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G. BROMBERGER W.75 421

Explosives and Materials Research and Development at Waltham Abbey

> CHIEF SCIENTIST'S SUMMER CONFERENCE 8 July 1966

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ORGANISATION

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Principal Superintendent/Development: Dr. G. H. S. Young

Special Merit 'B' Post: Mr. G. K. Adams

Superintendents:

| Explosives 1: | Dr. A. L. Lovecy | | |
|-------------------------------|------------------------|--|--|
| Explosives 2: | Mr. E. G. Whitbread | | |
| Propellants 1: | Dr. W. G. Williams | | |
| Propellants 2: | Mr. P. R. Freeman | | |
| Materials 1: | Dr. R. L. Williams | | |
| Materials 2: | Mr. J. E. Gordon* | | |
| Analytical Services | Dr. I. Dunstan | | |
| Chemical Engineering: | Mr. R. G. Ross | | |
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| Chief Administrative Officer: | Mr. S. F. M. Whiteside | | |
| * Individual Merit S.P.S.O. | | | |

FOREWORD The Work of E.R.D.E.

(1) E.R.D.E. devotes about 70% of its effort to work on solid rocket propellants, high explosives and initiators, and about 30% to work on materials. A small amount of work on cryogenics is carried on in support of the R.P.E. programme on liquid hydrogen.

(2) E.R.D.E. is an integrated establishment serving directly the three Service departments as well as the M. of A. itself, and thus has direct responsibilities to such bodies as R.A.R.D.E., The Ordnance Board, the R.O.Fs., the Inspectorates, etc. It is solely concerned with research, initial development and post design services, it does a good deal of advisory work, but has little direct responsibility for projects. Our contributions to these are made indirectly, for example, by developing propellants for R.P.E. rocket motors, gun and mortar ammunition, initiators, combustible cartridge cases, sealing compositions, etc., for R.A.R.D.E., and in the development of production processes for propellants, initiators and high explosives for the R.O.Fs.

(3) The Materials work is principally concerned with polymer chemistry and with refractory fibres for composites. We do a good deal of work on polymers in direct support of the various establishments of the Army Department but also carry out research on the engineering properties and on the thermal and oxidative stability of polymers, and adhesives. The work on whiskers is concerned to develop new engineering materials of ultra high strength.

(4) The existing White Paper strength, mainly chemists, is about sixty-five Scientific Officer grades, one hundred Experimental Officer grades and eight Engineers. No Service Officers are attached. The industrial strength is approximately five hundred. 1

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(5) Unlike most industries, there is no commercial network on military explosives. Only one firm in the country-I.C.I., Ardeer-has any real interest and then only as a side line to their business in commercial explosives. Similarly in propellants, there is only the I.M.I. agency factory at Summerfield working on cast double base compositions. For this reason it is particularly important that we should maintain expertise over the whole of these areas and apart from the development of new compositions our main function is to carry on research which will enable us to deal with the many and various ad hoc problems on foreign weapons, Service life, production difficulties, storage and safety problems and the like. For these reasons also we need to maintain a substantial interest in chemical engineering, including the capacity to manufacture pilot plant quantities of one or two tons of new compounds, and to maintain a variety of testing facilities capable of operation on a significant scale.

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(4) The existing White Paper strength; mainly chemists is about sixty-five Scientific Officer grades, one hundred Experimental Officer grades and eight Engineers. No Service Officers are attached. The industrial strength is approximately five hundred.

EXPLOSIVES 1

Military explosives, with very few exceptions, are mixtures of chemical ingredients, each of which contributes in its own particular way to produce the required set of properties. The classical example of gunpowder serves to illustrate the point, since good results in the gun were greatly dependent upon the careful attention given to the quality of the charcoal ingredient.

In modern explosive compositions, the ingredients are usually quite elaborate compounds and many of them have distinctive functions. For instance, organic compounds of many kinds are used to ensure adequate stability in adverse storage conditions; others again serve to control burning-rates or to modify sensitiveness; in these ways the usefulness of the major explosive compounds, such as guncotton, nitroglycerine, and RDX, can be extended to meet more exacting requirements.

It is the task of the E.1 branch to study the connection between the chemical constitution and the useful properties of the various substances, and also to ensure that all necessary information is available about methods of manufacturing those which are actually or prospectively needed. Novel and unusual compounds required for the programmes of work in other branches are made on a laboratory scale and, where larger supplies become necessary, details are worked out to assist the Chemical Engineering branch in setting up pilotplant operations. Similar help as regards normal production and quality control of ingredients is made available to the the Chemical Inspectorate and the Royal Ordnance Factories, and in the case of initiatory explosives the procedures are developed and introduced in very precise detail to the factories concerned.

Research is in progress on the basic reaction-mechanisms involved in the formation of RDX by nitrolysis and of HMX in the very complex conditions, still quite obscure, of the Bachmann process of manufacture.

Several explosives favoured in U.S. for high-temperature applications have been made and one, hexanitrostilbene, is

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being more closely studied with regard to optimum yield and reliable conditions of preparation. Advice based on extensive previous studies has been given recently to R.O.F. Bridgwater concerning DATB manufacture.

Research on thermal decomposition behaviour of these explosives has now begun again after a long interruption through loss of staff. Methods of measuring rates of consumption of explosives on the scale of a decigram are also being investigated to provide a basis for comparative evaluation procedures.

Senior Staff

Mr. G. W. C. Taylor (Special Merit S.P.S.O.).
Mr. W. E. Batty.
Mr. C. H. Miller.
Dr. A. J. Owen.
Dr. R. J. J. Simkins.
Mr. A. T. Thomas.

EXPLOSIVES 2

E.2 branch is particularly concerned with the development of high explosive and propellant compositions of improved performance.

In each case it is probable that a marked increase in performance can now be obtained only by the use of ingredients which increase the explosion hazard in manufacture and use of the final product. These ingredients are the so-called high energy fuels and oxidisers. The key problem therefore is to understand and manipulate in a favourable manner the factors affecting the risk of accidental explosion whilst retaining, in the case of high explosives, the ability to detonate on demand and this forms a large part of our work.

Because this programme involves work with materials which are much more sensitive than those usually employed in propellants and secondary explosives, it has been necessary to set up an entirely new facility. In this we will take in relatively inert raw materials, process these into explosive compositions, fill and fire rocket motors or explosive charges and thus gain technical data, entirely by remote control from behind adequate cover.

