

Gic and its manufacture 1909 F.L. Nathan

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industrial plants in Universities would, he believed, rove highly costly and quite unsuccessful. Dr. R. S. MORRELL said there was one suggestion he

wished to make, and if adopted he thought it would help in clear up the difficulty of the application of University research to the wants of the manufacturer. Research night be made of more immediate use to the manufacturer i there were a closer connection between works chemists and the University. Speaking from experience, he found that there was much pure laboratory work to be done shich could be well carried out in the University. The work would be good training in research for a post-craduate student in his second year. The results would craduate student in his second year. The results would rare no direct money value—they could only be useful in the hands of a works chemist, who with his knowledge  $\epsilon$  industrial conditions could turn them into  $\mathfrak{L}$  s. d. Such a scheme of research would enable the manufacturers to encourage research scholarships and fellowships, and would eliminate the feeling that there was a danger  $\kappa'_{1}$  disclosing trade secrets by so doing. It might be .kcd, "How much of the research work done in the (riversity would be of practical use or application in this generation ?" He feared it would be far too little. He did not think that the manufacturer would ever allow the student, and much less the professor, to have the ran of the "works"; but he thought he could influence University research through the suggestions of his works chemist so that results might be obtained which would

is of value to the present generation. Mr. E. A. LEWIS was of opinion that applied chemistry could only be taught by applied chemists, and that every University should have a lecturer on the chemical and metallurgical staff with at least 10 years' works experience. Many teachers had an idea that if their students passed examinations they were competent chemists. Theoretical examinations might be necessary, but examinations in practical chemistry were a waste of time, being no test whatever of competency in applied chemistry. When ar employer engaged a young chemist he required to know if he was an accurate worker during inc whole of his college career, not during a few hours in an examination, he also wanted to know if he could think in tons. In many colleges the estimation of impurities in commercial products was never taught, and yet an employer was often more interested in the traces of impurity present than the principal constituents. The art of sampling was usually ignored altogether, the art of sampling was usually ignored altogether, although in many commercial laboratories it was as important as analysis. The granting of degrees or diplomas after a 3 years' course of training was wrong; no degree or diploma for technical chemistry or metallurgy should be given except after 5 years' works experience or for some original research on applied chemistry. He erred with Mr. Heathcote that analyses of commercial materials should be practised. A large amount of applied chemistry could only be learnt by practical experience in a works.

Mr. HEATHCOTE, in reply, said Dr. Price and Professor Frankland had touched upon the main difficulty—the question of time necessary for such a course as he suggested. His attitude was that some training in industrial chemistry was necessary, and he assumed that as his major premise. In this, pure chemistry was necessary; and after that he placed research, and then as much of the general The trend of the Chairman's remarks was to point to the lack of openings for University-trained industrial chemists. While he (the speaker) admitted the truth of all that the Chairman said, he felt it true—and a truth that should always be associated with the admitted. lack of demand—that the cure for the defect lay with industrial chemists themselves. They and they only could prove the value of their abilities and it was for them could prove the value of their abilities and it was for them to increase the availability of their information and express it in such terms and in such places that it could reach and be understood by manufacturors. If they found by experience that their training had not been quite the most suitable, it was for them to voice their requirements and hope for the sympathetic attention of the Universities and other educating institutions. He was glad that Professor Frankland had no objections

to express, other than time considerations, to the scheme outlined in the paper. Professor Frankland did not think the question of value a very difficult one. He (the speaker) had found it a very complicated one. A man (the speaker) had found it a very complicated one. A man placed in a responsible position might soon come to appreciate what "value" is, but that would be no reason for neglecting to study it, and its far-reaching issues, in the preparatory training. Economic conditions were referred to by Mr. Holcroft as the obstacle to the develop-ment of the coal tar industries here. This is generally admitted as one cause, but another and greater one was the departure of Hofmann for Germany because the salary offered him in England was not sufficient. He took it that Mr. Holcroft did not intend to suggest that economic conditions in the British Isles were sufficiently malign to render the training of industrial chemists undesirable. No doubt the conditions were temporarily detrimental in some chemical industries, but not in all. Furthermore, where capital had been extensively invested in chemical plant it became all the more desirable to employ a research chemist, because, on account of the activity among scientific workers, the rate of depreciation

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of chemical plant was apt to be very high. Mr. Tucker doubted whether theory is a labour-saving device. He (the speaker) supposed that each strongly advocated the food he was brought up on. It would be the height of folly to advocate neglect of theory in the training of industrial chemists. Theory was the off-spring of practice. Practice comes first, and when sufficiently grown and extensive to show the rules and regularities running through it, "Theory" follows— as a rule—and probes beneath the skin of fact to get to the foundation and extensive rules and light to get as a rule—and probes beneath the skin of fact to get to the foundations and structures upon which facts or phenomena are superposed. He believed that in attempts to initiate new practices the first thing was to ensure sound foundations, and theory was needful for this. At the same time the "ounce of practice" which knows nothing of theory ought to be used, for it often embeddied wide and long experience. He heartily economic with wide and long experience. He heartily concurred with the suggestion of Dr. Morrell. University professors certainly had the precedent of inanimate naturo when they, like the trees of yore which buried the results of their chemical activities in the earth, expended their energies now for application many centuries hence. It cannot be said, however, that this post-dating cf the draft commends itself to one's common sense. Let men who excel only in this kind of work do it by all means, but do not encourage them to engage the youthful, eager, and adaptable minds of the students in like pursuits. In conclusion, he would like to say that there must be

a considerable subconscious demand for applied chemists in the engineering trades which bulk so large in and around Birmingham. If one cycle firm could employ a staff of eight in their research laboratories and keep them continually busy, there must be other industries, not usually included under the heading of chemical industries, in which chemists could do valuable work.

# London Section.

Meeting held at Burlington House on Monday, February 1st, 1909.

DR. J. LEWKOWITSCH IN THE CHAIR.

### GUNCOTTON AND ITS MANUFACTURE.

### BY COL. SIR FREDERIC L. NATHAN, R.A.

Guncotton was discovored early in 1846 by Christian Friedrich Schönbein, at Basle. It was not until forty years later that it fulfilled his expectations, referred to in a letter from him to Faraday, dated the 25th August, 1846, "of becoming a dangerous rival to gunpowder," as regards its use as a propulsive explosive. Schönbein came to England in August, 1846, and was present at Woolwich on the 9th October of that year when some

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successful experiments were made with his guncotton, and immediately afterwards, he, together with Böttger, who independently discovered guncotton shortly after Schönbein, entered into negotiations with Messrs. John Hall and Son, of Faversham, for the manufacture and sale of guncotton in England. This firm erectéd a guncotton factory adjacent to their gunpowder works, but a very disastrous explosion on the 14th July, 1847, hereaf the fectory, which was never rebuilt.

but a very disastrous explosion on the 14th July, 1847, destroyed the factory, which was never rebuilt. The early history of the manufacture of guncotton abroad, and of the attempts made, particularly in Austria, to adapt it for propulsive purposes in small arms and cannon, have been so fully described by S. J. von Romocki in Vol. II. of his "Geschichte der Explosivstoffe," by Dr. R. Escales in "Die Schiessbaumwolle," and by others, that it is not proposed to deal with these matters in this paper. It is only necessary to remark that a factory paper. It is only necessary to remark that a factory was erected at Hirtenberg in Austria, where the production of guncotton on a manufacturing scale was worked out by Baron von Lenk, of the Austrian Artillery. As the general principles he elaborated with such care and perseverance are in the main those still in use, it is of interest to give briefly the general lines of his processes of manufacture. As his raw material, von Lonk employed hanks of loose cotton yarn, purified by boiling for a few hanks of loose cotton yarn, purified by boiling for a few minutes in a weak solution of potassium carbonate, centrifugaling out the liquid, and then thoroughly washing the yarn in cold water. The cleansed yarn was dried in a hot air chamber; when dry, it was placed in airtight receptacles to cool; when cool, it was immediately dipped in the nitrating acid. The nitrating acid consisted of one part by weight of nitric acid of 1.52 specific gravity, and 3 parts by weight of sulphuric acid of 1.84 specific gravity, thoroughly mixed together and cooled. The and 3 parts by weight of sulphuric acid of 1.84 specific gravity, thoroughly mixed together and cooled. The dipping pans containing the nitrating acid were of cast. iron and stood in troughs surrounded by cold water. About 6 oz. of cotton, or two skeins, were immersed in the acid at a time, and moved about in it for a few minutes, until the action actions the action was then were the until thoroughly saturated, the cotton was then removed by means of an iron fork, placed on an iron grating above the pan, and some of the excess acid got rid of by gentle the pan, and some of the excess acid got rid of by gentle squeezing, the proportion of acid retained being about 10.5 parts by weight to 1 part by weight of the original cotton. The acid bath was made up by the addition of fresh acid after each removal. Six to eight skeins were next introduced into a stoneware pot, fitted with a lid, in which they remained for 48 hours. The pots were placed in cold water to keep their contents cool. After the 48 hours digestion the contents of the pots were emptied into a centrifugal machine and as much of the excess acid as possible extracted. On removal from the centrifugal machine, the hanks were immersed as suddenly and completely as possibly in a cascade of water, washed until no longer acid to the taste, and afterwards placed in crates or perforated boxes and kept in flowing water for about three weeks. At the expiration of that time the skeins were centrifugaled to remove the bulk of the water, and boiled for 15 minutes in a solution of potassium carbonate of 1.02 sp. gr. The alkaline liquid was removed, and the skeins again immersed in flowing water for a few davs.

