yards at the base, and sometimes even narrower, with retaining walls. In black powder factories also two buildings of the same kind are frequently found separated by a thick brick wall only, and built close to each other. Explosions that have happened have shown that if one of such a pair of buildings explodes, the other one is frequently protected. This is especially the case with explosions of nitro-compounds, where the "plume" rises rapidly and takes the form of a steep funnel. In this case, the effect of the explosion in the immediate neighbourhood is not so great as at a greater distance, where it spreads far more. Cases have happened in such a double building where not even the window-panes were broken, but at distances of one hundred yards and more the crowns of trees were mowed down, as it were, and buildings greatly damaged. It can, however, be assumed that the presence of another building in the immediate neighbourhood is only desirable in exceptional cases, when the arrangement of the plant demands it, for instance, because it is far more probable a building near to will be wrecked and even set on fire, than that it will escape damage. The English system of regulating the distances by the quantity of explosive in the building is on the face of it rational, and has proved to be very effective.

The construction of buildings with hollow, double-boarded walls, filled in with small coke, sand, sawdust, or similar non-conducting material, is not quite free from objection. The durability of the buildings is not very great, as it is frequently difficult to keep their foundations free from moisture from the soil. The filling is frequently apt to absorb moisture from the air, and wood-pulp, sawdust, and such-like are further objectionable, because they are liable to spontaneous ignition under certain conditions. On the other hand, if the walls are left hollow, and merely boarded on both sides, it is difficult to permanently keep them tight enough to prevent the air enclosed between the two layers of boards from circulating, and so make it act as a non-conductor. A double wooden wall, filled in with coke, at the same time makes the building more solid, and also better suited to resist the air-pressure from an explosion in the neighbourhood, than a rigid brick building.

The best coverings for roofs are probably felt, waterproof cloth, or sheet-zinc, and they can be brought now-a-days to great perfection. Slate, tile, and such-like roofs are quite inadmissible, except, perhaps, in some cases for magazines. For other danger buildings they are decidedly objectionable, because in the event of an explosion such thin, flat bodies, especially slates, always fall end on, and fly through the air like so many knives. The author has found whole rows of such slates sticking fast in the ground at great distances after the explosion of such a building.

Varnishing or papering the buildings inside; daily sweeping and frequent washing of the floors; the wearing of felt or leather boots, which must be so put on that the foot, when the ordinary boot is on, never comes into the interior of the building, whilst, when wearing the protecting boot, it never comes outside it; the thorough daily cleaning of all apparatus and machinery; the carrying away of used cotton waste; the wearing of uninflammable clothes (wool, leather, etc.) with no pockets; proper facilities for the workmen to wash thoroughly after the work is over, and to change their clothes; the frequent searching of the workmen to prevent metal or any articles liable to cause fire being brought in; and, in general, the care of each detail in manufacture, and the constant maintenance of the whole installation in perfect order, although they may sometimes appear like unnecessary refinements, or unduly costly supervision, are certainly of great importance for the safety and proper working of an explosives factory. As the author has previously mentioned, it is certainly not the absence of a little sand in the building which makes the workman safe, because in nine hundred and ninety-nine cases out of a thousand it would be harmless, but it is the general habit of cleanliness and caution which is inculcated in the workmen, by insisting on order and attention in every direction, so that it finally becomes a second nature with them, and imbues them with a general feeling of safety and comfort.

The artificial lighting of an explosives factory is of considerable importance, where both day and night work is necessary, or in the winter, when it is dark in the mornings and evenings. Where no other means of lighting but paraffin are available, reflector lamps placed outside the windows of the buildings are used. They should be filled and cleaned every day by a person especially told off for the work. The lamps must be so constructed that the wind cannot extinguish them, and also so that no explosive dust can get into them. The latter cannot really be done at all perfectly, and for this reason, and also because lighting by paraffin oil is costly and cumbersome, electrical lighting is now-a-days used almost everywhere. Electric lighting is not without danger, however. It has frequently been stated that an incandescent lamp can be taken into a danger building without fear, because such a lamp can be touched with the hand, without giving a feeling of excessive heat. Nevertheless, experiments made in this country and in Austria have shown that the heat radiated from an electric incandescent lamp can so accumulate in a suitable medium, as to ignite with ease materials the temperature of combustion of which is even over 200° C. If an incandescent lamp be intentionally covered with gun-cotton dust, the latter will soon be ignited, and a lamp placed in a fairly small vessel with water will heat the water to boiling. If such an incandescent lamp be put into a glass globe of such diameter that the heat given off by the lamp can radiate gradually, and if this globe be completely closed to the atmosphere by an india-rubber or cement joint, in general no danger exists, and such lamps can be placed in ordinary danger buildings. In buildings where large quantities of explosives dust are developed, even this precaution is not sufficient, because the dust can always enter through fine cracks or faults in the joint, and it is therefore advisable to put the lamp with the glass globe in a recess in the wall, which is made perfectly tight, and is closed on the inside of the building by a glass plate. In the author's opinion it is not advisable to put such lamps in a globe filled with water, because the hot water, which may under favourable circumstances be heated to near the boiling-point, always makes the explosives dust very sensitive, and because, in case the conducting wires are defective, a short circuit may occur.

The author always leads the wires to the lamps, so that they enter the lamp from opposite sides of the building, if possible, but in any case with a space of at least 20 inches between them. Naked wires should never be allowed in a building; they must be well insulated and laid in casings of impregnated wood or metal. Each lamp should have its own switch, and a fusible cut-out as well. Groups of buildings should be arranged to be switched out by a main switch, and it is further of advantage to divide the whole factory into several groups, which can be cut off from the main circuit by means of switches, both at the dynamo and at suitable places near each group.

Lightning-arresters should be provided in various places in the circuit, and an apparatus should be provided in the dynamo-room to cut out at once a whole group, in case the tension exceed a certain limit. Under such circumstances it is much better to have one part of the factory temporarily in darkness, than to allow the danger of an excessive current flowing through the circuit to continue.

In Great Britain special regulations for electric lighting have been issued by the Inspectors of Explosives. Their chief points are identical with what has been said above, and they will be found *in extenso* in the Appendix.

The general state of the factory ground and its maintenance are not without importance in an explosives factory. It is decidedly advisable, wherever it can be done, to plant the ground with as many trees as possible, as they give protection against the wind, and, what is more important, against damage in case of an explosion. In addition, they are excellent lightning-conductors, and moreover good vegetation in general prevents dust from being blown into the interior of the buildings. If the factory be built on sand, and the planting of trees therefore not possible, the grass should be carefully attended to. On the other hand, it should be frequently cut, to prevent the accumulation of dry long grass, rushes, furze, etc., since in the case of an explosion such materials easily catch fire, and by spreading it to great distances, as they are very likely to, may cause other buildings to ignite.

The waste waters from each single operation in an explosives factory must be rendered harmless with the greatest care. The discharge of acid waters in the public channels is nearly everywhere prohibited by law, but also in the interior of a factory it should be disposed of in such a way that the water-supply of the factory is not interfered with. In factories where the waste water carries particles of explosives, it is of still greater importance that it should be perfectly freed from them before getting into the soil. This is best done by means of settling tanks, filtering arrangements, etc. Explosions have repeatedly happened from such particles of explosives gradually collecting in dikes, channels, etc., and then being decomposed or exploded by the sun's rays or other accidents.

The degree of safety existing in a factory, and the perfection to which the precautionary measures providing for the safety of the factory are brought, will, in general, depend upon the attention and management of the supervising officials.

APPENDIX

I. HEAT TEST AS APPLIED TO EXPLOSIVES OF THE NITRO-COMPOUND CLASS.

(As defined by Order in Council, No. 1, of 5th August, 1875.)