In addition to simply increasing the energy content of explosives it is sometimes possible to make a considerable gain in effectiveness by correctly exploiting the mechanism of detonation; one example is the use of explosives in underwater weapons. For this application an attempt is being made to tailor the shape of the underwater shock wave by changes in the composition of the explosive, so as to achieve the most damaging pressure-time profile. This work is being carried out in close collaboration with the Naval Construction Research Establishment which has been able to define quite closely the optimum pressure pulse for various purposes.

Work on high energy propellants is as yet in an early stage but we will be making ballistic measurements by the spring of 1967. These materials will offer an advantage of a higher specific impulse or rate of burning but their use will probably be limited by reason of cost or explosive properties to special applications.

The Branch employs its specialist knowledge on the sensitiveness of explosives to provide a service to the rest of the Establishment and some outside bodies in that we carry out trials to provide information on the sensitiveness and hazard associated with all explosive materials. In addition, because we have certain expertise, we have undertaken two subsidiary tasks: —

(a) The development of special explosive charges to produce air blast waveforms of specified form, e.g., rocket motor take-off blast, sonic bangs; and the firing of these charges in trials run in collaboration with other establishments.

(b) The development of special techniques to employ the chemical energy in explosives to pump high energy lasers. This work has led to an interesting study of the radiation from dense plasmas.

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Certain fundamental work is also carried out partly in direct support of the main programme outlined above and partly as a contribution to the scientific well-being of the establishment;

(a) A study of the temperature and emissivity of the detonation front in liquid and solid explosives.

(b) A study of the temperature and emissivity of nonreactive shock waves in explosives.

(c) A study of the kinetics and reaction mechanisms of the oxidation of alkyl and alkoxyl radicals by oxides of nitrogen.

Senior Staff

Dr. R. Campbell.

Dr. J. Hicks.

Dr. L. Phillips.

Dr. R. M. H. Wyatt.

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PROPELLANTS 1

The work of this Branch is largely concerned with the so-called "colloidal" propellants, based on nitrocellulose, which are used in a variety of shapes and sizes, for a range of applications, including small arms, guns, mortars, rockets and power cartridges. Work is also undertaken on methods for the control of ballistics in production, and to devise ballistic assessment techniques for special requirements at high pressures and temperatures. The Branch advises on propellant aspects during the design stage of Service projects and makes contributions throughout the development stage, the many Service acceptance stages, and also is involved in problems of Inspection and assessment of Service life.

New compositions are devised to meet special requirements for ballistic and storage life. Production scale plant is available for manufacture and batches of some hundreds of pounds are supplied to assist weapon development by the design establishments. Advice is given to the production

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factories on processing problems and experiments are conducted to help resolve difficulties. Assistance is given to the Design and Inspection Authorities in the preparation of specifications for propellants and ingredients. The Branch is also responsible for the detailed technical control of extra-mural contracts on Cast Double Base (C.D.B.) and gives considerable technical support.

Recently, effort has been devoted to devising and standardising suitable propellant charges for the Martin-Baker Rocket Ejection seat and the Giant Viper project. New problems have been presented by designers requiring burning times in the milliseconds range for the smaller weapons and novel charges are being devised. Increasing emphasis is being placed upon minimal smoke for line of sight weapons and a programme of basic work is proceeding to untangle propellant and inhibitor problems.

The case-bonding adhesives system for C.D.B. devised by the Branch has been applied at I.M.I. to the four motor projects currently in hand; a joint patent application has been made. Basic work has been undertaken to establish the capabilities of case-bonded C.D.B. as single and dual charges for Land Service usage. Work on re-formulation of composite modified C.D.B. to give higher impulses by use of alternative nitric esters has continued and has involved development of new solvent systems for more highly filled casting powders.

The E.R.D.E. Mk. III Ultrasonic flaw detector is being assessed for the inspection of solid cigar burning C.D.B. charges. An improved process is being devised for the manufacture of combustible cartridge cases for gun ammunition.

Senior Staff

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PROPELLANTS 2

This Branch is divided into four Sections, as follows:

Composite Propellant Section

This Section is responsible for research and development on plastic and rubbery propellants; it is the only place in the U.K. where such work is carried out. The facilities include laboratories for research and quality control, including ballistic and physical property assessment, together with smallscale and large-scale manufacturing plants. It provides a service to the Rocket Design Authorities (R.P.E. and R.A.R.D.E.) for formulating and developing propellant compositions to meet specified ballistic and mechanical properties, for developing manufacturing and, sometimes, rocket filling methods, and for supplying tonnage quantities of experimental compositions required for the development phase of new rocket motor projects.

It is also responsible for the improvement and development of manufacturing plant and for research aimed at improving ballistic and physical characteristics of plastic and other composite propellants.

Combustion Section

This Section carries out research aimed at improving the understanding and control of factors which determine the burning rate and combustion efficiency of propellants in rocket motors. Current investigations include the pyrolysis of binders and the surface temperatures and burning rates of porous beds of oxidant. More applied research is concerned with the enhancement of burning rate by the inclusion of metal staple and foil.

Adhesion and Rheology

This Section is responsible for rheological and surface chemical investigations of solid propellants with the objective of improving their mechanical behaviour and of relating laboratory measurements with Service performance. Basic

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research is carried out on the measurement of adhesive, tensile and shear strengths, and on the interfacial properties of oxidant/fuel combinations. The Section also provides an advisory service for the use of sealants, adhesives and lutings.

Heat Transfer and Low Temperature Research

(Mr. Ziebland. Individual Merit S.P.S.O.)

This Section is concerned with the study of heat transfer characteristics and the measurement of heat transfer parameters of materials of interest as rocket fuels or oxidants under conditions encountered in liquid propellant rocket engines. At the moment the work is concentrated on the hydrogen/oxygen system which is of current interest as a rocket fuel combination. The work is largely specialised engineering because of the unique types of measuring apparatus required and the results are of importance not only for rocket design but in a wider scientific context in that they add to the store of fundamental thermodynamic data.

Senior Staff

Mr. H. Ziebland (Individual Merit S.P.S.O.).

Mr. W. A. Dukes.

Dr. J. Powling.

Dr. J. H. C. Vernon.

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MATERIALS 1

The research activities of this Branch are directed towards obtaining a better understanding of the behaviour of nonmetallic materials exemplified by plastics, rubbers, adhesives and fibrous substances, in order to promote their successful application in Service equipment. All these substances are polymers, and the branch is therefore divided into sections dealing with polymer physics, polymer chemistry, polymer development and applications, and autoxidation. The branch is also responsible for the programme of work of the Joint Tropical Research Unit at Innisfail, Australia.