Manufacture proceeded at Hirtenberg without serious accident until the 30th July, 1862. On this day the guncotton in the magazines exploded, owing to spontaneous decomposition, and another serious explosion occurring on the 11th October, 1865, the manufacture of guncotton was officially prohibited in Austria. Early in 1863, by the desire of the Secretary of State for War, Sir Frederick Abel took up the study of guncotton

Early in 1863, by the desire of the Secretary of State for War, Sir Frederick Abel took up the study of guncotton and its manufacture in this country. In his first memoir on "Researches on Guncotton," read before the Royal Society on the 19th April, 1866, Abel doscribed the important steps in von Lenk's system of manufacture, mainly in connection with the production of a stable product. He was of opinion that the alkaline boiling was the most important part of the purification process, and that its function was not so much to neutralise and remove residual traces of the nitrating acid, as to dissolve unstable bodies produced by the action of the nitrating acid on small quantities of resinous and other foreign substances still retained by the tubular fibre.

The important modification introduced by Abel in the manufacture of guncotton and patented by him in 18%. (Eng. Pat. 1102), had for its object the reduction of guncotton to a pulp, and forming from this pulp grain suitable for propulsive purposes. But the "pulping" process had, he considered, a further advantage, which was described by him in the following words :----" As the difficulties attending the perfect removal of the acid with which the guncotton remains impregnated after its conversion are mainly attributable to the tubular structure of the cotton fibre, and to the circumstance that the latter contracts considerably upon conversion into pyroxylin, the complete purification of the material is very greatly facilitated by reducing the guncotton fibre to a fine state of division, similar to that of the pulp used in paper manufacture, in which form it appears likely that guncotton will receive advantageous application. By submission to the 'pulping' process, the guncotton is divided into very minute fragments, and it is at the same time violently agitated for some considerable time with a very large volume of water (rendered slightly alkaline if necessary), which is afterwards thoroughly expressed when the pulp is converted into cylinders or other form, so that a more searching process of purification can scarcely be conceived than this disintegration of the guncotton."

It is very probable, in view of the system of unification, consisting mainly of prolonged washing in cold running water, in use at the time of Abel's introduction of the pulping process, that his treatment did produce a much purer guncotton than was formerly obtained, but recent work on the subject of the purification of guncotton has demonstrated that other points besides the complete removal of traces of the nitrating acids underlie the chemistry of the purification process. This important question will be dealt with later.

question will be dealt with later. Although the pulping and granulating of guncotton did not succeed in transforming it into a propulsive explosive, the greater purity of the material resulting from the pulping process, re-established confidence in guncotton for other military purposes, such as for mines, torpedoes, and field service work, as well as for blasting purposes.

Messrs. Prentice and Co., of Stowmarket, had commenced the manufacture of guncotton according to von Lenk's system in 1863, but a serious accident occurred at this factory not long after starting manufacture. In 1865 they rebuilt and enlarged their factory and recommenced the manufacture of guncotton, introducing Abel's improvements. The Stowmarket factory now belongs to the New Explosives Company, Ltd., and is still making guncotton by the Abel process. The manufacture of guncotton was commenced in 1863 at Waltham Abbey on a small scale under Abel's direction; it was not until 1872 that a factory capable of turning out about 250 tons of guncotton per annum

The manufacture of guncotton was commenced in 1863 at Waltham Abbey on a small scale under Abel's direction; it was not until 1872 that a factory capable of turning out about 250 tons of guncotton per annum was established there. The main portion of this factory consisted of old buildings which had formed part of the Saltpetre Refinery, and abutted on the principal street of the town. Fresh land, away from the town, was acquired in 1885, and a guncotton factory was built on it, and commenced work in 1890. It was considerably enlarged and altered in 1904-5, and is now capable of producing about 2000 tons of guncotton per annum by the displacement dipping process of manufacture, to be reforred to later.

It has already been stated that the invention of Abel had for its object the production of guncotton in a form in which it was hoped it would be suitable for use as a propellant. To attain this object, he reduced the finished guncotton to a very fine state of division. It was subsequently found that guncotton in this condition was more readily purified than when it had to be kept in the form of skeins throughout all the processes of manufacture. Abel's process had other advantages, however. As the guncotton in its final form was reduced to a pulp, it was no longer necessary to use expensive long stapled cotton wall instead of this material, waste from the cotton suitable machinery, opened out, and in this condition it was more completely exposed to the action of the acids than were the skeins. Again, the guncotton pulp could,

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iv- hydraulic pressure, be compressed into dense and compact masses of any desired shape, making it much more suitable for use in mines, torpedoes, and for blasting perposes, and in this condition it could be stored wet ic: indefinite periods. This form of compressed guncotton was rendered more valuable still when it was discovered in 1869 by Mr. E. O: Brown, of the Chemical Department, Reval Arsenal, that it could be fully detonated in its wet condition and unconfined, by fulminate of mercury. Guncotton compressed into slabs and discs is still the

Guncotton compressed into slabs and discs is still the ferm of guncotton used for mines, for torpedoes, and in cennection with demolition work in the field. The recesses by which these slabs and discs are produced will re dealt with later.

The von Lenk process of manufacture, as modified and improved by Abel, is universally employed at the present car, except as regards the actual process of nitration. The modifications this process has undergone will be accribed when dealing with this particular operation.

Before considering the actual processes of manufacture, it is desirable to say a few words about the raw materials. The base of guncotton is cellulose. The purcr the cellulose, the pure the guncotton and the better the yield, other transpire the guncotton and the better the yield, other transpire gund. There is little doubt that the skeins ci long stapled yarn used by von Lenk were of great prity, and were free from foreign substances generally. A: the same time, the fact that the skein condition was maintained in the finished guncotton made it more difficult to free it from the subsidiary products of the nitration process. This was due to the tubular structure of the cotton cellulose rendering it impossible for the purification process to remove the impurities enclosed in the long fibres. When the fibres are cut into extremely short lengths in the pulping process, this difficulty is in a great measure overcome. Cotton waste in its raw state from the mills is anything but pure; but a special industry sprang up for ine purpose of converting the ordinary mill wasto into a condition suitable for guncotton making. The processes it undergoes are : de-greasing by means of solvents, treatment with caustic soda, bleaching, and washing. The effect of these treatments, if properly carried out, is to produce a fairly pure resistant cellulose, and they have now been perfected to such an extent that there is no difficulty in obtaining cotton waste containing on an average not more than 0.2 to 0.3 per ceut. of matter soluble in ether, and having very little altered cellulose of the nature of oxy- or hydro-cellulose. Attempts have been made to find substitutes for cotton waste equally suitable, and, if possible, less expensive. This is a matter which has received some considerable attention at the Royal Gunpowder Factory. During the last few years various materials have been investigated, and guncotton made from them on a manufacturing scale. The materials so far tested may be divided into three groups :- 1. Ordinary cotton waste and weaving mill waste. 2. Short fibre from the cotton seed. 3. Fibrous materials other than cotton cellulose. As the results of experimental work the advantages and disadvantages of the three groups may be summarised as follows :-

1. Ordinary collon waste from different sources varies considerably in character, and this is not without effect on the resulting guncotton. Its chief disadvantage, however, is that it generally contains considerable quantitics of wood and other foreign bodies. All these substances are possible sources of regions of low stability in the guncotton. On the other hand it is plentiful, easily procured, and reasonably cheap, and it can be relied upon to produce a satisfactory guncotton as regards nitrogencontent and percentage of soluble in ether-alcohol.