1. GENERAL INSTRUCTIONS.

Apparatus required.

1. A water-bath, consisting of a spherical glass or copper vessel (A, Fig. 323), of about 8 inches diameter, and with an aperture of about 5 inches; the bath is filled with water to within a quarter of an inch of the edge. It has a loose cover of sheet-copper about 6 inches in diameter (B), and rests on a tripod stand about 14 inches high (C), which is covered with coarse iron wire gauze (E), and is surrounded with a screen of thin sheet-tin or copper (D). Within the latter is placed an argand burner (F), with glass chimney. The cover (B) has four holes, arranged as seen in Fig. 324; No. 4 to receive the regulator, No. 3 the thermometer, Nos. 1 and 2 the test-tubes containing the gun-cotton or other materials to be tested. Around holes 1 and 2 on the under side of the cover are soldered three pieces of brass wire with points slightly converging (Fig. 325); these act as springs, and allow the test-tubes to be easily placed in position and removed.

*2. Scheibler's or Page's temperature regulator.

*3. Two cells of Le Clanché's battery No. 1 } if Scheibler's regulator
*4. A few yards of insulated copper wire } be used.

5. Test-tubes from $5\frac{1}{4}$ to $5\frac{1}{2}$ inches long, and of such diameter that they will hold from 20 to 22 cubic centimetres of water when filled to a height of 5 inches.

6. India-rubber stoppers, fitting the test-tubes and carrying an arrangement for holding the test-paper, viz. a narrow glass tube

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* This is not absolutely required, as the temperature of the bath can be kept constant by proper attention to the heating flame.

passing through the centre of the stopper, drawn out so as to form a hook, or terminating in a platinum wire hook (Fig. 326).

7. A thermometer, with range not less than from 30° to 212° Fahrenheit.

8. A minute clock.

Materials required.

(*a*) *Test-paper.*—The test-paper is prepared as follows:—45 grains of white maize starch (corn-flour), previously washed with cold water, are added to $8\frac{1}{2}$ ounces of distilled water, the mixture is stirred, heated to boiling, and kept gently boiling for 10 minutes; 15 grains of pure potassium iodide (*i. e.* which has been re-crystallized from alcohol) are dissolved in $8\frac{1}{2}$ ounces of distilled water. The two solutions are thoroughly mixed and allowed to get cold. Strips, or sheets, of white English filter-paper, previously washed with water and re-dried, are dipped into the solution thus prepared, and allowed to remain in it for not less than 10 seconds; they are then allowed to drain and dry in a place free from laboratory fumes and dust. The upper and lower margins of the strips, or sheets, are cut off and the paper is preserved

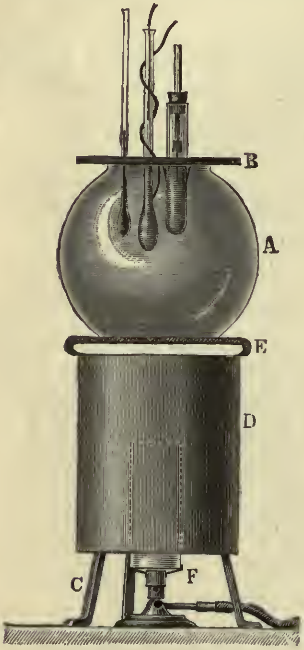

Fig. 323.

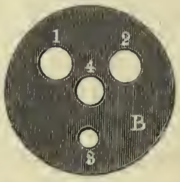

Fig. 324.

Fig. 325.

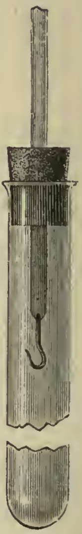

Fig. 326.

in well-stoppered or corked bottles, *and in the dark.*[1] The dimensions of the pieces of test-paper used are about $\frac{4}{10}$ inch by $\frac{8}{10}$ inch (10 millimetres by 20 millimetres).

(*b*) *Standard tint-paper.*—A solution of caramel in water is made of such concentration that when diluted one hundred times (10 cubic centimetres made up to 1 litre) the tint of this diluted solution equals the tint produced by the Nessler test in 100 cubic centimetres of water containing 0·000075 gramme of ammonia or 0·00023505 gramme of chloride of ammonium. With this caramel solution lines are drawn on strips of white filter-paper[2] by means of a clean quill pen. When the marks thus produced are dry the paper is cut into pieces of the same size as the test-paper previously described, in such a way that each piece has a brown line across it near the middle of its length, and only such strips are preserved in which the brown line has a breadth varying from $\frac{1}{2}$ millimetre to 1 millimetre ($\frac{1}{50}$ of an inch to $\frac{1}{25}$ of an inch).

2. TESTING DYNAMITE, BLASTING GELATINE, AND OTHER EXPLOSIVES OF THE FIRST DIVISION OF THE NITRO-COMPOUND CLASS.

A.—Dynamite, etc., etc.

Nitro-glycerin preparations, from which the nitro-glycerin can be extracted in the manner described below, *must* satisfy the following test, otherwise they will not be considered as manufactured with "thoroughly purified nitro-glycerin" within the terms of the license.

This test, however, though at present looked upon as the most important, as far as testing the purity of the nitro-glycerin is concerned, is only one of several which any given sample of nitro-glycerin preparation has to satisfy in order to establish its compliance with the definition in the license.

The test, although at present accepted as regulating and defining the meaning of the term "thoroughly purified," may nevertheless be

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[1] When the paper is freshly prepared, and as long as it remains in good condition, a drop of dilute acetic acid, put on the paper with a glass rod, produces no coloration. In process of time, however, the stronger the light to which the paper is exposed, the sooner a drop of the acid produces a brown or bluish coloration (a single hour of direct sunlight produces a marked effect), and whenever this is the case the paper should be rejected. On this account it is advisable not to prepare too much of the test-paper at one time, but to prepare it fresh every month or so.

[2] This paper must be carefully washed with distilled water in the first instance, to remove any traces of bleaching matter, and dried.

modified or superseded if, in the opinion of the Home Office, such alteration may at any time be deemed necessary.

Apparatus required.

A funnel 2 inches across, and a cylindrical measure divided into grains.

Mode of Operation.

About 300 to 400 grains of dynamite finely divided are placed into the funnel, which has previously been loosely plugged by some freshly-ignited asbestos.

The surface is smoothed by means of a flat-headed glass rod or stopper, and some clean washed and dried kieselguhr is spread over it to the depth of about $\frac{1}{8}$ inch.

Water is next carefully dropped from a wash-bottle upon this kieselguhr, and when the first portion has been soaked up, more is added; this is repeated until sufficient nitro-glycerin has been collected in the graduated measure below.

If any water should have passed through with the nitro-glycerin, it should be removed with a piece of blotting-paper, and the nitro-glycerin, if necessary, filtered through a dry paper filter.

Application of the Test.

The thermometer is fixed so as to be inserted through the lid of the water-bath described under (1), p. 387, into the water (which is to be steadily maintained at a temperature of 160° F.) to a depth of $2\frac{3}{4}$ inches. Fifty grains of nitro-glycerin, to be tested, are weighed into a test-tube in such a way as not to soil the sides of the tube. A test-paper is fixed on the hook of the glass rod so that when inserted into the tube it will be in a vertical position. A sufficient amount of a mixture of half distilled water and half glycerin to moisten the upper half of the paper is now applied to the upper edge of the test-paper, by means of a camel's-hair pencil, the cork carrying the rod and paper is fixed into the test-tube, and the position of the paper adjusted so that its lower edge is about half-way down the tube; the latter is then inserted through one of the perforations of the cover to such a depth that the lower margin of the moistened part of the paper is about five-eighths of an inch above the surface of the cover. The test is complete when the faint brown line which after a time makes its appearance at the line of boundary between the dry and moist part of the paper equals in tint the brown line of the standard tint-paper.