The polymer physics group is primarily concerned with obtaining data on the mechanical properties of polymers such

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as the elastic moduli, elongation at break, and ultimate strength, for use by structural engineers. Much experimental work is done in this field, particularly over the effect of extremes of temperature, wide ranges of rates of stress application, the influence of long term loading, and fatigue.

The polymer chemistry group makes a detail study of the mechanism of chemical breakdown of polymers under the influence of heat, radiation, oxidation, etc. From this information it is hoped to suggest ways of altering the structure to resist these effects. Work also goes on with the characterisation of polymers by molecular weight and its dispersion, nuclear magnetic resonance and infra-red spectra, cohesion energy density and glass transition temperature.

The polymer development and applications group is almost self-explanatory. The group is responsible for advising establishments such as R.A.R.D.E., M.E.X.E., F.V.R.D.E., etc., on the choice and use of polymers, and for carrying out applied research on behalf of these places. They maintain contact with producers of polymers and examine any new developments in this field.

The section concerned with autoxidation is run by Dr. N. Uri, an individual merit S.P.S.O. It is a small group concerned with the discovery of new additives to prevent the slow spontaneous oxidation of polymers by the atmosphere. Some materials of considerable promise have been found and are being developed.

The Tropical Research Unit, which is a joint project with Australia, carries out long term exposure trials of both metallic and non-metallic equipment to tropical conditions of both the hot/wet and hot/dry type. This information is of the greatest importance to the designers of Service equipment.

Senior Staff

- Dr. N. Uri (Individual Merit S.P.S.O.). Dr. J. H. Golden.
- Dr. B. L. Hollingsworth.
- Dr. B. J. MacNulty.

MATERIALS 2

The design of radically new and better structural materials and the development of their constituents is difficult because a structure which is both light and safe requires a balance of properties in the material so that it is generally not of much value to take the easy route of increasing one property at the expense of the others. The construction of materials with increased strength, Young's Modulus and toughness generally involves the preparation of strong fibres from covalent chemical species such as graphite or ceramics and although the chemical purity of the fibres is not usually very important, their physical condition and especially their surface smoothness and crystal structure almost wholly controls the fibre strength. One is, of course, looking for strengths which are substantial fractions of the theoretical values.

The attainment of these conditions, which involves a hitherto neglected area of high temperature chemistry, is becoming a new technology which may have considerable industrial importance. Nowadays it is possible to make fibres which are not only much stronger for their weight than metals but, unlike glass fibres, are also much stiffer, moreover there are now several different ways of doing so.

Of the various fibres and processes which are being developed, whisker crystals offer much the highest strengths in conjunction with high or moderate stiffness and for this reason most of the work on fibre preparation at E.R.D.E. has been on whiskers. Although the present cost of experimental production is fairly high, it is thought that unattended crystal growth processes should be potentially cheap. Silicon nitride (Si₃N₄) whiskers are being grown here in kilogram quantities in the large "bran-tub" furnaces which can be seen and present research is directed both to cheapening the process and to increasing the yield. We are also interested in the production of silicon carbide (SiC) whiskers which have better all-round properties.

The improvement of whisker growth involves both furnace engineering and also a basic study of the growth conditions

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which are difficult to observe in any detail because of the high temperatures used ($\sim 1500^{\circ}$ C.). With the aid of computers the very complex chemical thermodynamics is being studied.

Although whiskers are very strong, they are fairly short and it may be more difficult to exploit their properties effectively in some matrices than is the case with continuous fibres and for this reason among others a mathematical study of the elastic theory of two-phase systems is being made. On the whole the indications are that whiskers are best employed in a metal matrix which transmits the stress to them more effectively than does a resin.

In many ways, however, short fibres such as whiskers and asbestos are easier and cheaper to handle during the manufacturing processes than are continuous fibres because they can be manipulated in liquids and pumped around as suspensions or slurries. We have developed a number of processes for the automatic cleaning, sorting, fractioning by length and spinning into parallel threads of short fibres, many of which may have wide applications and these processes can be seen. This is a field where we are ahead of the Americans at present.

Asbestos is a cheap material fibre in many respects similar to a whisker but its use as a reinforcing agent has been handicapped in the past by poor handling techniques. We have been able to treat asbestos by much the same processes as those used for whiskers and although the fibre is unsuitable for reinforcing metals because of its poor temperature resistance, asbestos makes an excellent fibre for reinforcing plastics. We are now actively developing an asbestos reinforced plastic which is about $2\frac{1}{2}$ times as stiff as fibre glass and has much the same strength. This material, which is potentially quite cheap, is stronger and stiffer for its weight than most metals and although its specific stiffness is lower than carbon fibre material, its strength is better.

The main field for whiskers at the moment seems to be in the reinforcement of metals, especially aluminium, for service

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up to moderately high temperatures. We are developing an aluminium material, reinforced with Si_3N_4 whiskers (perhaps later with SiC) which has excellent properties. Unlike conventional aluminium alloys, it retains useful strength and stiffness up to at least 300° C. and unlike other reinforced metals, it behaves elastically and shows little or no hysteresis.

For service at the highest temperatures (> 1000° C.), for instance for the blades of gas turbines, the strongest fibres are unsuitable, chiefly because they dissolve in the matrix after long periods at $1100-1200^{\circ}$ C. On the other hand the highest strengths and stiffnesses are not at present needed for these applications. We are therefore developing a thick polycrystalline alumina (Al₂O₃) fibre which although of only moderate strength, is stable for long periods in nickel alloys at high temperatures. All this work can be seen in L.157 Building.

The group is run by an individual merit S.P.S.O., but reports direct to the director.

Senior Staff

Dr. C. C. Evans. Dr. T. J. Lewis. Mr. N. J. Parratt.

ANALYTICAL SERVICES

This Branch has the following functions:

- 1. To develop new analytical methods for all experimental explosive compositions; these subsequently form the basis for specification and control tests.
- 2. To provide an analytical service for the whole establishment and assist other branches in specialised analytical applications which arise in their research.
- 3. To advise on all aspects of stability and surveillance testing of propellants. Conventional methods of surveillance testing are supplemented by investigation of newer techniques, particularly those based upon thermochemical analysis,

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4. The Branch is responsible for deciding questions of compatibility of propellants, high explosives and initiators with the materials with which they come into contact. A large amount of testing and advisory work is involved. A related interest which is being developed is concerned with the appraisal of chemical hazard, particularly in unit processes, e.g., nitration, distillation and thermal decomposition.

In addition to its service to the establishment, the Branch has customers in many outside organisations, including the various industries working on Government contracts (e.g., B.A.C., Ferranti, Hawker Siddeley, I.M.I. Summerfield, I.C.I. Ardeer) other government departments (e.g., R.A.R.D.E., R.A.E., R.P.E., D.C.I.) and N.A.T.O.