Weaving mill waste is a material composed entirely of woven cotton fabric of various natures, partly broken down by mechanical means. It differs materially in character, and, as at present in the market, is liable to contain, mixed together, both soft material and hard, stiff, starchy rags. It may also be over-bleached, and contain an undue proportion of altered cellulose. In its best form it is pure cotton cellulose, remarkably free from foreign matters, and makes excellent guncotton. It is, however, apparently not procurable at present, in its best form, in anything like sufficient quantity, and its price would probably be considerably higher than the normal price of ordinary cotton waste.

2. The short fibre from the cotton seed, several samples of which, from different sources, have been experimented with, would no doubt make a good guncotton, if it could be obtained freed from particles of the seed husk. Unfortunately it does not appear possible to remove the husk particles entirely by mechanical means, and the fibre is therefore subjected to a very drastic chemical treatment, which, whilst it does not remove all the husk, produces non-resistant cellulose to a considerable extent, and the final product, in consequence, gives a guncotton low in nitrogen, and with a high percentage of soluble in etheralcohol.

3. Certain fibrous materials which, when submitted to the chlorination method of Cross and Bevan for the estimation of cellulose, were found to give a high result. were selected for experiment. Amongst these were Ramie fibre, Marsdenia tenacissima, to be found in certain parts of India, and Asclepias semilunata, which comes from Uganda. These materials contain ligneous matter, which, in the case of Ramie, is present not as an incrus-tation, but forming a compound cellulose with the more resistant material, and difficult to remove completely on a manufacturing scale. Even when this ligneous material has been got rid of to a considerable extent, the fibre requires a much longer nitration and boiling to produce a correct and stable guncotton. Its price also is very high. Marsdenia tenacissima, by a simple treatment, easily carried out on the manufacturing scale, can be made into a fairly pure resistant cellulose, giving on nitration a distinctly promising guncotton. At present. nitration a distinctly promising guncotton. At present, however, it is not procurable in anything like sufficient quantity, and its price is unknown. Asclepias semilunata can also, by a simple process of purification, be reduced to a very highly resistant normal cellulose, capable, by slightly altering the nitration process, of being converted into a guncotton similar in its properties to that prepared from cotton cellulose. So far it has not been possible to obtain this material in quantity, and its price is also unknown.

So far, therefore, the best material which has come to light for the manufacture of guncotton is weaving mill waste, if the best form could be procured in sufficient quantity, at a reasonable price. Failing weaving mill waste, cotton waste as used since the early days of guncotton manufacture still remains the best commercial form of raw material.

The nitric and sulphuric acids universally employed are commercial products of a fairly high standard of purity. It is essential that they should not contain solid impurities which might adhere to the guncotton, and which would certainly interfere with the recovery of the waste acid. Of recent years it has been possible to obtain Nordhausen sulphuric acid at a fairly reasonable price, and its use has enabled a larger proportion of waste acid to be revivified than was possible when ordinary sulphuric acid only was available. It is a common practice now for explosives factories to manufacture their own pitric acid, and to revivify and recover their waste acids, and most of them possess installations of considerable size for carrying out these processes, and for mixing the two acids on a large scale. These matters hardly come within the scope of the present paper, however, even if time permitted. Before these installations existed, it was the practice to buy acids of a certain strength and mix them in the proportion of 3 parts by weight of sulphuric acid to 1 part by weight of nitric acid; now, the more correct system is being adopted of mixing large quantities, analysing the mixture, and adjusting the composition of the mixed acid by the addition of nitric or sulphuric acid, so as to obtain the correct percentage composition. The nitrating acid is of course made up of a cartain properties of wester acid is, of course, made up of a certain proportion of waste acid to which new nitric acid and new sulphuric acid are added.

The manufacture of guncotton comprises the mechanical processes in connection with the preparation of the cotton waste, the chemical processes of nitration and purification, and the final mechanical operations of pulping and washing.

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# TEASING.

Cotton waste as supplied to the factory contains a notable percentage of hygroscopic moisture, also wood, pieces of iron, metal, string, rubber, etc. These impurities are removed as far as possible by hand picking, and the cotton is then passed through what is known as the teasing machine, which, by means of a combination of rollers, armed with iron teeth, separates the fibre of the cotton and opens out knots and lumps. As the cotton leaves the machine, it is picked over again. It was formerly the practice to cut the teased cotton in a description of guillotine into 3 inch squares. This operation was one of the features of the original Abel process, but it has been disused of late years at Waltham Abbey, because it was found that the cotton along the cut edges was felted together to such an extent as to resist the action of the autirating acid.

### DRYING.

At Waltham Abbey the cotton, as it leaves the teasing machine, is delivered on to an endless band, which carries it to the drying machine. The moisture is expelled from the cotton by a blast of hot air, supplied by a fan through a steam heater. The cotton waste passes very slowly through the machine, the operation lasting about threequarters of an hour, and issues from it containing about half a per cent. of moisture. It is immediately weighed out into charges and placed in sheet iron boxes or other suitable receptacles with lids, to cool, for which a period of about 8 or 9 hours is sufficient. During the cooling the cotton waste re-absorbs about half a per cent of moisture. The object of removing the moisture from the cotton waste is to prevent dilution of the nitrating acid. The above is a typical method of drying, but it is obvious that this simple operation can be effected in other ways.

# NITRATION.

In von Lenk's system of manufacture the operation of nitration was carried out in two stages. The first stage was the dipping of the cotton in mixed acid contained in nitrating pans; and the second, the completion of the nitration by allowing the partially nitrated cotton to remain in contact with a weaker acid, in earthware pots, for a prolonged time. The loss of guncotton caused by occasional decompositions in the digesting pots and the labour entailed in handling them were undoubted disadvantages, and led some manufacturers to revert to the even older method of completing the operation in one stage. In this system, which has been called the direct dipping process, the size of the nitrating pans was reduced to enable them to be lifted on to trucks, wheeled to the acid centrifugals and their contents tipped into them. From this process it was but a short step to a method

From this process it was but a short step to a method which has been adopted to a large extent on the Continent, and known as the nitrating centrifugal method of nitration. Instead of nitrating in pans and tipping into the centrifugal machine, the operations of nitration, digestion, and removal of the acid are all carried out in the centrifugal machine itself. In this way labour is economised and it is claimed that fewer decompositions occur and that therefore a higher yield is obtained.

It will be seen that the improvements in methods of nitration so far enumerated are of the nature of modifications of the original process such as characterise the gradual evolution of almost every manufacturing operation. The next noteworthy improvement, however, marks an entirely new departure. The inventors of the displacement system of nitration, Messrs. J. M. Thomson and W. T. Thomson, of the Royal Gunpowder Factory, Waltham Abbey, have discarded the older methods entirely, and introduced a system founded on new principles.

Before describing the displacement method, it is necessary to give a somewhat detailed account of the various processes preceding it, referred to above. It is not, however, necessary to refer to details of the earlier nitration processes, and the method of nitration used by von Lenk has been sufficiently described. The Abel nitration process, which followed very closely on the lines of von Lenk's process, was the one universally employed from about the year 1865 until comparatively modern times, and as it is still in use in many factories both in this country and abroad, it will be described in detail as formerin carried out at Waltham Abbey.

The nitrating acid was composed of three parts of sulphuric acid of 96 per cent. mono-hydrate to one pan of nitric acid of 91 per cent. mono-hydrate, thoroughly mixed and cooled. This acid was run from the storr tanks into cast iron dipping pans, holding about 220 lb. tanks into cast iron dipping pans, noting about 120 in, each, the pans being supported in an iron tank through which cold water circulated, to keep the temperature below 70° F. The dipping pans were provided at the back with gratings, on which to press out some of the acid from the charge. The charge of cotton waste weighed 1 lb 4 or and on removal from the cooling hor was 1 lb. 4 oz., and on removal from the cooling box was passed from the back through an earthenware pite in the partition running along the back of the pans, and raked by a dipper, as rapidly as possible, into the acid. After remaining in the acid bath for about eight minutes. the cotton was removed to the grating and a portion of the acid squeezed out by means of an iron lever having an iron plate attached to one end. After a charge had been removed from the dipping pan about  $13\frac{3}{4}$  lb. of the mixed acid was run into it to replace the amount removed with the charge. The charge, now weighing with the adhering acid about 15 lb., was placed in an earthenware par provided with a cover and transferred to the cooling pitthrough which a stream of cold water flowed, and where it remained for 12 hours. During this period of digestion the conversion of the cotton into guncotton was completed. The contents of the pots were now emptied into a centrifugal wringing machine, and the bulk of the waste acid extracted. The guncotton was then removed from the centrifugal machine and placed in galvanised iron pars with long bandles. These pans, when filled, were carried quickly across to the immersing tank and the guncotton thrown into a large bulk of water, the workmen standing by the tank and pushing the guncotton at once under the water with a stout wooden paddle. The immersing had to be done as quickly as possible, as, if the guncotton wer: allowed to come gradually in contact with water, it was liable to fume off. The immersing tank was fitted with a perforated copper plate, to allow the water to overflow, so that fresh water was constantly passing through the tank. The guncotton was kept well stirred by means of a wooden paddle. When 2 cwt, had been intersed, the inflow of water was stopped and the tank drained down. When all the water had been run off the tank was filled up again with fresh water. This was repeated six times or until the guncotton no longer tasted acid. When this stage had been reached the guncotton was wrung in a centrifugal machine, water from a hosopipe being turned on the guncotton for one minute during the wringing, and it was then ready for boiling.