The nitro-glycerin under examination will not be considered as "thoroughly purified" within the terms of the license, unless the time necessary to produce the standard tint as above described is at *least* 15 minutes.[1]

B.—BLASTING GELATINE, GELATINE DYNAMITE, AND ANALOGOUS PREPARATIONS.

Fifty (50) grains of blasting gelatine are to be intimately incorporated with one hundred (100) grains of French chalk.[2] The mixture is to be gradually introduced into a test-tube of the dimensions prescribed above for the dynamite heat test, with the aid of gentle tapping upon the table, between the introduction of successive portions of the mixture into the tube, so that when the tube contains all the mixture it shall be filled to the extent of $1\frac{3}{4}$ inches (one inch and three-quarters) of its height. The test-paper is then to be inserted and the heat is to be applied in the manner prescribed above for the dynamite heat test, and the sample tested is to withstand exposure to 160° F. for a period of ten (10) minutes, before producing a discoloration of the test-papers corresponding in tint to the standard colour test which is employed for governing the results of the dynamite heat test.

(For Exudation and Liquefaction tests for Blasting Gelatine, etc., etc., see p. 394.)

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[1] In factory laboratories where many tests have to be made every day, larger water-baths are preferred, and further, the nitro-glycerin is not weighed but is taken up by a pipette holding about 50 grains when filled to a mark, and its contents are allowed to drop on to the bottom of the test-tube. The test-paper must never be touched with the hands, since the least impurity on them influences the paper. It is, therefore, advisable to have a large piece of cork in readiness, on to which the test-paper is put from the store-glass by means of a pair of pincers, and held there with them, whilst with a second pair of pincers a hole is first made in the paper, and then the glass hook is inserted into the hole. The glycerin mixture can then be put on to the paper by means of a glass rod, and as a rule a small drop is sufficient for the purpose. O.G.]

[2] This can be readily effected by carefully working the two materials together with a wooden pestle in a wooden mortar.

The French chalk should be of good commercial quality, and, after being carefully washed with distilled water and dried in a water-oven, it should be exposed under a bell-jar to moist air until it has taken up about 0·5 per cent. of moisture. It should then be bottled for use ; and with ordinary care the limits of 0·5 per cent. of moisture can be maintained in keeping.

3. TESTING GUN-COTTON, SCHULTZE GUNPOWDER, E. C. POWDER, AND OTHER EXPLOSIVES OF THE 2ND DIVISION OF THE NITRO-COMPOUND CLASS.

A.—Compressed Gun-cotton, Tonite, etc., etc.

Sufficient material to serve for two or more tests is removed from the centre of the cartridge by gentle scraping, and, if necessary, further reduced by rubbing between the fingers.

The fine powder thus produced is spread out in a thin layer upon a paper tray 6 inches by $4\frac{1}{2}$ inches, which is then placed inside a water-oven, kept, as nearly as possible, at 120° F.

The wire-gauze shelves in the oven should be about 3 inches apart. The sample is allowed to remain at rest for 15 minutes in the oven, the door of which is left wide open.

After the lapse of 15 minutes the tray is removed and exposed to the air of the room for two hours, the sample being at some point within that time rubbed upon the tray with the hand, in order to reduce it to a fine and uniform state of division.

Application of the Test.

The cover of the water-bath is fitted with the gas regulator which is inserted through the centre hole (No. 4). The thermometer is fixed into hole No. 3. The water in the bath is then heated to 150° F., and the regulator set to maintain that temperature. Twenty grains of the sample to be tested are weighed out, placed in the test-tube, and gently pressed down until the specimen occupies a space of as nearly as possible $1\frac{5}{16}$ inches in a test-tube of the dimensions specified. A test-paper is affixed to the hook of the glass rod or tube, and moistened by touching the upper edge with a drop of distilled water containing 50 per cent. of Price's glycerin. The quantity of liquid used must be only sufficient to moisten about half of the paper. The cork carrying the rod and test-paper is then fixed into the test-tube, and the latter inserted into the bath to a depth of $2\frac{1}{2}$ inches, measured from the cover, the regulator and thermometer being inserted to the same depth. The test-paper is to be kept near the top of the test-tube, but clear of the cork, until the tube has been immersed for about five minutes. A ring of moisture will about this time be deposited upon the sides of the test-tube a little above the cover of the bath; the glass rod must then be lowered until the lower margin of the moistened part of the paper is on a level with the bottom of the ring of moisture in the tube; the paper is now closely watched. The test is complete when a very faint brown coloration makes its appearance at the line of boundary between the dry and moist parts of the paper.

The interval of time between the first insertion of the tube containing the sample of gun-cotton in the water at 150° and the first appearance of discoloration on the paper constitutes the test, and this interval of time must be *not less* than 10 minutes, or the sample will not be accepted as properly purified.

B.—SCHULTZE POWDER, E. C. POWDER, COLLODION COTTON, ETC., ETC.

Sufficient of the sample, without further mechanical division, is dried in the oven as above, and then exposed for two hours to the air. The test as directed above for Compressed Gun-cotton, etc., is then applied, the minimum duration of test being the same, viz. 10 minutes.

V. D. MAJENDIE, COLONEL,
H. M. CHIEF INSPECTOR OF EXPLOSIVES.

HOME OFFICE, WHITEHALL, S.W.,
September 1st, 1886.

4. TESTING CORDITE AND SIMILAR SMOKELESS POWDERS.

From each end of each piece of cordite selected for the test, pieces of ½-inch length are cut off. With thicker cordite each piece is further cut into about four parts. (With flake or cube powder a division is made in a similar manner.) The pieces so cut are passed two or three times through a pug mill, whereby the part first passing is put aside, because it may contain foreign substances from the mill. The material ground is passed through a set of three sieves. That which has passed through the upper sieve and is retained by the second one is used for the test. After each grinding the mill must be taken to pieces and carefully cleaned.

To make the test, 25 grains of cordite are put into the test-tube with light shaking. They are provided with a test-paper as described above, and this is moistened with glycerin. The water in the water-bath is kept at a temperature of 180° F. The lower end of the moistened part of the paper should be about ⅝ inch above the surface of the cover. The brown line on the test-paper must not appear before 15 minutes.

5. EXUDATION AND LIQUEFACTION TEST FOR BLASTING GELATINE, GELATINE DYNAMITE, AND ANALOGOUS PREPARATIONS.

Test for Liquefaction.

A cylinder of blasting gelatine is to be cut from the cartridge to be tested, the length of the cylinder to be about equal to its diameter, and the ends being cut flat.

The cylinder is to be placed on end on a flat surface without any wrapper, and secured by a pin passing vertically through its centre.

In this condition the cylinder is to be exposed for one hundred and forty-four (144) consecutive hours (six days and nights) to a temperature ranging from 85° to 90° F. (inclusive), and during such exposure the cylinder shall not diminish in height by more than one-fourth of its original height, and the upper cut surface shall retain its flatness and the sharpness of its edge.

Note.—If the blasting gelatine and the gelatine dynamite to be tested be not made up in a cylindrical form, the above test is to be applied with the necessary modifications.

Test for Liability to Exudation.

There shall be no separation from the general mass of the blasting gelatine or gelatine dynamite of a substance of less consistency than the bulk of the remaining portion of the materials under any conditions of storage, transport, or use, or when the material is subjected three times in succession to alternate freezing and thawing, or when subjected to the liquefaction test hereinbefore described.