In order to exercise these functions, the Branch operates a wide range of specialised techniques and instrumental facilities and carries out research into new and improved methods of analysis. Research is also carried out on the preparation, properties and reactions of substances which are of importance to the establishment. Recently the main interests have been thin-layer chromatography, mass spectrometry and isothermal heat-flow calorimetry.

Thin-layer chromatography has been very successful in qualitative and quantitative analysis and may provide a new method for examination of propellants in the field.

Mass spectrometry has been successfully used in conjunction with thin-layer chromatography and for measurement of isotopic composition of labelled reaction products.

Construction of an isothermal heat-flow calorimeter is nearly complete; this should be of very great use in the assessment of new propellant and explosive compositions.

The methods of infra-red spectroscopy, gas chromatography and X-ray crystallography are used extensively. Compatibility and stability work has continued with studies of stabilisation mechanisms and reaction kinetics.

Senior Staff

Mr. J. R. C. Duke. Mr. N. J. Blay. Mr. F. W. S. Carver. Dr. J. C. Wright.

CHEMICAL ENGINEERING

A major activity of this Branch-" Process Research "is the development of processes for making on the larger scale chemicals which are required in the Establishment's work. They have included high explosives, ingredients for rocket propellants, high energy ingredients for advanced explosives, whiskers and aligned inorganic fibres in collaboration with Materials 2 Branch, as well as unusual chemicals for basic research purposes and various materials for other establishments. This work involves designing-up a laboratory process, often re-casting it to make it suitable for scale-up, and usually operating on a pilot plant to confirm data for designing larger plants and/or to make product for use or assessment. Apart from normal and industrial laboratory facilities we have a range of chemical plant which we assemble and operate in shed-buildings for non-explosives, or mounded buildings for explosives. We have also provided remote-control emplacements where the operator is in a safe control room for possibly hazardous operations.

In developing processes it is frequently necessary to investigate an operation in more detail in the laboratory. This leads to our second activity, "Unit Operation Research," the basic study of operations to improve our understanding and use of them, and contribute to the general fund of knowledge; for these operations are of universal application whatever the product being made. We are studying crystallisation in several aspects, most explosives and propellant ingredients being crystalline materials, and remote control techniques.

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In the third field—" Equipment Research "—the present research concerns paste mixing and mixers, both as regards performance and safety design, because this is a frequent and important operation in making solid propellants, and remote processing methods, control equipment and instrumentation.

To some extent therefore the Chemical Engineering Branch works in support of other Branches in making materials and advising on processing and plant problems, but the work includes also a direct contribution to processing technology.

The Branch also includes the Instrumentation and Glass Engineering Sections. The former develops, provides and services instruments and electronic equipment for the Establishment, and the latter does the same for glass apparatus and equipment. But, again, their work can also be of value outside the Establishment.

At present the Chemical Engineering Branch is in the middle of a major rebuilding programme; many of the buildings are being replaced or rehabilitated. This programme will be completed by 1967.

Senior Staff

Dr. A. W. H. Pryde (Individual Merit S.P.S.O.). Mr. R. P. Ayerst.

Mr. R. Fisher.

Mr. I. C. P. Smith.

MISCELLANEOUS RESEARCH ACTIVITIES

A small group under the direction of Dr. L. J. Bellamy and Mr. G. K. Adams is engaged in research on the following topics. Many of these are essentially short term probing investigations by not more than one man with the object of deciding whether or no a particular field merits a more elaborate study.

The theory of unsteady, chemically reacting flow with application to the growth of shock waves in explosives to steady detonation waves.

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Thermodynamic calculations of explosive and propellant performance.

Spectral analysis and loudness calculations of real and simulated sonic booms.

Infra-red studies of hydrogen bonding and interactions between molecules in solution.

Electron impact spectroscopy of organic molecules.

Study of the luminescent properties of lanthanide chelates with particular reference to their use as laser materials.

The growth of metal oxide crystals by vapour phase transport.

The use of field emission microscopy to study the influence of surface contaminants and growth rate on the morphology of crystals.

Senior Staff

Mr. M. J. Harper. Dr. A. R. Osborn.

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Historical and Geographical

The previous Director, Dr. C. H. Johnson, was a considerable authority on the history of the old Royal Gunpowder factory and of the earlier commercial enterprise that preceded it on this site. On the occasion of the previous Chief Scientist's Conference at Waltham Abbey he wrote an historical account of the establishment. This was so well done that no apology is offered for reproducing it on this occasion.

abitation of a fitzer out increasing the second and the oldest gunpowder factory in Britain, boasting four hundred years of continuous operation. Not infrequently these ancient powder mills entered into the calculations of the country's leaders, as for example William Cecil's when at the beginning of the reign of Elizabeth I he decided to accelerate the production of certain vital war materials, gunpowder amongst them. For us the most significant occasion was the outright purchase of the mills by the Government in October, 1787, just prior to the twenty years' struggle with Napoleon. Thereafter they were known as the Royal Powder Mills; much later as the Royal Gunpowder Factory. Yet barely four years earlier Prime Minister Pitt had given serious consideration to closing the Government-owned factory at Faversham, Kent, representations having been made to him that "the powder merchants could make better gunpowder and much cheaper than the King's servants". The business was extremely lucrative. Pitt's change of mind had been assisted by General (later Sir) William Congreve, Controller of the Royal Laboratory, Woolwich, and the Duke of Richmond, Master General of the Ordnance, who recommended that instead of withdrawing from gunpowder manufacture the Government should extend its interests by acquiring the mills at Waltham Abbey, "... reckoned the most complete in England ". The price paid to John Walton, in whose family they had been for a century, was £10,000 plus interest at 5 per cent. per annum, pending settlement of the complex legal issues over land and waterways. During the next decade, the Government spent nearly £35,000 more on buying surrounding land extending their water rights and on new construction.

General Congreve's son, also named William, is renowned for his advocacy and development of the military rocket-

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initially an instrument of flame warfare—first demonstrated to Pitt and members of the Cabinet in 1805. William Congreve Jnr. joined the staff of the Royal Laboratory in 1791 at the age of nineteen, later becoming Deputy Controller and, on his father's death in 1814, succeeding to the baronetcy and to the post of Controller. In view of all these coincidences it is understandable that the part the father played in the Government's acquisition of the powder mills should have come to be attributed, mistakenly, to his famous son.