It was then ready for boiling. This process, although it undoubtedly produced a good guncotton, had certain disadvantages, and the amount of labour required was very great. The plant, although individual items were not expensive, very rapidly deteriorated, and the cost of renewals and replacements was heavy. Power was required to drive the centrifugal machines, large quantities of water were used both for cooling and immersing, and decompositions, both in the pans, pots, and acid centrifugals, were by no means an infrequent occurrence.

### DIRECT DIPPING.

The first attempt attended with any success to overcome some of the disadvantages of the Abel process was the introduction of what is known as the direct dipping process. This system was used on a large scale at Nobel's factory at Ardeer, in Scotland, and I am indebted to the kindness of Mr. Lundholm for a description of it, of which the following is an abstract.

of it, of which the following is an abstract :--The installation consists of parallel double rows of long iron tanks known as "coolers." Iron pots termed "dippers" in which nitration is carried out stand in the coolers, 62 to each cooler. Sliding wooden covers rest on the coolers to guide the fumes from the dippers into earthenware pipes with oponings at intervals, through which they are drawn by exhaust fans. The mixed acid, either cooled or warmed as necessary, is carried by lead pipes placed

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between each row of coolers, and is supplied to the dippers through earthenware cocks at intervals.

Nuration.—The water in the coolers is kept at  $15^{\circ}$  C. The dippers having been placed in position in the coolers, are each filled with 127 lb. of mixed acid by measurement, from the acid taps,  $4\frac{1}{2}$  lb. of cotton waste are steered in each dipper. To minimise decompositions each charge of cotton wasto is added in about 10 instalments. The wooden covers are only removed to allow steeping to ie done, and are then at once replaced. The temperatures of nitration are :—Initial temperature of mixed acid.  $15^{\circ}$  C.; maximum after steeping,  $25^{\circ}$  C.; temperature at end of nitration,  $29^{\circ}$  C. The duration of the nitration varies according to the output required from the plat. One, two, or three shifts may be worked per 24 rours, and the time of nitration may therefore be 24, 12, et 8 hours respectively.

The average composition of the mixed acid for a 12 hours immersion is as follows:—Sulphuric acid, 75-0 per cent.; nitric acid, 15-75 per cent.; nitrous acid, 1-30 per cent.; water, 7-95 per cent. For an eight hours immersion a higher percentage of nitric acid and less water is used; for a 24 hours immersion less nitric and more water. The average composition of the waste acid for a 12 hours immersion is:—Sulphuric acid, 77-8 per cent.; nitric acid, 11-0 per cent.; nitrous acid, 1-5 per cent.; water, 9-7 per cent.

cent.; water, 9.7 per cent. Recovering the waste acid.—When the nitration is complete, the "dippers," covered with light aluminium lids, are placed on barrows, wheeled to the centrifugals, situated at tho end of the "coolers." and the whole contents tilted out into the centrifugal. Four dippers are loaded into each centrifugal, and the guncotton having been uniformly spread round the basket, the centrifugal is run for six minutes, to remove waste acid. At the end of that time about 1 lb. of waste acid is still adhering to each pound of guncotton. The centrifugal in any way, so that as little resistance as possible may be offered when there is a decomposition. This is the usual arrangement in the case of acid centrifugals. The cone of the centrifugal projects through a circular opening in the centre of the lid and is covered by a small loose aluminium box. Small holes are cut in the sides of this box, and are of service in warning the workmen when there is a decomposition, reference the composition, there first.

in warning the workmen when there is a decomposition, as fumes are generally seen to issue there first. Drowning the guncotton.—When the waste acid has been removed, the guncotton is quickly lifted out of the centrifugals and thrown under the revolving paddles of the drowning tanks, which immediately immerse it. The men who do the discharging are provided with rubber gloves and wear thick flannel hoods, which completely cover the head, arms, and breast. The hoods are fitted with strong glass windows, and are connected by light rubber tubing to a supply of pure compressed air.

Preventing.—After a given quantity of guncotton has been drowned, the water in the tanks is run off and the guncotton thrown on to draining tables forming part of the drowning tank. It is then loaded into the prevashing centrifugals, the acid water wrung out, and washed for a few minutes with cold water from a hose, to remove adhering acid. No special precautions, however, are taken to remove all acid at this stage. The bulk of the water having been removed, the guncotton is loaded from the centrifugals into bogies, and conveyed to the boiling house.

to the boiling house. The 62 dippers in each cooler form a "charge." Eight charges are worked by each shift. The yield is 159 per cent. of dry guncotton on the dry carded cotton. The output per shift consisting of 17 men is, therefore :—  $4.5 \times 159 \times 62 \times 8 \div 100 = 3549$  lb.

### NITRATING CENTRIFUGALS.

The next attempt at simplifying the Abel process was one in which the nitration was effected in the acid centrifugal. A number of nitrating centrifugals have been patented, particularly in Germany, but the best known patterns are those of Messrs. Solwig and Lange, of Brunswick.

The latest pattern is known as the "Nitrating centrifugal with acid circulation." It consists of the usual

outer casing with cover, and an under-driven rotating basket perforated with a number of holes. The machine is provided with a hinged cover with communication to an exhaust fan, and there are pipes with cocks suitably arranged for running in the nitrating acid and drawing off the waste acid. The method of working is briefly as follows :--The basket is rotated slowly, and the nitrating acid run into it and between it and the iron casing, up to about the rim. The cotton waste is introduced in small quantities at a time, and this may be done whilst the nitrating acid is running in. During nitration the basket is rotated at the rate of 20 to 30 revolutions a minute. The effect of this rotation is to cause the nitrating acid to circulate continuously through the cotton waste. On completion of the nitration the bulk of the waste acid is drawn off and the centrifugal set into rapid motion to get rid of as much more of the waste acid as possible.

According to Selwig and Lange's circular their centrifugals are now made in two sizes. The larger size nitrates 22 to  $26\frac{1}{2}$  lb. of cotton waste, the smaller  $14\frac{1}{2}$  to  $17\frac{1}{2}$  lb., respectively. The yield is stated to be 160 per cent. The time of a complete nitrating operation is an hour.

respectively. The yield is stated to be 160 per cent. The time of a complete nitrating operation is an hour. Messrs. Curtis's and Harvey have an installation of these centrifugals at their Dartford Works; they are of the earlier or "without acid circulation" type, and of the smaller size. Mr. MacDonald has kindly supplied me with some details in connection with their working. The charge is 17½ lb. of cotton waste, the proportion of nitrating acid to cotton waste is 50 to 1. and its average

The charge is  $17\frac{1}{2}$  lb. of cotton waste, the proportion of nitrating acid to cotton waste is 50 to 1, and its average percentage composition: nitric acid,  $23\cdot15$ ; sulphuric acid,  $69\cdot35$ ; water, 7.5. The nitrating operation for the production of cordite guncotton, from the running in of the nitrating acid to the removal of the guncotton, takes about an hour. The initial temperature is  $15^{\circ}$  C., the final  $23^{\circ}$  C. After extraction of the waste acid, the guncotton retains approximately its own weight of waste acid containing a fairly high percentage of nitrio acid, which is lost in the immersing. Annlyses of the waste acid made at Dartford, show the following mean alteration in the composition of the nitrating acid, viz., a loss of  $1\cdot70$  in nitric acid, and gain of 0.91 in sulphuric, and  $0\cdot76$  in water.