II. ELECTRIC LIGHTING OF FACTORIES AND MAGAZINES FOR EXPLOSIVES.

MEMORANDUM.

A.—Introductory Observations.

The question of whether, if the globe of an incandescent electric lamp be broken in gunpowder dust, the carbon filament will retain momentarily enough heat to explode the dust, was experimentally investigated at the Royal Gunpowder Factory, Waltham Abbey, in 1881, with the result that the dust was fired. (In this experiment a 40-volt incandescent electric lamp was placed in a box with a sieve containing powder dust fixed to the top. The sieve was then given a shaking motion, while at the same time the glass of the lamp was broken. The powder dust was fired by contact with the incandescent filament.)

This experiment established the insecurity of electric glow-lamps in a dusty atmosphere, unless special precautions be taken against the risk which this experiment illustrated.

But in addition to the risk due to the lamp itself, there are the—probably more formidable—risks connected with the wires, and the possibility of "short circuiting," heating of conductors, and "sparking." There is also the risk due to the fall of a lamp from any cause (*e. g.* from the shock of a neighbouring explosion), causing fracture of the wire and a spark where the lamp is in circuit.

In dealing with factories and magazines for explosives, the character and extent of what for convenience may be called the "danger areas" differ sensibly. Thus while the expression "danger building" is statutorily applied to nearly every building containing explosive, it is obvious that the character or degree of danger (in relation, that is to say, to any system of artificial lighting) differs widely. Between the condition and degree of risk existing in such a building as the corning or dusting-house of a gunpowder factory, for example, and the risk in a danger building where no explosive dust whatever is present, there is really no comparison, and arrangements which would be in the highest degree dangerous in the one case might possibly be safe and suitable in the other.

Again, it is important to recognize that the danger area may (and in the case of dusty buildings generally does) extend to some distance beyond the building itself; while even this secondary danger area (if it may be so described) is liable to occasional extensions by air currents, and especially such as might be readily established in the event of a heavy neighbouring explosion.

Lastly, as continuity of lighting is of great importance, suitable provision should be made for securing it in the case at any rate of important buildings, whether by the use of two or more completely independent supplies, or by the employment of auxiliary storage batteries and two or more independent circuits.

Accordingly, it is desirable that in the electric lighting of all factories and magazines for explosives, the following precautions should be observed :—

B.—Rules.

1. *General.*—The installation generally should be in accordance with the Phœnix Fire Office Rules for Electric Light Installation, except in so far as they may be modified by the following regulations.

2. *Protection of Lamp.*—In all cases the incandescent lamps should be protected by at least one strong outer glass globe, so proportioned to the size of the lamp within that when the latter is burning under its normal conditions the temperature of the exterior of the outer globe or other exposed part shall not exceed 140° F.[1]

3. *Lighting of non-dusty Danger Buildings.*—In the case of danger buildings in which the work carried on is not of a nature to give rise to the presence or formation of explosive dust, it is desirable that the means of lighting should be external to the building, that is to say, although the protecting glass globes may be, strictly speaking, within the building, the interior of the globes should be accessible only from the exterior, and should have a stout sheet of thick glass between them and the interior. But where circumstances render this impracticable, or where the character of the work necessitates a stronger light than can be secured by lamps satisfying the above conditions, internal lamps will not be objected to, provided that the conditions laid down in paragraphs 4, 5, 6 of this Circular, as to the enclosing of the lamps and conductors, and as to the stability and character and sufficiency of all supports, are carefully observed.

4. *Lighting of dusty Danger Areas.*—In the case of dusty danger areas,[2] in addition to all lamps and wires being wholly external to such danger area, the lamps in the immediate vicinity of such danger area should be so enclosed as to render it impossible for dust to settle upon them, an end which may be attained either—

(1) by immersing the lamps in globes containing distilled water,[3] or

(2) by placing them in globes so perfectly sealed by elastic washers, liquid joints, or otherwise, that dust cannot enter.

(N.B.—In the case of (1) the whole of the hollow portion of the lamp should be below the level of the water; and the insulation of the lamp wires or method of protecting them should be such as to be permanently unaffected by the liquid or the moisture which would necessarily spread over the whole. In the case of (2) the sealing must be mechanically strong, capable of examination, renewal, or

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[1] It is desirable that where the globe is exposed to the risk of a blow, it should be protected by a cage of strong wire.

[2] Inclusive not merely of the danger buildings, but of the secondary danger area.

[3] In frosty weather glycerin may be usefully added.

repair, and so disposed that it cannot readily be tampered with or become defective under ordinary conditions.)

5. *Stability of Lamps.*—Each lamp should be so supported as to render it improbable that it could be shaken down by the shock of an explosion in a neighbouring building or otherwise; and when practicable the lamp should be so disposed that, in the event of its falling, it would not be likely to fall into any explosive material.

6. *Conductors, completely enclosed.*—In all cases where the lamps are situated in the immediate vicinity of any danger area,[1] the conductors should be completely enclosed up to the outer globe of the lamp itself, in metal or other non-flexible incombustible casing, so sealed as to be dust-proof, and this casing, if metallic, should be efficiently connected to earth in several places. The lamp wires should be soldered to their leads and not fixed in the ordinary holders provided with contacts, or if holders be used, they should be of such a pattern that an imperfect contact cannot be produced by oxidation or mechanical failure; and, further, no screw-clamps or terminals for connecting the leads, except at the distributing centre, should be permitted.[2] No joints should exist in the leads in the vicinity of any danger area.

7. *Fuses.*—Each separate wire, whether branch or main, should be efficiently protected by a fuse or "cut out" not less than one inch in length, placed outside the danger area, and not in the immediate vicinity of the same, arranged so as to be readily inspected. The fuses should consist of tin wire, and no single wire should be thicker than No. 20 S.W.G. (=0·036″ diameter), additional wires being added in parallel if required.

8. *Switches.*—In every case where a lamp is fixed within any danger area, a double pole-switch must be provided outside the danger area, by means of which the conductors to the lamp can be completely disconnected from the source of supply; and no lamp is on any account to be touched unless it has been entirely disconnected from the source of supply.

9. *Precautions against Fire.*—The arrangements for enclosing and protecting all fuses and switches must be such, that no danger of originating a fire will be incurred from any normal or exceptional action or failure of the fuses or switches.

10. *Leakage Detector.*—A safe and efficient leakage detector should be permanently connected with the system of lighting, and placed in a conspicuous position in the engine or dynamo-room; and suitable orders should be issued to the man in charge concerning the action to be taken on any appearance of leakage.

11. *Wires not to be subjected to Vibration.*—With a view to avoiding

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[1] This expression includes not merely danger buildings, but the secondary danger areas indicated above.

[2] Even if concentric wires are employed, the foregoing rules as to the complete enclosing of the same should be observed.

fracture of wire, with the attendant risk if the current be on, suitable precautions should be taken to prevent any wire carrying a current from being subjected to excessive vibration, such as might be caused by the motion of heavy machinery in its vicinity. Where the boughs of trees overhang wires, it is desirable that the wires should be either carried underground, or if possible protected from the fall on to them of a bough.

12. *Mechanical Strain.*—No wire carrying a current should be used to support a lamp, or be otherwise subjected to mechanical strain.

13. *Lightning.*—The arrangements adopted to protect the mains and wires generally from the effects of lightning, where such mains and wires are unavoidably exposed, should be such that in no case would it be possible for the supply current to follow the "lightning" discharge; in other words, that it would not be possible for the lightning discharge to establish an arc fed by the supply current.[1]

14. *Electrical Pressure.*—The electrical pressure between any two conductors, or between any conductor and the earth, should in no case exceed—

(*a*) In the immediate vicinity of any danger area (as above defined), 110 volts; and

(*b*) Elsewhere within the licensed area of any factory or magazine, 300 volts if continuous or 150 volts if alternating, or such other limits as may for the time being be assigned by the regulations of the Board of Trade as those for a low-pressure supply.