William Congreve Jnr. began systematic investigations at Waltham Abbey and at Faversham on quality control of gunpowder, especially in respect of the charcoal ingredient which gave a lot of trouble. By 1810, in a little over twenty years, the two Government factories together had produced more than 18,000 tons of powder and "recovered" a further 6,000 tons. Congreve calculated that the Exchequer had thereby been saved £341,448 17s. $3\frac{3}{4}$ d. which would otherwise have gone into the pockets of the trade. In a contemporary mortar trial carried out on Marlborough Downs (the Lea Valley Growers' Association would have approved) Waltham Abbey's gunpowder carried 4,430 yards, Faversham's 4,340, while, it almost goes without saying, samples submitted by six merchants, poor wretches, ranged between 3,800 and 4,270 yards. Whatever Congreve's qualifications may have been in accountancy-he was Member of Parliament for Plymouth from 1818 until his death in 1828 and wrote pamphlets on improving the currency -the King's servants, under his direction, certainly appear to have justified themselves.

saq: seconara Gunpowder Works in England , e

The first Storekeeper at Waltham Abbey was a remarkable character named James Wright. (A sergeant of that name was enrolled in the original Rocket Brigade whose first action was the battle of Leipzig in 1813.) As Storekeeper, at a salary of £150 per annum, he seems to have combined the duties of Superintendent, Administrative Officer, Chief Clerk and Assistant (Sci.). He submitted technical progress reports in the form of letters to his superior officers. "Sir, I beg to inform you of the result of this day's trial . . . Sir, I have the honour to transmit a Sketch for laying the pipes from the Gasometer for conveying the Hydrogen gas . . . also is enclosed an

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4.

Estimate of the expense for converting such means (as) we have by us to adapt the same to your Suggestion, as called for by the Board under date 16th ult...." A collaborator of Wm. Congreve Jnr. and Wright was Major By, the ill-fated founder of By Town, renamed Ottawa, Canada's capital city.

The descendants of James Wright served the Royal Gunpowder Factory throughout the 19th and into the 20th century. A grandson, Henry, packed off to Canada, restored his reputation and gained his return passage by quick thinking and bold action in persuading the General commanding the Quebec garrison (luckily for Henry the aide-de-camp was a native of Waltham Abbey too) to retract an ill-considered order to blow up the magazines when these were threatened by a spreading fire in the locality, and instead to get the troops to dig protective earthworks. As the magazines contained several hundred tons of powder the city was undoubtedly saved from disaster. Another grandson, James, emigrated to Mooresburg, Tennessee, U.S.A., and it has recently come to light (from information supplied by a previous Director of E.R.D.E., exiled, albeit meritoriously, in that far-off land) that the design of the Confederate Powder Works at Augusta, Georgia, owed, by chance, a great deal to inspiration from Waltham Abbey. Long after the Civil War was over, Colonel (General) George Rains, late of the Confederate Army, wrote: ----

"But one man—Wright—could be found in the Southern States who had seen gunpowder made by an incorporating mill, the only kind that can make it of the first quality; he had been a workman at the Waltham Abbey Government Gunpowder Works in England . . . I was much indebted to his knowledge and experience . . . I (also) came into possession of an invaluable pamphlet by Major Bradley the Superintendent of the Waltham Abbey Works; in this the entire process and machinery employed at that Factory—the best existing in any country —was succinctly stated; Drawings, or working plans, or details of the buildings, or apparatus, however, were not given."

On a small point of detail, Major Bradley was never Superintendent of the Works. The story is reminiscent of what

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happened in the first and second World Wars in which, notwithstanding the quantities of explosives manufactured, it could be asserted that Waltham Abbey's contribution in chief was expertise, in the persons of numerous staff and foremen who left to help the new Ordnance Factories get going in Britain and the Commonwealth, and in America.

From history to riverine, or brookish, geography. The Horsemill Stream which defines the western boundary of North Site, is a reminder of the earliest means of turning the stone wheels of the incorporating mills which mixed the solid ingredients of gunpowder. Horses were not finally dispensed with until well into the next century, but about 1730 Bouchier Walton, father of John (?), taking advantage of the fall of the land, constructed an artificial high level canal with mills spaced out along its banks, the water spilling over paddle wheels into the tail streams a few feet below on either side. This "new scheme" seemed to be threatened by contemporary Government action aimed at increasing London's water supply, but in the end the Act "for ascertaining, preserving and improving the navigation of the River Lea . . ." which passed the Lords in 1739 left sufficient water for Bourchier to work his mills.

With the expansion of the factory under Government control water again became an issue early in the next century when additional rights were acquired over the Lea extending from Nazeing to the Black Ditch (the latter is marked on the map of South Site). During the same period mills were built along Lower Island, south of Highbridge Street, which with its canal and the Lea forms a tenuous link between our North and South sites. Unhappily a succession of serious explosions, the last occurring on April 13th, 1843, by which seven operatives died, caused their abandonment. Steam power arrived in the mid-fifties and with it comparatively modern looking presshouses for gunpowder, one of which was recently demolished to make way for new electricians' workshops on North Site. Rather surprisingly a few of the old mills at the northern end of Walton's canal were converted to steam in the first World War. Nemesis overtook all of them in the second when a land mine from a German bomber dropped on Hoppit during the winter of 1940-41. From that moment gunpowder manufacture at Waltham Abbey virtually ceased. Following the

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extensive flooding of North Site in the Spring of 1947, it was decided to empty the "high level" canal by diverting it into the Horsemill Stream, dredging and widening the latter and regulating the flow of water by a weir. Visitors who stand on the bridge (not too many at once) connecting Hoppit with the new Conference Room and Library can view the old sluice gates which once held back the high level water, and the now quiescent pool that carried away the over-spill along the Millhead Stream. The process of disengagement from the past was completed in April, 1960, when the skeletons of the old mills were demolished, their cement and brick foundations sold for hardcore and the area bulldozed.

John Walton's old house (it has been added to) and pillared sundial in front stand on the far side of the Library from Hoppit bridge. It is occupied by the Editorial and Reports centre.

When Ministry of Supply came on the scene in 1945 metalled roads were non-existent apart from the approach to the main gate along Powdermill Lane which is as ancient as the factory itself. The network of canals and narrow gauge railways with their hand-trundled covered wagons had served instead. Access to many of the magazines was by water. Barges carrying cargoes of cordite were towed by horses down the Lea as far as the lock at Bromley-by-Bow, at which point they hoist sail for Woolwich. The "Lady of the Lea" made her last voyage from Waltham Abbey on May 20th, 1946. The present road circuits were not completed until 1957, twelve years after the takeover.