### DISPLACEMENT PROCESS.

Guncotton has been made at Waltham Abbey by the displacement process since August, 1905. The installation consists of a number of units of 4 pans worked together. The pans are of earthenware and circular, 3 ft. 6 in. in diameter, and 10 in. deep at the side of the pan; tho bottom has a fall of 2 in. to the outlet, which is threequarters of an inch in diameter; they are supported on earthenware pedestals about 1 ft. 10 in. above the floor level. The 4 pans are connected together by lead pipes, and these are again connected to the nitrating acid supply pipe, to the strong and weak waste acid pipes, and to a waste water pipe, through a gauge-box, where the rate of flow is determined whilst the waste acids are being run off. Gravities of the acids are also taken in this box. The process proceeds as follows:--

A small perforated plate is placed over the outlet of each pan, and four perforated segment plates making a complete disc about one inch less than the inside diameter of the pan, are placed on the bottom. Aluminium fume hoods, which are connected to an exhaust fan, having been placed on the four pans, the stoneware cock on the acid supply pipe is opened, and the acid allowed to rise in the pans to the proper level. The nitrating acid is cooled in summer and warmed in winter, so as to maintain the same temporature of final nitration all the year round. The composition of the nitrating acid is 70½ per cent. sulphuric acid, 21 per cent. nitric acid, 0.6 per cent. nitrous acid, and 7.9 per cont. water; the quantity in each pan above the bottom plates is 600 lb., and below the plates is an additional 50 lb. A charge of 20 lb. of cotton waste is then immersed in the acid, handful by handful, aluminium dipping-forks being used for the purpose. When all the cotton waste has been pushed under the surface of the acid, perforated plates in segments are placed on the top of it, care being taken that all cotton waste is below the surface of the acid,

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and a film of water at a temperature from 5° to 8° C. is run very gradually on the surface of the plates through The following table gives the principal figures in connection with the four nitration processes described :--

	Nature of dipping vessel.	Acids.						Acid	_	Yield Output
Process.		Analysis. Per cent.					Cotton	used per lb. of cotton	Time of nitra-	on per dry man cotton per
		Sulphuric acid.	Nitric acid.	Nitrous acid.	Water.	Quantity. lb.	used. lb.	waste. lb.	tion. Hrs.	waste. week.
	Quet leas sea and		i							i
ABEL	Cast iron pan and earthenware pot	74.00	18.00	0.60	7.40	13.75	11	11.0	12	163-75 458
ARDEER: Direct dipping . DARTFORD:	Cast-iron pot	75.00	15.75	1.30	7.95	127	41	. 28-2	12	159-00 1112
Nitrating centri- fugal	Centrifugal machine	69.35	23.15	_	7.50	800-1100	16-24	· 50·0	1	160.00 -
WALTHAM ABBEY: Displacement	Earthenware pan	70-50	21.00	0.60	7-90	650	20	32.5	23	170 1742

a distributor. The film of water prevents the escape of acid fumes and the fume hoods are then removed. The time required for dipping a charge is a quarter of an hour.

The nitration is allowed to proceed for  $2\frac{1}{2}$  hours. At the expiration of this period the cock leading to the gauge-box is opened, and the waste acid allowed to run off at the rate of about 17 lb. a minute. Water, cooled, if necessary, is run on the top of the perforated plates, through the distributor, at an equivalent rate. The major portion, amounting to about 80 per cent. of the total waste acid, is returned to the acid store tanks to be revivified with Nordhausen sulphuric and new nitric acids. The composition of this waste acid is 72·70 per cent. sulphuric acid, 17·30 per cent. mitric acid, 0·65 per cent. nitrous acid, and 9·35 per cent. water. The remaining 20 per cent. of the waste acid is sent to the acid concentration factory for denitration and concentration. The quantity of acid thus dealt with amounts to about 4 lb. for every pound of guncotton. Its composition is 61·0 per cent. nitrous acid, and 21·10 per cent. water. A small proportion of the water which follows the recoverable waste acid is slightly acid to the cretent of 0·1 lb. for every pound of guncotton made. This is the total quantity of acid that is lost during the process. In the direct dipping and nitrating centrifugal processes the quantity of waste acid left in the guncotton is at least equal to the weight of the guncotton.

The whole of the acid is displaced in three hours, and the water, which should fill the pan, is run through the guncotton, the guncotton drained down and sent over to be boiled. These operations occupy about an hour. The following are the principal advantages which the displacement process possesses over the Abel process, and over the direct dipping and nitrating centrifugal processes where they are similar to the Abel process.

where they are similar to the Abel process. 1. The displacement process takes the place of the processes of dipping, squeezing out excess acid, digesting in pots, acid centrifugaling, immersing, and water centrifugaling.

fugaling. 2. The actual dipping of the cotton waste is a very much less laborious operation—the heavy labour of squeezing out the excess acid is done away with, the absence of fumes makes the work much healthier, and injuries to workmen from acid splashes are almost unknown.

3. Loss of guncotton due to decomposition in the digesting pots and acid centrifugals, and consequent inconvenience and danger to workmen from nitrous fumes, are done away with, and the heavy loss from breakages of pots and lids is saved. Three and a half years' experience has proved that the earthenware pars are very lasting.

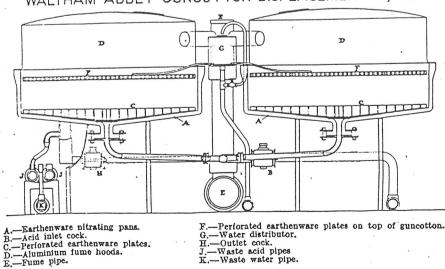
lasting. 4. Fumes during dipping, loading, and unloading acid centrifugals and immersing, are avoided.

centrifugals and immersing, are avoided. 5. The quantity of acid lost is very much reduced. This reduction means also very much less pollution of the escaping washing water.

6. The recovered waste acid is very much cleaner, a matter of the greatest importance from the point of view of revivilication and concentration.

7. The mechanical loss of guncotton in the acid and water centrifugaling processes, and in the immersing process, is saved.

8. A more thorough preliminary washing of the guncotton is obtained with an expenditure of about one-fifth



WALTHAM ABBEY GUNCOTTON DISPLACEMENT PLANT

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of the quantity of the water, and less boiling, with consequent consumption of steam, is required in order to reach a given standard of purity.

9. Great saving in power is gained by the abolition of the acid and water centrifugals, and in the reduction in the quantity of water which has to be pumped.

10. Renewals of plant, and repairs to plant and buildings are exceedingly low.

11. The number of hands employed for any given output is much less—the total cost of labour being reduced by two-thirds.

12. The yield is improved ; it averages 170 per cent.

13. Finally, a more stable guncotton, of more uniform composition, is produced. It 'is also far cleaner, and contains notably less mineral matter.

#### STABILISATION.

Boiling.—Originally stabilisation was effected by prolonged washing in cold running water followed by a very short treatment with a boiling alkaline solution. Boiling, as now understood, did not form part of the process of guncotton manufacture when manufacture was started at Waltham Abbey early in 1872. About the middle of 1873, however, boiling vats were put up at Waltham Abbey, but no records exist, unfortunately, about the details of the early boiling processes. In the official "Notes on Gunpowder and Guncotton," published by the War Office in 1878, it is stated that guncotton manufactured at Waltham Abbey underwent two boilings by steam in wooden vats for 8 hours each, the water being extracted after each boiling by wringing for 3 minutes in clean water centrifugal machines. The same boiling process was in use in 1888, according to a later edition of the same book. Five years later each boiling was extended to 12 hours, and the boiling lasted for 5 days and nights—that is, the guncotton received 10 boilings of 12 hours each. In April, 1894, this system of boiling was replaced by a system characterised by short boilings at the commencement of the process, the time of successive boilings being gradually increased. The scheme of boiling was a follows :—

No. of	Duration	No. of	Duration
boiling.	in hours.	boiling.	in hours.
1 3 4 5 6	2 2 4 6 6	7 8 9 10 11 12	6 6 9 9 12 12

This system of boiling was continued with but slight modifications until August, 1905. On the introduction of the displacement dipping process it was found, as already stated, that guncotton made in this way was brought to a condition of stability by the boiling process then in use, and just referred to, at an earlier stage than guncotton made by the Abel process. A probable explanation of this fact is that during the displacement process a zone of acid liquid at a comparatively high temperature somewhere about 40° C.—passes through the whole of the guncotton in the dipping pan. The action of this hot acid liquid may be to oxidise certain organic impurities which are certainly present, and to cause the breakingdown of unstable nitrogen compounds into soluble or nonreactive bodies. Systematic experiments were therefore carried out, in 1905, to determine the most suitable and most economical method of purification by boiling, for displacement process guncotton. In the principal experiments two types of boiling were used at first, followed by short boilings; the other in which short boilings were used at first, followed by long boilings. The following deductions were made from the results obtained in these experiments :—

 neutrality is obtained earlier; that less decomposition of the guncotton takes place; that the stability. as shown by the various stability tests, is greater; and that a stable condition is attained earlier.