15. *Concentration of the Heat from Sun's Rays.*—Globes, whether or not containing water, if unavoidably placed where the sun's rays may fall upon them, should have one side of the protecting external globe painted (or otherwise so treated) as to render any concentration of the sun's rays impossible.

16. *Arc Lamps not to be used.*—In no case should arc lamps be used in a factory for explosives.

17. *Inspection and Maintenance.*—It must be borne in mind that all electrical installations tend to become defective in time from ordinary wear and tear, as well as from electrical action.

It is therefore essential that a thorough system of testing and inspection should be provided, and that the whole installation should be maintained up to the original standard as regards insulation and mechanical strength.

V. D. MAJENDIE, Colonel,
H. M. Chief Inspector of Explosives.

Home Office,
December 1st, 1892.

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[1] It is suggested that in the case of overhead wires a piece of barbed fencing wire run above the electric lighting wires, and connected to earth at each pole, would form a desirable protection from lightning.

ADDENDA

To vol. i. *p.* 40.—In the 'Chemiker-Zeitung' of April 4, 1894, Major A. Hellich describes some experiments he made with saltpetre containing small traces of chlorine only. When 10 grammes of saltpetre were heated with 0·5 gramme of powdered Pyrolusite in a strong flame on a platinum dish, the mass fused and became perfectly green. He then dissolved the fused mass in 50 cubic centimetres of distilled water, added 20 cubic centimetres of dilute sulphuric acid to the solution when cold, allowed it to rest for a few minutes, and then filtered through a filter that had been previously well washed with dilute sulphuric acid. The addition of silver nitrate to the solution invariably gave a milky turbidity. Without entering into further details of his experiments, it may be mentioned that he came to the conclusion that this turbidity was to be ascribed to some chlorine combination, probably a perchlorate, which in its original form did not give a precipitate with silver nitrate. In some very impure samples he found as much as 0·25 per cent. of this compound.

These experiments led Prof. C. Häussermann ('Chemiker-Zeitung,' 1894, No. 63) to examine a number of samples of reputedly pure saltpetre for the manufacture of black powder. He found perchlorates in all of them without exception. Häussermann is of opinion that the presence of these perchlorates in the saltpetre is due to the sodium nitrate used for the manufacture of Conversion saltpetre already containing perchlorates, which is transformed by the potassium chloride into potassium perchlorate.

Dr. N. Panaotovich ('Chemiker-Zeitung,' 1894, No. 81), by order of the Royal Servian War Office, examined the saltpetre in store at the Royal Gunpowder Factories of Stragare and Obiličevo by Hellich's methods. He examined eighteen kinds of saltpetre, of which some had already been used for the gunpowder manufacture in the fifties and sixties, also some which had been purified by crystallization in the last decade. He found the largest percentage of perchlorates in saltpetre from old liquors, some of which contained from 2 to 2½ per cent., and the lowest in saltpetre extracted from old powders, in which there was under 0·1 per cent.

The question arises whether such a percentage of potassium perchlorate can produce either directly or indirectly an explosion in black powder factories. Panaotovich cites the case of some illuminating stars, made in the Royal Servian Pyrotechnic, which contained potassium perchlorate, and which spontaneously ignited after eleven hours' storage. He further mentions that formerly all saltpetre, however pure, was re-crystallized at Stragare, and that no explosions happened for thirty years, but that they did occur after this purification was given up. The author has no doubt that a large percentage of potassium perchlorate can give rise to objectionable alterations in the powder. It is known that powders containing potassium chlorate do not possess sufficient stability for military and industrial explosives, and the English Inspectors of Explosives have, as a rule, reported unfavourably on any powders containing chlorates. These objections apply to a much greater extent to potassium perchlorate. A smaller percentage (under 0·1 per cent.) may perhaps produce a protracted gradual alteration in a powder, although it is not probable that it will be the direct cause of explosions. Panaotovich purified the saltpetre by dissolving it in a clean copper boiler with filtered river water free from chlorine until it marked 48° B. (1·498 specific gravity), adding a solution of glue to eliminate mechanical impurities, filtering the solution and stirring it in a copper pan, until the solution cooled down to 25° C., when the liquor showed 28° B. (1·241 specific gravity). The crystals which separated out were freed from the mother liquor and well washed five or six times with clean water and subsequently dried. In this way he reduced the percentage of perchlorate in the saltpetre from 0·5 per cent. to merely traces. The mother liquors which contained up to 1·5 per cent. of perchlorate were evaporated by themselves to 48° B., and the saltpetre separating out, as it cooled to 25° C., was treated again as above. From the last mother liquors he obtained by re-crystallization water-clear rhombic columns or crystals which did not dissolve in alcohol, upon which hydrochloric acid did not act, which on heating with sulphuric acid decomposed, and which were perfectly identical with pure potassium perchlorate.

To vol. i. *p.* 122.—According to a report in the 'Mémorial des Poudres et Salpêtres,' 1894, p. 79, the crude ether is now no longer condensed by itself and transferred to the rectifying still, but is at once led from the etherizing apparatus into the rectifying apparatus. In this way it is no longer necessary to heat the rectifying still by steam, since the ether vapours themselves enter at a high temperature.

To vol. i. *p.* 260.—The French powders S. P. 1, S. P. 2, and S. P. 3 are cubical. They are made at the gunpowder factory at St. Chamas by pressing in india-rubber moulds consisting of a rubber plate in which are a number of regular square recesses, and which fit into a bronze frame. The powder is put into the moulds with a scoop, a straight-edge passed over the surface, and the frame transferred to a hydraulic press, where, in the same way as pressing powder cakes, a number of the frames are placed one on the top of the other and compressed together. After pressing, each cube is perfectly free, and it is not necessary to break them apart, as in the Du Pont process. The single cubes also have a perfectly good, smooth surface. According to a report of Hagron and Bruley, comparative experiments made with this powder showed that it was very regular in its action.

To vol. ii. *p.* 8.—In the 'Mémorial des Poudres et Salpêtres,' 1894, p. 91, M. D'Alsace, an engineer in the French service, describes a continuous pump for acids which is made by Kuhlmann Bros. of Lille. Fig. 327

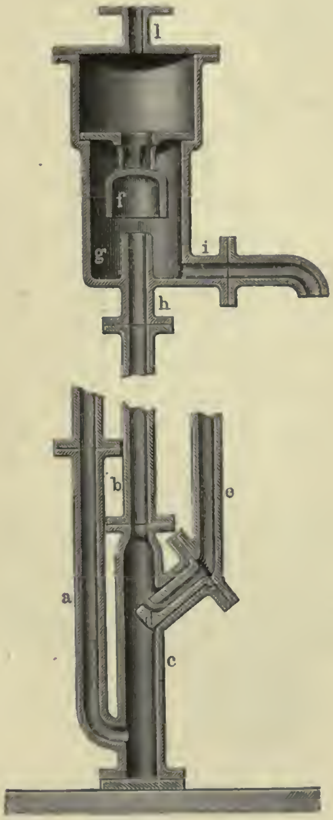

Fig. 327.—One-20th full size.

gives a section of the apparatus, and Fig. 328 the general arrangement of same. The apparatus is stated to be working satisfactorily at the gun-cotton factory at Angoulême. The principle on which it is constructed is as follows:—If a liquid be contained in a U-shaped tube, and air blown into the lowest part of the longer arm, the liquid and air form a sort of emulsion, the specific gravity of which will be lower than that of the liquid in the other arm of the tube which is not mixed with air. On account of this, the emulsioned liquid will stand at a higher level than the liquid in its natural state. If a continuous stream of liquid be run into the shorter arm of the U-tube, and at the same time a current of air be introduced into the longer arm, then a continuous stream of emulsioned liquid will rise in the longer tube, which will run out at a height corresponding to the difference of densities of the liquids in the two arms. Experience has shown that the shorter arm must be from $\frac{3}{5}$ to $\frac{1}{2}$ the length of the longer arm,—or in other words, that in order to lift the liquid to a height of 30 feet it must first drop from 15 to 18 feet in the shorter arm. The longer the falling arm, the better the apparatus works.