A reporter who had been on a conducted tour of the Royal Gunpowder Factory in the summer of 1898 wrote an account of it in the Strand Magazine. The following is an extract: ---

"... we walked miles; we plunged into thickets, crossing innumerable streams and occasionally gliding from one building to another in a swift electric launch, the panting of whose screw scared the birds and rabbits that abound in this extraordinary place."

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The launch referred to had been designed by Major-General W. H. Noble, R.A., Superintendent from 1885 until his death in 1892. Swiftness is not reckoned a virtue in an explosives establishment; powered by Grove cells, "The Spark" could make six knots, the batteries serving also to light up waterside buildings as the General went his rounds. The launch, alas! like the rabbits which used to be had at the back door of the Superintendent's house for a shilling, have passed into history. But the extreme tip of North Site —a magazine area—up along the Cornmill Stream by Newton's Pool to Fishers Green is an extraordinary place still, and despite occasional loud bangs from the Sensitiveness Section on New Hill, innumerable birds and waterfowl find refuge there; pheasant and duck, heron and kingfisher are often to be seen.

Since 1945, the plan has been, as far as practicable, to concentrate research on North Site and development activities on South Site. The individual buildings which compose the two prominent ranges flanking the eastern boundary of North Site were put up, some in early, some in late Victorian time, for gunpowder manufacture. The later ones—dates on the towers span the years 1877 to 1889—may also have been used for smokeless powder (guncotton, i.e., nitrocellulose), the art of producing which in acceptable stable condition having been mastered by that time. During the nineties most were converted to cordite manufacture. Today, they have all been transformed into research laboratories.

Quinton Hill Farm, purchased in 1886 to accommodate the first cordite factory in Great Britain, forms the bulk of South Site. In addition to the incorporating and press houses, drying stoves and other cordite processing buildings, provision had to be made for the manufacture of guncotton, nitroglycerine and nitric acid, and for recovering spent acids. The new R. & D. establishment inherited a vast slum, vestiges of which are to be seen in the Chemical Engineering area, slowly being tidied up, however, by demolitions and conversions. The new facilities on South Site for solid propellant development are a different kettle of fish, several buildings for special purposes having been constructed and many old ones converted to new uses. This is a major asset and in some respects unique.

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The spirit of scientific enquiry and invention has been a feature of Waltham Abbey since 1787, with ups and downs of course. The history of explosives technology abounds in the names of men who worked here. The Honourable Board of Ordnance were accustomed to consult members of the Royal Society on technical matters of difficulty (William Congreve, Jnr., was elected Fellow in 1812) and close ties with the academic world have persisted to this day. Having regard to the fog in which chemistry was enveloped early in the nineteenth century-Dalton's "New System of Chemical Philosophy" was first published in 1808-it is remarkable to find Storekeeper Wright expressing his results on charcoal "distillation" with complete lucidity. In 1801 a committee of the Royal Society visited the Royal Powder Mills to report on the possibility of electrostatic hazards arising from rolling wooden barrels on leather-covered floors and from the use of silken screens for sifting powders. Michael Faraday headed another investigating board after the fatal explosion on Lower Island in 1843.

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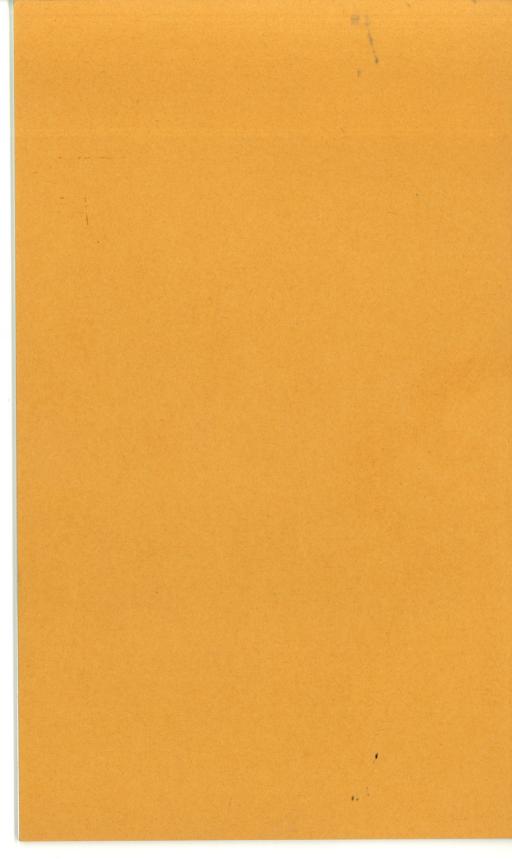
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Memoranda







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Mr. G. Bromberger

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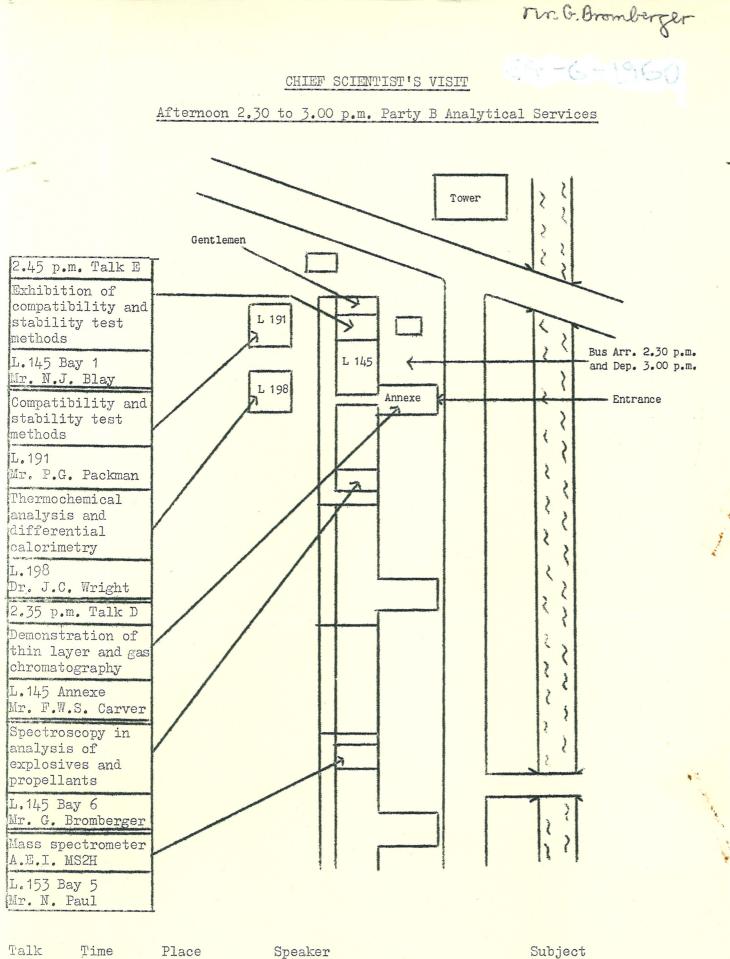
CHIEF SCIENTIST'S VISIT