2. A displacement washing after a long acid boiling at an early stage is a beneficial treatment. This treatment is probably responsible for the early attainment of neutrality.

The system of boiling determined on as a result of these experiments was as follows :---

No. of boiling.	Duration	No. of	Duration
	in hours.	boiling.	in hours.
1 2 3 4 5	12 12 4 4 4	6 7 8 9 10	4 4 2 2 2 2

with a cold water displacement wash after the first two boilings. A full account of these investigations was given in a paper on the purification and stabilisation of guncotton, read by Dr. Robertson before this Section on June 16th, 1906. This system of boiling is still in use at the Royal Gunpowder Factory.

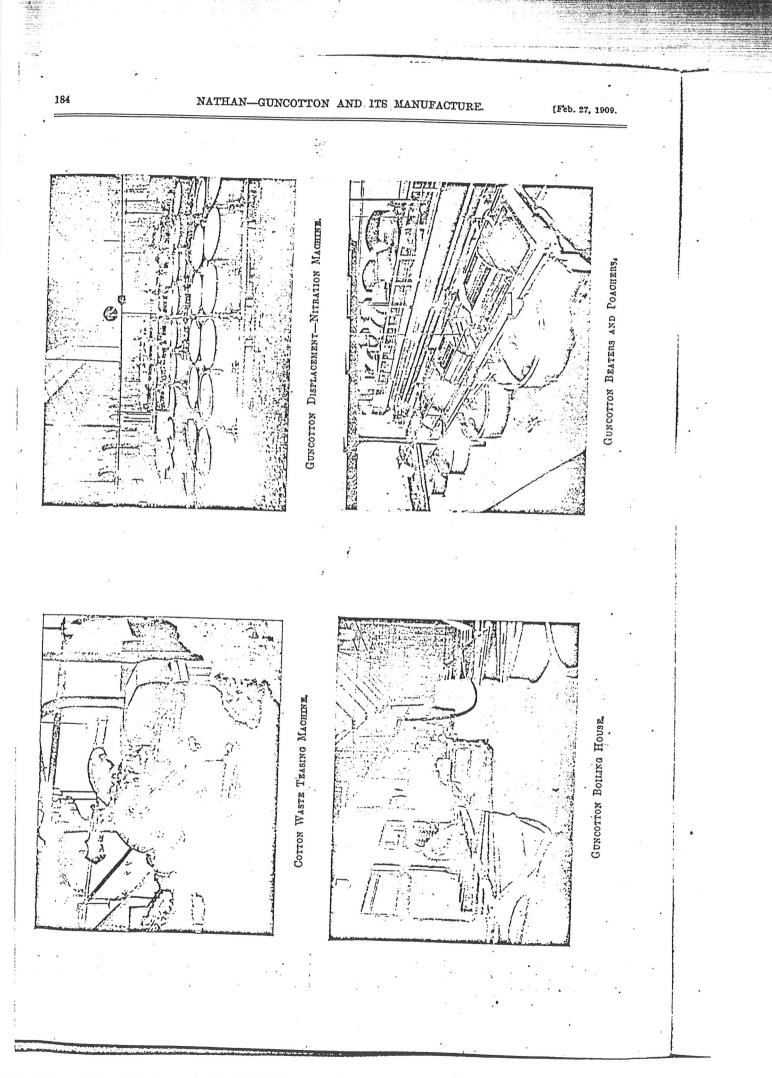
At the Royal Gunpower recorg. The question of how the purification of guncotton can best be effected cannot, however, be considered as settled, nor can the system which has just been described, although it undoubtedly gives an excellent guncotton at the Royal Gunpowder Factory, be applied to guncotton made by other processes, at other factories, without full investigations as to its suitability. Another matter which must be taken into account in connection with the purification of guncotton by boiling, is the nature of the water available. The water at Waltham Abbey is very hard, and its alkalinity may be an important factor in the success of the boiling treatment in use there. This question is perhaps connected with another one, and that is, that the boiling of guncotton can be carried too far. The effect of boiling, whilst it no doubt breaks down impurities, also, no doubt, breaks down the stable ester itself. It is well known that if guncotton is boiled for a sufficiently prolonged period, the percentage of soluble matter will rise and the nitrogencontent will fall. The breaking-down of the ester will be accompanied by the formation of acid bodies, and the presence of alkali in the water will neutralise them and prevent them from reacting on the guncotton.

I have been obliged, owing to want of time, to treat this question of purification very briefly, but it is undoubtedly the most important one in connection with the manufacture of guncotton. One or two matters have been touched upon, in connection with which further work is necessary, but there are many others which will repay very careful investigation and research.

# PULPING.

On completion of the boiling process the guncotton is transferred to a beating engine somewhat similar to that employed for pulping the raw material used in the manufacture of paper. It consists essentially of a large iron roller armed with steel knives, and a bed-plate also provided with knives. The roller revolves, and as the guncotton passes between the two sets of knives, it is reduced to pulp of any desired fineness. As the pulping process proceeds, the roller is gradually lowered nearer to the bed-plate.

Since the introduction of a thorough system of purification by boiling, Abel's original idea that the pulping and washing the guncotton received in the pulping process had a very material effect on its purification, no longer holds good to the same extent. At the same time there is no doubt that the very long staple guncotton before pulping retains in its tubes unstable bodies which no reasonable amount of boiling will remove. The effect of pulping is to materially reduce the length of the fibres and, at the same time, to produce a certain amount of crushing in them. This allows of impurities of an acid character in the tubes being removed, either mechanically or by diffusion.



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## REMOVAL OF FOREIGN BODIES.

After pulping, it is now customary to treat the guncotton in some mechanical way, in order to remove from it particles of metal, grit, and foreign bodies of a similar character. At the Royal Gunpowder Factory this is effected by running the guncotton pulp, suspended in a large volume of water, through grit traps, placed at intervals in a long shallow trough, the bottom of which is cevered with blanket. The foreign bodies, being almost entirely heavier than the guncotton pulp, are retained in the grit traps, and the fine sand, also present in some chantity, is caught by the woolly blanket. An electromagnet in the last grit traps. It is surprising what a large quantity of foreign bodies are removed by these arrangements. In addition to grit traps and troughs, seme factories use what is known as a knotter, the function c' which is to remove small knots and any large pieces of guncotton which may have escaped complete pulping.

### POACHING.

Washing the guncotton during the pulping is effected ir. some factories by the use of drum washers fixed to the beating engine; in other factories and at the Royal Gunpowder Factory this washing is done in separate vessels, termed "poachers." The poachers in use at Waltham Abbey hold about 10 cwt. of guncotton and 1100 gallons of water, and are fitted with power-driven paddles for agitation purposes. The guncotton receives at least three washings; it is allowed to settle down after each washing, and the washing water is removed by a skimmer. The washing water contains in suspension foreign bodies of 'a lower specific gravity than guncotton, and in the case of the earlier washing waters, there is always present a scum centaining nitro-bodies of low stability.

#### BLENDING.

A further purpose served by poaching is the thorough blending of a number of different batches. This is a final blending, but at the Royal Gunpowder Factory there exists a regular system of blending right through the whole of the manufacturing processes. This system is briefly as follows :—The cotton waste reaches the factory in consignments from different contractors. The waste is drawn from store in proportion to the quantities on the contracts, and is mixed and passed through the teasing machine in these proportions.

The next process where blending is possible is in charging the boiling vats. Two vats are filled simultaneously from a number of sets of pans—two pans of each set of four going into one vat; the other two of the set into the other vat. On completion of the boiling, four vats are emptied simultaneously into 32 beaters. This ensures the guncotton from the four vats being blended together in the beating process. On completion of the pulping the beaters are run alternately into the poachers in such a manner that the

On completion of the pulping the beaters are run alternately into the poachers in such a manner that the contents of the 32 beaters are blended into eight poachers. The guncotton in the eight poachers is therefore uniform throughout.