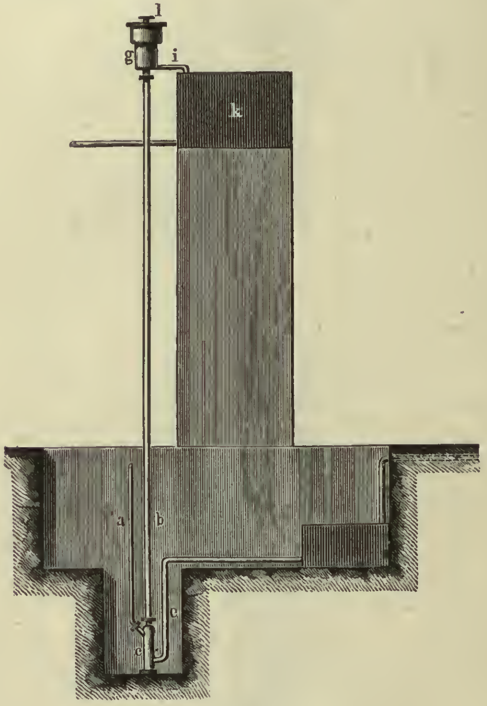

Fig. 328.—One-100th full size.

From the section of the apparatus in Fig. 327 it will be seen that the two tubes (a and b) are of equal diameter, but the lower part (c) of the longer arm is a little wider, to leave room for the admission of air. This is done by means of a nozzle (d) attached by a flange to the admission pipe for the air (e), and passing into the liquid pipe through a branch. Of course the two tubes can be taken to any height so long as the above-mentioned proportion is maintained, and in order to gain in height, the admission pipe (a) can be sunk into the ground with advantage, as will be seen from Fig. 328. In order to prevent the liquid frothing at the top of the longer tube, a discharge pipe is made on the side and an enlarged vessel is provided on the top. This consists of a bell (f) for checking the stream of liquid, placed in a wider chamber (g) so that it is lifted by any nitrous vapours formed. The vapours can then escape through a branch (l) on the cover. The liquid enters at the bottom of the surrounding pipe (g), and the admission pipe is prolonged for this purpose. The stream of liquid which is thus thrown back from the bell (f) goes round the tube (h) in the box to the discharge tube (i), and thence into the storage vessel (k).

At Angoulême air of 1½ atmospheres pressure is blown in, and the apparatus forces 800 kilogrammes of waste acid per hour, with a specific gravity of 1·6 to 1·7, to a height of 4 or 5 metres. According to a calculation of D'Alsace the apparatus has only an efficiency of 0·02 as compared with that required for the production of compressed air, but this seems so extraordinarily low, that the compressors themselves probably had a very low efficiency. At any rate, the calculation of D'Alsace shows that even with such a very low efficiency the cost of pumping the acids by means of this apparatus is considerably lower than with manual work.

To vol. ii. *p.* 52.—In the action which Mr. Alfred Nobel brought against the English Government for alleged infringement of his Ballistite patent, Sir Henry Roscoe described some very interesting experiments which he had made on the action of acids of different strengths and varying temperatures in the manufacture of soluble and insoluble gun-cotton. In the one case, he nitrated at 20° C., and did not allow the temperature to rise above 25° C., when by merely altering the strength of the acid he obtained a soluble gun-cotton containing 12·28 per cent. of nitrogen. According to his view, the difference was to be ascribed chiefly to the large quantity of sulphuric acid used in the second instance, although he used 60·6 parts for the soluble and 64·3 parts for the insoluble variety; thus there was only a difference of about 4 per cent. He also used a mixture consisting of

	PARTS.
Nitric acid mono-hydrate	25·1
Sulphuric acid mono-hydrate	60·9
Water	14
	100·0

in 30 parts of which he nitrated 1 part of cotton for two hours, at a temperature of between 20° and 25° C. He also used a second mixture, consisting of

	PARTS.
Nitric acid mono-hydrate	17·55
Sulphuric acid mono-hydrate...	77·05
Water	5·40
	100·00

In 50 parts of this acid mixture he nitrated 1 part of cotton at a temperature of 20° to 25° C. for two hours. In the first case he obtained a soluble gun-cotton with 12·73 per cent. of nitrogen, in the second case an insoluble one with 12·83 per cent.

To vol. ii. *p.* 230.—The resin used for westphalite is gum-lac.

To vol. ii. *p.* 236.—According to Macnab and Ristori, the new kind of E. C. powder consists of nitro-cellulose, barium nitrate, and a small quantity of camphor.

To vol. ii. *p.* 254.—The Cannonite Syndicate of Trimley manufacture a gun-cotton powder which, according to an analysis made by Dr. Dupré, consists of

	PARTS.
Nitro-cellulose and graphite	86·00
Potassium nitrate	6·88
Resin	6·19
Moisture	0·93
	100·00

According to a private communication, the powder is made by the aid of a solvent in the well-known manner in a Werner and Pfleiderer kneading machine, and the dough then formed into cords by a press. The die contains 1700 holes of the size of an ordinary pin, and the whole of the cords are wound on drums. After drying, the Cannonite, which is of the thickness of coarse horsehair, is first granulated by a revolving disintegrator, and then in fluted rollers, both of phosphor bronze. The pieces are transferred by hand to a sieve, which is worked by an eccentric below the disintegrator, and any pieces that pass through it are carried by buckets on an endless band to the top of the disintegrator. There they fall into another disintegrator, whence they pass between three pairs of fluted rollers, and again into the sieve at the bottom. The grains of serviceable size are polished with black-lead in a glazing drum.

To vol. ii. *p.* 257.—Mr. William Macnab, giving evidence in the patent action of Nobel *v.* Anderson, usually known as the Cordite case, stated that by allowing a mixture of insoluble gun-cotton and ether alcohol to stand at a temperature of – 50° C., and then heating it up to 60° C., he obtained a gelatinous transparent body similar to ballistite. He obtained the low temperature by means of Professor Dewar's "vacuum jacket" and liquid carbonic acid. The fact that insoluble gun-cotton can be converted into a gelatinous body by previously subjecting it to intense cold and subsequent heating was only discovered a short time before Macnab's experiments, and only known to very few, and as his observations were made quite independently, he is quite entitled to claim it as an original discovery.

To vol. ii. *p.* 262.—Another kind of amberite is said to consist of gun-cotton, barium nitrate, and solid paraffin.

To vol. ii. *p.* 266.—According to analyses of Professor Charles Munroe, Rifleite and S. R. Powder have the following compositions :—

RIFLEITE.