8-7-1966

Afternoon 4.00 to 5.00 p.m. Party A Analytical Services and Materials 1

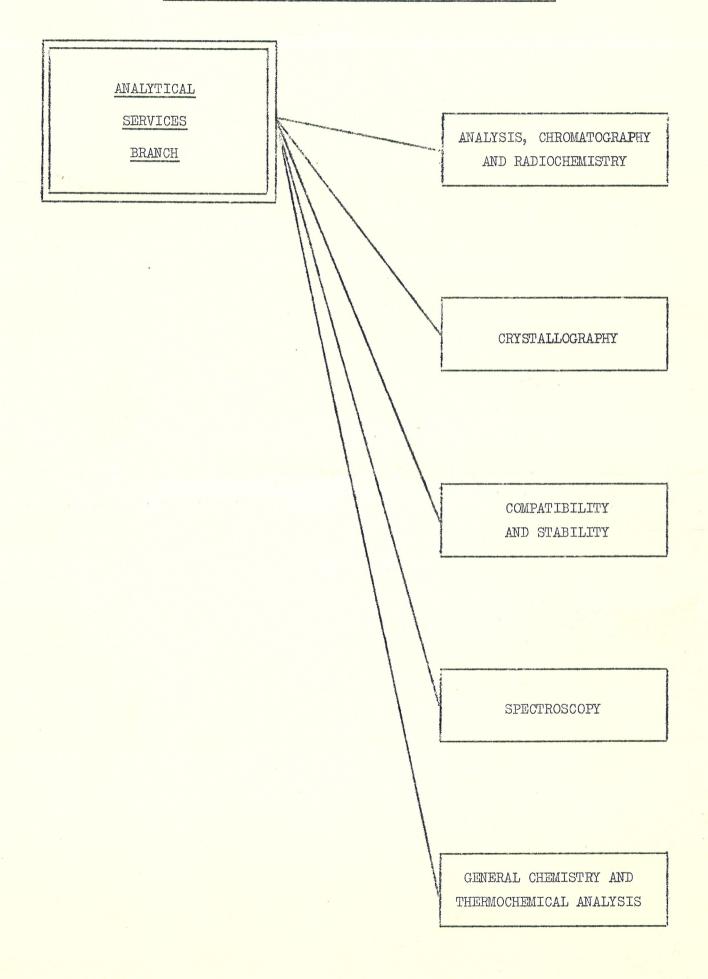
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| | 5 | | La della d | N.M.R. and I.R. Spectroscopy of |
| Gentlemen | | | | Polymers |
| | 1 | | | L.134 Bay 9 |
| Tower | RS | HINI | | Mr. G. Howell |
| 4.15 p.m. Talk E | R) | | | 4.40 p.m. Talk B |
| Exhibition of | 1.1 | ENTER | | Polymer chemistry |
| Compatibility and | 1 | LAN A | | L.134 Bay 9 |
| Stability test methods | - | H+1 | | Dr. J. Golden |
| | The | Bus Dep. 5 F | M | 4.30 p.m. Talk A |
| L.145 Bay 1 Mr. N.J. Blay | 12 | 1 HT | | Autoxidation of |
| Compatibility Bus | ? | | | Polymers |
| Arr. | R's | YV | N | L.137 Bay 2 |
| | 21 | | | Dr. N. Uri |
| L.191 | 1 | | V ` | Polymer Physics |
| Mr. P.G. Packman | 2 | 5 | N | L.134 Bay 6 |
| Thermochemical | 2 | | | Mr. E. Hazell |
| analysis and | RS | | $\langle \rangle$ | Joint Tropical |
| differential | 5 | | V | Research Unit |
| calorimetry | 3 | | N | (Australia) |
| L.198 Dr. J.C. Wright | 1. | | | L.134 Bay 5 |
| | 12 | | $ \rangle$ | Mr. D.J. Evans |
| 4.05 p.m. Talk D | | | | Thermal degradation |
| Demonstration of | 2 | | V | of polymers |
| thin layer and gas chromatography | R ₃ | | Λ | L.134 Bay 4 |
| L.145 Annexe | 15 | | 1 | Dr. A. Davis |
| Mr. F.W.S. Carver | 15 | | 1 | 4.50 p.m. Talk C |
| Spectroscopy in | R 2 | | | Polymer Applications |
| analysis of | L'L | | | L.143 Bay 1 |
| explosives and | | | | Dr. B.L. Hollingsworth |
| propellants | | | | |
| L.145 Bay 6 | 2 | | | |
| Mr. G. Bromberger | | | | |
| Mass spectrometer | 5 | | | |
| A.E.I. MS2H | { ? | | | |
| L.153 Bay 5 | 5 | | | |
| Mr. N. Paul | | | | |

| Talk | Time | Place | Speaker | Subject |
|------|-----------|-----------------|------------------------|--|
| D | 4.05 p.m. | L.145 Annexe | Mr. F.W.S. Carver | Application of Chromatography to Propellant Analysis. |
| E | 4.15 p.m. | L.145 Bay 1 | Mr. N.J. Blay | Compatibility and Stability Testing of Propellants and Explosives. |
| A | 4.30 p.m. | L.137 Bay 2 | Dr. N. Uri | Autoxidation of Polymers. |
| B | 4.40 p.m. | L.134 Bay 9 | Dr. J. Golden | Polymer Chemistry. |
| C | 4.50 p.m. | L.143 Bay 1 | Dr. B.L. Hollingsworth | Polymer Applications and Development. |



D Mr. F.W.S. Carver 2.35 p.m. L.145 Applications of Chromatography to Annexe Propellant Analysis E Mr. N.J. Blay Compatibility and Stability Testing 2.45 p.m. L.145 Bay 1 of Propellants and Explosives

EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT



Ministry of Aviation

Explosives Research and Development Establishment

Analysis, Chromatography and Radiochemistry (Analytical Services Branch)

The functions of the Section are to provide an analytical service for the whole of the Establishment, and to assist other Branches in specialised analytical applications which arise in their research. In order to do this efficiently, it is necessary to develop new analytical techniques and to have a wide knowledge of the activities of other Branches with a view to anticipating future analytical requirements.