The system produces guncotton of very uniform nitrogen-content. In the 1907-8, 291 tests, representing 600 tons of guncotton, gave the following nitrogen result :--

Maximum.	Ninimum.	Meau.	
per cent. 13-05	, per cent. 12-93	per cent. 13-0195	

#### MOULDING.

For convenience in drying the pulped guncotton it is moulded by light hydraulic pressure into cylinders which measure about  $5\frac{1}{2}$  inches in height and 3 inches in diameter. This is effected by running the guncotton pulp into a moulding machine provided with a number of holes into each of which fits a hollow plunger. These plungers are connected with a vacuum engine, and a good deal of the water is sucked out of the pulp by their means. The

mould block containing the guncotton is transferred to an hydraulic press, and pressure is applied, which has the effect of removing more water and of squeezing the pulp into a condition of sufficient consistency to allow of its being handled with care. In this lightly compressed form a very much larger quantity of guncotton can be dealt with in a drying chamber of any given dimensions than if it is dried in the condition of ordinary pulp, and in its compressed form it possesses the further advantage of being able to be dried on fixed racks. This does away with the necessity and risk of moving drying trays or similar arrangements in a stove. It is also obvious that much less dust is produced.

### PRESSING.

If intended for use in torpedoes, mines, or other demolition work, the guncotton is moulded into suitable shapes, as described above, and the moulds are then subjected to powerful hydraulio pressure, amounting to about 6 tons on the inch, to produce the finished slabs or primers.

### CONCLUSION.

I have endeavoured, very imperfectly I am afraid, to give in a comparatively brief time some account of the history of the manufacturing processes involved in the production of guncotton. Other nitrocelluloses, for the manufacture of which some of the processes are slightly modified, have not been touched upon. The subject is a very wide one, and if it were attempted to go into details, each process would require more time devoted to it than has been given to the whole manufacture. This paper has consisted almost entirely of manufac-

turing details; very little attempt has been made to deal with the chemical questions involved, and nothing at all has been said about the chemistry of the nitration of cellulose nor of the chemistry of the nitrocellulose molecule. The published information on both these subjects is very considerable, and is constantly increasing. I had originally intended to attempt a brief summary of the more impor-I had originally tant papers, but I had to abandon it as quite impracticable. What must, however, strike any manufacturer of nitro-cellulose when he consults the literature of the subject. is that the great bulk of it, although of intense interest, is either too theoretical for practical application, or else that the data, being for the most part the result of laboratory experiments, are not always a sure guide as to what will happen on a manufacturing scale. Our experience at the Royal Gunpowder Factory is, and it is also no doubt the experience of other manufacturers, that all experimental work should be based on sound chemical principles, but to be of practical use it must be conducted on a manufacturing scale wherever possible, and that laboratory work comes in when it is required to ascertain the nature of the results obtained. I venture to think that this is true in the case of several chemical manufactures, and it is most undoubtedly true in the manufacture of guncotton. and I therefore offer the suggestion to any of those chemists who wish to further improvements in the pro-duction of guncotton, to take the manufacturer into their confidence, work with him, and to get him to work with them.

#### DISCUSSION.

The CHAIRMAN asked whether there was any difference in the behaviour of cotton fibres of different origin, such as Bombay, Egyptian, and Sca Island fibres; of course, they might all be too expensive. There, were some other fibres which might perhaps be used, such as the Kapok fibre and the fibre of *Cochlospernium Gossypium*, which the "Indian Museum" in Calcutta was examining at present. Those who had been allowed to see Waltham Abbey in 1900 would realise the vast advances that had been made since then, judging from the pictures thrown on the screen. He could not fully agree with what had been said about the functions of a works laboratory. In the presence of so many military men, it was rather obvious to draw the parallel between strategy and tactics in military matters and in a chemical works. The laboratory could be likened to the General NATHAN-GUNCOTTON AND ITS MANUFACTURE.

taff which laid down the plan of campaign. And just the the tactician had to work it out in the field, so in the ame way the chemist who had made the experiments on a amell scale, had his work cut out for him when endeavourng to carry them out in the factory on a larger scale. No doubt the chemist would meet then with many comingly insuperable difficulties; but having once got the result in the laboratory, he knew he had only details to deal with, however long and patient the work involved in overcoming them might be. At the same time the aboratory must supervise the large scale operations from the beginning, and in all its stages, and not simply come in at the end. Sir Frederic Nathan was in the enviable position of being able to show what he was doing, whilst manufacturers as a rule had to be more ceticent; this paper, therefore, was especially instructive to the younger members of the Society, who learnt the supreme importance of details, and what amount of foresight was required to carry on a manufacture successfully. The thanks of the members were also due to the Government, who permitted the publication of all the details.

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Mr. JOHN SFILLER said he had listened with great pleasure to this history of things as they were, in bygone years, when he was one of Sir F. Abel's assistants; and also to the interesting narrative of successive improvements introduced into the manufacture. He noticed particularly the considerable saving of labour, the larger production, and especially the greater purity of the finished product. It must afford every chemist the greatest satisfaction to find that matters were brought up apparently to the ultimate stage of perfection. He was in Abel's laboratory in the early days of guncotton, even before it was manufactured at Waltham Abbey or only made on quite a small scale; and he was there later when the disastrous Stowmarket explosion occurred. A year or two afterwards he was Mr. E. O. Brown's colleague when he cleared up the mystery, and gave the first indication of the true cause of that disaster, which he did by proving that even moist guncotton could be fired by detonation. There were many narrow escapes in those early days. Brown himself only escaped with his life on one occasion by a few minutes, when he came away from inspecting the magazine at Plumstead Marshes, and told them he had hardly time to lock up the place and run away. He was intending to report to Prof. Abel, but had no time to do so, because the whole thing exploded, in consequence, as it was proved, of the lost the bair off one side of his face through a premature explosion in testing guncotton by heating it in a flask so as to determine the temperature at which the red fumes made their appearance. At the time they did not know everything about guncotton, and it was probably not so well purified as now. At any rate two men, a corporal and a foreman of the Boolison which occurred during the compression of the blocks.

Colonel Sir HILARO BARLOW said he was probably the largest user of guncotton in England, as they filled at the Royal Laboratory all the mines and torpedoes. Wet guncotton was a very innocent thing if it were pure, and there was not much to be apprehended from handling it, but the great difficulty was to arrange to keep it wet. Owing to the circular section of torpedoes it was necessary to shape the oblong slabs of guncotton used for filling in order to fit them in, and the slabs had to be cut up with band saws, and of course there was a great deal of fluff or moist dust produced, and it was not an easy matter to keep the floors on to which this dust fell from getting dry. They endeavoured to keep them constantly moist, but everybody knew what workmen were, and how difficult it was to make them look after themselves. Amongst other things they tried a floor sloping from the centre to the sides with a perforated water pipe running down the middle, so as to get the flow in each direction, but it did not answer, because such an enormous flow of water from one pipe was necessary to keep a large

building wet all over, while a multiplication of pipes made many undesirable excrescences on the floor. many undesirable sector to the humble watering can, which they had to revert to the humble watering can, which was very excellent if the men would use it. With regard to filling mines and torpedoes, there was always a hankering after using enormous blocks of guncotton, which looked at first sight an admirable system, but it was not as good as it looked. He made no criticism on the methods employed by one company he knew of who made the most admirable solid guncotton he had ever seen, so solid that a block of it over 400 lb that a block of it over 400 lbs. in weight had to be put that a block of it over 400 lbs. in weight had to be put on the floor and cut with a cross-cut saw like a tree, in order to get blocks of it cut up for certain tests of the distribution of moisture. It was in connection with the distribution of moisture in wet guacotton that these have blocks were so inconvenient. With avalaging large blocks were so inconvenient. With explosives nothing could be taken for granted; you had to find what the moisture really was, and therefore if a large block were being tested as a complete charge a piece had to be taken from the inside as one would from a cheese. and they had to invent a tool for taking these tests. It was a difficult problem, because one could not allow the water to run on the tool, as it would at once destroy the water to run on the tool, as it would at once destroy the moisture test; therefore they had to have a tool with a water casing round it, and to bore in to a depth of something like 14 inches to take out the sample column and test the moisture therein. Further as to repairs. When the torpedoes were filled with a few large specially shaped blocks, the blocks retained as spare for the carrying out of repairs were only of use for the one purpose, whereas if the charges were built up of slabs like bricks of about, 2 lbs. weight each, and such slabs were used for all purposes. the keeping of spare slabs for repair purposes was a simple matter. At the same time, better density was obviously obtained by filling with a few blocks rather than with a lot of small bricks.