	PARTS.
Di-nitro-cellulose	22·48
Tri-nitro-cellulose	74·16
Phenyl-amido-azo-benzene	2·52
Graphite	traces
Volatile substances	0·84
	100·00

S. R. POWDER.

	PARTS.
Di-nitro-cellulose	28·18
Tri-nitro-cellulose	46·97
Aurin	1·06
Barium nitrate	19·97
Potassium nitrate	2·35
Volatile substances	1·45
	99·98

To vol. ii. *p.* 274.—William Macnab and E. Ristori, in a paper read before the Royal Society (' Proceedings of the Royal Society,' vol. lvi., p. 8), described exhaustive experiments which they had made to determine the heat developed by, and the quantity and composition of the products of combustion from modern smokeless powders, when exploded in a confined state. They used for this purpose a Berthelot calorimetric bomb. The results of their experiments are given in the following tables, which are taken from the ' Proceedings of the Royal Society ' :—

TABLE I.—INDICATING THE QUANTITY OF HEAT, ALSO THE VOLUME AND ANALYSIS OF THE GAS DEVELOPED PER GRAMME WITH DIFFERENT SPORTING AND MILITARY SMOKELESS POWDERS NOW IN USE.

NAME OF EXPLOSIVE.	Calories per gramme.	Permanent gases.	Aqueous vapour.	Total volume of gas, calculated at 0° and 760 mm.	PER CENT. COMPOSITION OF PERMANENT GASES.					Co-efficient of potential energy.
					CO_2	CO	CH_4	H	N	
		Cubic centimetres per gramme.	Cubic centimetres per gramme.	Cubic centimetres per gramme.						
E.C. Powder English	800	420	154	574	22·9	40·6	0·5	15·5	20·5	459
S.S. Sporting Powder	799	584	150	734	18·2	45·4	0·7	20·0	15·7	586
Troisdorf, German ...	943	700	195	895	18·7	47·9	0·8	17·4	15·2	844
Rifleite, English ...	864	766	159	925	14·2	50·1	0·3	20·5	14·9	799
B. N., French... ...	833	738	168	906	13·2	53·1	0·7	19·4	13·6	755
Cordite, English manufacture	1253	647	235	882	24·9	40·3	0·7	14·8	19·3	1105
Ballistite, German manufacture	1291	591	231	822	33·1	35·4	0·5	10·1	20·9	1061
Ballistite, Italian and Spanish manufacture	1317	581	245	826	35·9	32·6	0·3	9·0	22·2	1088

TABLE II.—INDICATING THE QUANTITY OF HEAT, ALSO THE VOLUME AND ANALYSIS OF THE GAS DEVELOPED PER GRAMME WITH NITRO-GLYCERIN, NITRO-CELLULOSE, AND WITH SEVERAL DIFFERENT COMBINATIONS OF THESE TWO EXPLOSIVES MADE AT ARDEER FACTORY.

Composition of Explosive.	Calories per gramme.	Permanent gas.	Aqueous vapour.	Total volume of gas, calculated at 0° and 760 mm.	Per Cent. Composition of Permanent Gases.						Co-efficient of potential energy.
					CO_2	CO	CH_4	O	H	N	
		Cubic centimetres per gramme.	Cubic centimetres per gramme.	Cubic centimetres per gramme.							
A. Nitro-glycerin	1652	464	257	741	63·0	—	—	4·0	—	33·0	1224
B. Nitro-cellulose (nitrogen = 13·30 per cent.)	1061	673	203	876	22·3	45·4	0·5	—	14·9	16·9	929
C. 50 per cent. nitro-cellulose (N = 12·24 per cent.) 50 per cent. nitro-glycerin	1349	568	249	817	36·5	32·5	0·2	—	8·4	22·4	1102
D. 50 per cent. nitro-cellulose (N = 13·30 per cent.) 50 per cent. nitro-glycerin	1410	550	247	797	41·8	27·5	0·0	—	6·0	24·7	1124
E. 80 per cent. nitro-cellulose (N = 12·24 per cent.) 20 per cent. nitro-glycerin	1062	675	226	901	21·7	45·4	0·1	—	15·7	17·1	957
F. 80 per cent. nitro-cellulose (N = 13·30 per cent.) 20 per cent. nitro-glycerin	1159	637	227	864	26·6	40·8	0·1	—	12·0	20·5	1001
G. 35 per cent. nitro-cellulose (N = 13·30 per cent.) 5 per cent. vaseline 60 per cent. nitro-glycerin	1280	627	236	863	26·7	39·8	0·5	—	12·8	20·2	1105

TABLE III.—SHOWING THE HEAT DEVELOPED BY EXPLOSIVES CONTAINING NITRO-GLYCERIN AND NITRO-CELLULOSE IN DIFFERENT PROPORTIONS.

Composition of Explosive.			Calories per Gramme.
Nitro-cellulose (N = 13·3 per cent.).		Nitro-glycerin.	
100 per cent. (dry pulp)		0	1061
100 ,, (gelatinized)		0	922
90 ,,		10 per cent.	1044
80 ,,		20 ,,	1159
70 ,,		30 ,,	1267
60 ,,		40 ,,	1347
50 ,,		50 ,,	1410
40 ,,		60 ,,	1467
0 ,,		100 ,,	1652
Nitro-cellulose (N = 12·24 per cent.).		Nitro-glycerin.	
80 per cent.		20 per cent.	1062
60 ,,		40 ,,	1288
50 ,,		50 ,,	1349
40 ,,		60 ,,	1405
Nitro-cellulose (N = 13·3 per cent.).	Vaseline.	Nitro-glycerin.	
55 per cent.	5 per cent.	40 per cent.	1134
35 ,,	5 ,,	60 ,,	1280

TABLE IV.—SHOWING THE HEAT DEVELOPED AND THE ANALYSIS OF THE PERMANENT GAS PRODUCED IN A CLOSED VESSEL FROM WHICH THE AIR HAS NOT BEEN EXHAUSTED, THE EXPLOSIVE BEING IN EVERY CASE BALLISTITE OF ITALIAN MANUFACTURE.

Charge.	Calories per Gramme.	Analysis of the Permanent Gas.			
		CO_2	CO	H	N
2 grammes	1587	37·0	17·6	3·2	42·2
3 ,,	1485	36·4	22·0	4·6	37·0
4 ,,	1446	36·2	24·6	6·1	33·1
5 ,,	1415	36·2	26·0	7·2	30·6
6 ,,	1380	36·3	27·0	7·9	28·6

Traces of CH_4 were found, but in this series of experiments the quantity of this gas was not determined.

TABLE V.—SHOWING THE ORIGINAL COMPOSITION AND METAMORPHOSIS OF NITRO-CELLULOSE, NITRO-GLYCERIN, AND OF SEVERAL GUNPOWDERS MADE BY COMBINATIONS OF THESE TWO EXPLOSIVES.

Nature and Description of Explosive.	Per Cent. Composition by Weight.				Per Cent. Products of Combustion by Weight.						
	Carbon C.	Oxygen O.	Hydrogen H.	Nitrogen N	Carbonic acid CO_2.	Carbonic oxide CO.	Marsh gas CH^4	Oxygen O	Hydrogen H	Nitrogen N	Water H_2O.
A. Nitro-glycerin	15·7	63·0	2·3	18·8	57·6	—	—	2·7	—	18·8	20·7
B. Nitro-cellulose (nitrogen = 13·3 per cent.)	24·58	57·68	2·73	13·6	29·27	38·52	0·24	—	0·86	13·6	16·30
C. 50 per cent. nitro-cellulose (N = 12·24 per cent.) 50 per cent. nitro-glycerin	21·15	60·67	2·67	15·58	41·0	23·10	0·08	—	0·4	15·58	20·01
D. 50 per cent. nitro-cellulose (N = 13·30 per cent.) 50 per cent. nitro-glycerin	20·47	61·23	2·49	16·35	45·3	19·0	0·00	—	0·3	16·35	19·90
E. 80 per cent. nitro-cellulose (N = 12·24 per cent.) 20 per cent. nitro-glycerin	24·37	58·98	2·98	14·0	28·9	38·4	0·05	—	1·0	14·0	18·2
F. 80 per cent. nitro-cellulose (N = 13·30 per cent.) 20 per cent. nitro-glycerin	23·11	58·98	2·71	15·84	33·4	32·6	0·04	—	0·7	15·84	18·2
G. 35 per cent. nitro-cellulose (N = 13·30 per cent.) 5 per cent. vaseline 60 per cent. nitro-glycerin	22·2	59·0	2·88	15·46	33·0	31·3	0·2	—	0·7	15·46	19·0
H. Cordite, English manufacture ...	22·91	57·72	2·95	15·19	31·76	32·68	0·32	—	0·86	15·19	18·08
K. Ballistite, Italian and Spanish manufacture	21·47	60·83	2·68	15·80	41·11	23·76	0·12	—	0·47	15·8	19·69

Further investigations into the transformation and ballistic properties of modern smokeless powders containing gun-cotton as a principal agent have been made by Sir Andrew Noble and Sir Frederick Abel, and a preliminary note on the research was published in the 'Proceedings of the Royal Society,' vol. lvi., p. 205.