(Feb. 27, 1909.

Mr. WALTER F. REID said the author rather excused himself for not referring to the chemistry of the nitration of cellulose, but in his opinion those with most experience of the subject attached least importance to the many different views put forward on nitration, and the products derived from the operation. He thought also there was some little misunderstanding with regard to his con-cluding remarks about the position of the chemist. There was really very little difference between the analytical operations in the laboratory and the operations carried out in such a factory. The author had very fully utilised the services of the chemists at his disposal, being himself an excellent chemist, and when he listened to the method by which these acids were first mixed in bulk, then analysed. and then again brought up to the right strength, it occurred to him that many who for years had simply been mixing their acid by volume according to analysis had not sufficiently utilised the services of the chemist. Research work, on which the importance of all factory operations so intimately depended, had not been omitted; as was clear not only from the elaboration of this method of nitrating cotton, but also from the method of making nitroglycerin, which was described in a recent paper. The mechanical proparation of cotton still appeared to him to be somewhat crude. He would not say that this was not the best method at present on a large scale : but there still seemed something wanting in the way of a fine subdivision of the material both before and after it came in contact with the acid. He thought there was an opening also for an improvement of the process of weighing. The English climate was not always dry; weighing. The English climate was not always dry; there were times, as had recently happened, when the atmosphere was fully saturated with moisture, and unless special precautions were taken in exposing this finely divided, very dry cotton, it would absorb a considerable and variable quantity of moisture. This was a very difficult problem, and still awaited solution. With regard to the yield obtained, and the very large output per man in the factory, in spite of all displacement of labour, he thought labour of that kind ought to be displaced wherever it could. because the men worked under great sanitary it could, because the men worked under great sanitary disadvantages, and if they could in any way supplant human labour of this kind by appliances it was good for humanity. With regard to the materials used, he did

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# NATHAN-GUNCOTTON AND ITS MANUFACTURE.

not think they had by any means reached finality, and there might be many possibilities ahead. Curiously enough, the material used was most unsuitable from its mechanical shape ; the cotton fibre was a capillary tube, and unless that tube was absolutely destroyed, one could not prevent the strong capillary action with which it would retain all liquids. Until the structure was destroyed would retain all liquids. Until the structure was destroyed one rould not be absolutely sure that the guncotton was perfectly pure. He had tried some small experiments in that direction; instead of the machine adapted from the paper industry, he had tried fine grinding, and it was quite possible on a large scale to absolutely reduce the fibres to a powder. Then the subsequent purification would be simpler, and probably the cost would be reduced. Many years ago he had tried a number of fibres which were not tubular, one of which, kapok, had heen already mentioned. He thought Sir Frederic Nathan found it was a very greasy material, and he did not get a good vield from it. Incidentally, the sample of kapok, of which he had obtained a considerable quantity, had been finally attilised for another purpose. It repelled water so effectually that it had been used as a material for lining the inside of the cars of the balloons that took part in the long distance race for the Gordon-Bennett Cup. It had since been used by balloonists for lifebelts. In one explosive industry, at any rate, that of Schultze powder, one of the first smokeless powders made for sporting purposes, the cellulose from wood had been used. It was quite possible that with higher purification of wood fibre and reducing it more to the terms of cellulose, it might ultimately become a substitute for cotton in this andustry. It must be remembered that they were in rather an awkward position in the explosives industry ändustry. being dependent both for nitric acid and cotton on other countries; so that if the supply of either or both should be cut off they would not be able to make cordite or the other nitrated material. It would therefore be an advan-tage if they could find some substitute for cotton, which could be used for making nitrocellulose.

Mr. OSCAR GUTTMANN said the figures by the Abel process were given as 13.75 to 1.25 of cotton waste or 1 in 10, which was the quantity remaining in the pots after dipping and squeezing out the acid on the grid at the back, but the nitration took place in 220 parts of acid. The nitration lasted 5 minutes, but the after nitration in the pots 24 hours. The nitration was probably finished as to over 90 per cent. within the first 5 minutes, and only the last 10 per cent. wren finished in the next 24 hours by the acid absorbed by the cotton. The reason The reason so large a quantity was required was that the cotton itself was so absorbent that it could not be dipped unless there had a large excess. There were also some figures missing with respect to the output per man in the nitrating centrifugal machines; he believed it was about the same as by the Ardeer process. While it was quite true that the output per man was very much larger in the case of the displacement process, they ought to consider that by the nitrating centrifugal at least six operations can be made in a day, whilst according to the description given in the Zeitschrift für das gesammte Schiess- und Spreng-stoffwesen in 1905, which was still true, it took at Waltham Abbey 7 to 8 hours to finish a charge. Still, this was only a feeble criticism on what was otherwise an excellent process, carried out in the absence of fumes and inconvenience to the workmen, and giving excellent guncotton such as required at Waltham Abbey. They had, how-ever, heard from the author that it probably would not be quite as useful for other kinds of guncotton; at any rate, it would have to be tried, and he thought these trials were wanting. He could not be expected to go fully into this process, nor into the whole question of the manufacture of guncotton, since about a month ago he had done this in another place. Mr. Reid suggested that wood fibres when better purified might be useful for the production of explosives, but trials had shown that, for the reasons Mr. Reid had given, they could not get the same good result from it as from cotton. He asked the author to what he attributed the yield of 170 as against 159 and 160 of other processes. They knew that he was not a friend of the nitrating centrifugal

machine, but he saw no reason from a mechanical and manufacturing point of view why the yield should be 6 per cent. better in one case than in the other. His impression was, that at Waltham Abbey they took much greater care in selecting and preparing the materials and in the manufacture, and that this was entirely responsible for the increase. Finally, as one who had seen more than half the guncotton factories in Europe, he would say that small laboratory experiments with explosives were not directly transferable to the factory, and that in this sense Sir Frederic was quite right. He was however the last man to deprecate the work of the laboratory, because it always showed the way, ard two of the most brilliant examples were the investigations of Professors Lunge and Will, who by their work had suggested a very large number of improvements. A laboratory experiment might be made on 5 grms. or on 5 lb.; and manufacturers nowadays make the latter, because experiments on large charges were absolutely and directly transferable to manufacture.

Sir FREDERIC NATHAN said the question raised by the Chairman with regard to laboratory and manufacturing experiments had been very ably answered by Mr. Guttmann. He had stated in his paper that all experimental work should be based on sound chemical principles; that implied that the laboratory initiated experiments, but that the experiments themselves must be conducted on the manufacturing scale. In writing as he had done, ho had in his mind an immense amount of very valuablo literature on the subject of guncotton, but it had been his experience that following the method described, resulted in failure to produce on the manufacturing scale anything like the bolies obtained by those very able chemists who had worked on the laboratory scale. As regards cotton waste substitutes he had tried unsuccessfully a large number of other materials besides those he had mentioned. By the time the percentage of resistent cellulose, suitable for nitration, had been got, a vast amount of time, labour, and material had been expended for a very small result. With regard to the "teasing" system, he had described it simply as it existed; other methods had been tried as methods the existed. methods had been tried, *c.g.*, reducing the cotton waste to the condition of cotton wool, but there was nothing at all gained ; on the contrary, considerable extra expense was incurred with no corresponding advantage. The method of weighing the cotton waste appeared perhaps rather crude, but when dealing with very large quantities, and a difference of } per cent. of moisture one way or the other was not a very serious matter, it affected the per-centage of soluble mainly, in view of the fact that in the later guncotton processes there was a considerable blending, which was moreover carried right through the operations of converting the guncotton into cordite. He entirely agreed with the views expressed by Mr. Reid with regard to the purification of nitrocellulose; it was no doubt the one operation in the manufacture which they knew less about than they would like to. Experiments on this point are being constantly made with the hope that it would not only be possible to shorten the boiling process but at the same time be able to produce a much better guncotton than was being produced even at the present time. He agreed with Mr. Reid that destroying the fibrous structure of guncotton prior to purification and boiling would probably be a material step in advance. It had occurred to him and to others, and he believed a patent had recently been taken out for that purpose. With regard to the low yield from the nitrating centrifugal, he was unable to afford any explanation. Some displacement process was that there was no loss due to fuming off, either in pots or centrifugals, or in the mechanical operations of immersing, water-centrifugally, acid-centrifugally, etc., but mainly that a more complete conversion of the cellulose into nitrocellulose took place in this process.

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