The arrangements for firing the explosive, collecting the gases, determining the heat developed, and the quantity of permanent gases were the same as in their former researches (*vide* vol. i., p. 346), and in addition special means were provided for determining exactly the amount of water formed.

In their results the following points are of most interest.

High pressure does not necessarily mean detonation. Both cordite and gun-cotton can be made to develop pressures close upon 15,000 atmospheres, but the experimenters were never able to detonate cordite, whilst gun-cotton can be detonated with the utmost ease.

Two kinds of gun-cotton were used for their experiments, one in hanks or strands, and the other in the form of compressed pellets. Both contained about 4·4 per cent. of soluble and 95·6 per cent. insoluble gun-cotton, and, as used, about 2·25 per cent. of moisture.

In the present research, as in their experiments with black powder, it was also definitely proved that the carbonic anhydride in the gases increased with the pressure, whilst the carbonic oxide decreased proportionately. With gun-cotton in the form of strands the proportion between carbonic anhydride and carbonic oxide at a pressure of 1·5 tons per square inch is as 26·49 to 36·66 by volume, and at 50 tons as 36·18 to 27·57,—thus for the carbonic oxide inversely proportional to the pressure. The same is the case with gun-cotton in the form of pellets, only there is a marked difference in the respective proportions. The proportions formed of the two gases are probably connected with the rapidity of combustion of the two kinds of gun-cotton.

The quantity of permanent gases given off on explosion under a pressure of about 10 tons per square inch (reduced to 0° C. and 760 millimetres of mercury) was found to be as given in the table on next page.

The erosion produced in guns by cordite is very slightly greater than that of brown prismatic powder, if considered in relation to the energy generated by the two explosives, but whilst with brown powder large holes and rough grooves are formed in the barrel, which are apt to develop into cracks, with cordite the erosion consists simply of a washing away of the surface of the steel barrel. Ballistite behaves like cordite, but the erosive action is greater on account of the higher heat developed.

Sir Andrew Noble never succeeded in detonating cordite, nor could he do it with ballistite containing equal proportions of nitro-glycerin and gun-cotton, but with ballistite of 40 and 60 per cent. respectively in cubes of 0·2 inch sides he obtained detonation with great violence on two occasions.

Explosive.	Permanent gases. Cubic centimetres per gramme.	Water formed. Cubic centimetres.	Total volume of gases. Cubic centimetres per gramme.	Heat measured (gramme units), water fluid.	Heat measured (gramme units), water gaseous.
Strand gun-cotton	689	162·6	851·6	1068	988
Pellet gun-cotton	725	162·6	887·6	1037	957
,, ,, from Stowmarket	738	—	—	—	994
Di-nitro-cellulose with 12·8 per cent. nitrogen	748	—	—	—	977
Service cordite 0·255 inch diameter ...	692	194·0	886·0	1284	1189
,, ,, 0·048 inch diameter ...	698	192·5	890·5	1272	1178
Ballistite (41·62 per cent. nitro-glycerin; 59·05 per cent. nitro-cellulose)	610	197·0	807·0	1365	1269
French B. N. powder	616	206·0	822·0	1003	902

The paper contains a large number of analytical results, and diagrams showing velocity and pressure curves of the various powders examined. For full details the reader must be referred to the original paper, but for the sake of comparison a few results are given below.

VOLUME OF PERMANENT GASES GENERATED UNDER PRESSURE OF GAS OF TONS PER SQUARE INCH.

	Strand Gun-cotton.		Pellet Gun-cotton.		Saturated Pellet Gun-cotton.	Cordite.		Ballistite.[1]	French B. N. Powder.
	Decomposition. 8 tons.	Detonation. 3 tons.	Decomposition. 11 tons.	Detonation. 3 tons.	10 tons.	0·048 inch diameter. 10 tons.	0·255 inch diameter. 10 tons.	6 tons.	6 tons.
CO_2	30·95	29·08	26·68	25·76	32·14	32·00	27·00	37·30	28·10
CO	32·27	32·88	36·97	39·34	27·04	32·90	34·20	27·80	32·40
H	19·10	20·14	19·59	18·71	26·80	18·00	26·90	19·10	21·90
N	17·20	17·50	15·91	16·19	13·83	17·10	12·00	15·80	16·80
CH_4	0·48	0·75	0·85	nil	nil	traces	traces	traces	0·80

[1] 50 per cent. nitro-glycerin; 50 per cent. gun-cotton.

To vol. ii. *pp.* 278 and 291.—The caps for cordite rifle ammunition are charged with 0·4 grain of the following composition :—

	PARTS.
Fulminate of mercury	6
Potassium chlorate	14
Antimony sulphide	18
Mealed powder	1
Ground sulphur	1

To vol. ii. *p.* 350.—An apparatus on the same principle as that of Cogswell and Harrison is made by Messrs. Nagant of Liège. Briefly, it consists of a breech-block screwed to the barrel of a gun in which a piston moves over a perforated cartridge, as in Cogswell and Harrison's apparatus, while a copper cylinder is held up against the end of the piston by an anvil.

BIBLIOGRAPHY OF EXPLOSIVES

PRELIMINARY REMARKS

In the following pages the author has endeavoured to compile as complete an index of the literature on explosives as possible. His own collection is considerable, and in the British Museum he has been able to find at least the titles of all works which have been published since the discovery of the art of printing. Every bibliophile knows that the compilation of an index of even so restricted a subject as explosives causes great trouble, and the author hopes this will be taken as a good excuse if, as he feels is rather probable, this index does not include all works that have been published on the subject. In the interest of the cause he would therefore be greatly obliged to any one who will point out any omissions, in order that they may be made good in any future edition of this work that is called for. (Address: Oscar Guttmann, 12 Mark Lane, E.C.)

Besides the literature of explosives proper, the index contains a number of works which either relate to the history, or which have been consulted by the author. All these are marked with an asterisk. On the other hand, the 'Büchsenmeistereischule,' and other works on artillery, have only been considered in so far as they bear upon the contents of this book.

The literature of fireworks has only been taken completely for the nineteenth century.

As a rule, only the first editions of books which appeared before the beginning of the present century have been inserted, and only the newest editions of those published since. Translations have only been mentioned in exceptional cases—namely, those in which fresh contributions to the subject appeared.

Unfortunately, it has not been possible to mention the many (sometimes very important) works published in periodicals, except when they have been reprinted, because such a compilation would assume enormous proportions, and therefore the following titles of periodicals which, amongst others, contain articles on explosives from time to time are alone given.

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II. ALPHABETICAL INDEX

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