Techniques involved are many, ranging from classical wet analysis to modern instrumental methods (e.g. polarography, spectroscopy and radiochemistry) and all forms of chromatography.

Equipment available in this, and other Sections of the Branch include

Cathoderay polarograph (Southern Instruments, K1000)

Analytical and preparative gas chromatographic equipment (Perkin Elmer Fractometer)

Standard and low background Geiger counters (Panax) Solid and liquid scintillation counters (Isotope Development Ltd.) Ultraviolet/visible spectrophotometers (Perkin Elmer 137 UV and Unicam SP 500) Double-beam infra red spectrophotometer (Grubb Parsons S3A)

Mass spectrometer (A.E.I. MS 2H)

Thermogravimetric balance (Stanton)

Craig counter current extractor

A large portion of the work is in support of Branches connected with production of propellants viz. P1, P2, and C.E. As new formulations arise, and new propellant ingredients are produced, methods for their complete analysis have to be devised. Chemical Inspectorate laboratories, I.C.I. departments and some Commonwealth laboratories engaged in propellant development work look to this Section for guidance in such matters.

Much effort has been devoted to the application of chromatography to propellant analysis. Thin-layer chromatographic methods have been devised for the rapid identification of the ingredients of propellants of unknown composition, and for the quantitative determination of residual stabilisers which are in common use in this and other countries. Gas chromatography is used for the quantitative estimation of propellant plasticisers and in a limited way, for propellant stabiliser determinations.

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In addition to propellants produced at E.R.D.E., Foreign propellants which are being subjected to Ordnance Board trials are submitted to the Section for complete analysis.

Liberal assistance is given by the Section to the Materials Branches, especially M.1. Purification of starting materials, separation of low molecular weight polymers, identification of unknown rubbers, and the examination of polymer pyrolysates are but a few of the varied requests received.

Staff

Laboratory and Exhibition

Building L.145 Annexe.

F.W.S. Carver, Miss A.R. Howieson, Miss J. Squire, E.J. Gallacher, F.I.H. Tunstall, D.H. Lee, R.J. Rapley, Miss C.A. Murray.

Ministry of Aviation

Explosives Research and Development Establishment

Crystallographic Section (Analytical Services Branch)

The methods of chemical crystallography are applied to the problems of the Establishment. The scope of the problems studied and the chief methods employed in their solution may be outlined as follows:

X-ray diffraction applied to single crystals and powders Crystal orientation Crystal structure analysis

These methods are currently applied in work on initiatory explosives, explosives chemistry, propellants and whisker crystals for use in high-strength materials. The problems are generally of a chemical nature, and concerned with the search for useful materials, the establishment of conditions of preparation and the study of degradation in use. Much of the work on whiskers is concerned with the determination of crystal orientation, since the physical properties of crystals vary with direction.

Equipment available in the Section includes three X-ray generators, one being a high-power rotating anode set, X-ray powder cameras, goniometers (Weissenberg, oscillating crystal and optical), an X-ray powder diffractometer and polarizing microscope with heating stage; the requisite ancillary facilities for preparation and treatment of specimens by chemical and physical means, including the measurement of crystal density, are also provided.

Much of the work on phase analysis and identification is accomplished by the use of X-ray powder methods, but single crystal studies are used in problems involving characterization, polymorphism and crystal orientation. The solution of some problems involves a determination of the detailed atomic arrangement in a crystal, in order to understand the chemistry involved, and in these cases also X-ray single crystal methods are generally employed. The crystal structure of tetrazene hydrobromide has been determined in this way and a model representing the results is displayed on the Analytical Services stand in the Library.

Most of the problems presented to the section originate within the Establishment, but some work is carried out for outside bodies e.g. other government establishments and commercial firms engaged on government contracts.

Staff

Laboratory and Exhibition Building, L.146, Bay 7

J.R.C. Duke B.W. Clements D.C. Mullenger

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Ministry of Aviation

Explosives Research and Development Establishment

Stability and Compatibility Assessment Section (Analytical Services Branch)

It is an obvious requirement for all explosives that their chemical stability shall ensure satisfactory performance and safety during their required service life, under whatever adverse conditions they are expected to encounter. In addition, materials brought into contact or proximity with explosives must not adversely affect their chemical or physical stability or sensitiveness to an unacceptable extent, or themselves be unduly affected by the explosives; i.e. they must be "compatible".

The Section is responsible for advising all those involved in the manufacture, inspection, storage and use of explosive stores in the military field and in weapon design, on these matters, and for carrying out the necessary tests. It is also concerned with the assessment of these aspects in foreign weapons considered for purchase by the United Kingdom.

In addition to work for other branches within E.R.D.E., the Section provides this service for many outside organisations including firms working on Government contracts (e.g. B.A.C., Hawker Siddeley, Ferranti, I.M.I. Summerfield) and other government establishments (R.A.R.D.E., R.A.E., R.P.E., D.C.I., D.I.Arm., Ordnance Board, Royal Ordnance Factories). It is also concerned with the standardisation of test methods with other NATO countries.

Apart from work of immediate applied interest the Section investigates problems of a broader nature, including at present the chemical reactivity of stabilisers in nitric ester based propellants, and factors contributing to the deterioration of lead azide in service detonators.

The methods employed in this work encompass both the old and the new. The well established methods of stability testing are widely used, particularly for compatibility work where assessments can often be made, with economy of effort, by considering the effects which a material has on the performance of an explosive in standard stability tests. On the other hand, considerable attention is being given to the application of more sophisticated thermal techniques, and much of the work, involving chemical examination of explosive mixtures after hot storage trials, is dependent upon the application of modern analytical techniques such as thin layer and gas chromatography and spectroscopic methods.

Staff

N.J. Blay P.G. Packman E.F. Pembridge J. McDougall B. Drake B. Stephen Laboratory and Exhibition Building L.145, Bays 1 and 2 Building L.191



Ministry of Aviation

Explosives Research and Development Establishment

Spectroscopy (Analytical Services Branch)

Infra red Spectroscopy

Infra red radiation refers broadly to that part of the electromagnetic spectrum of longer wavelength than the visible and shorter than the microwave region. Of greatest practical use to the organic chemist is the very limited portion of infra red radiation between 4000 cm⁻¹ and 660 cm⁻¹ (i.e. 2.5μ and 15μ). Radiation energy in this region is absorbed by the organic molecule and converted into energy of molecular vibration and rotation.

The plot of absorption intensity versus wavelength is known as an infra red spectrum and this is characteristic of any given molecule and therefore finds great use in identifying unknown materials.

Infra red spectroscopy is used to identify unknown compounds. Certain structural features give rise to absorptions at characteristic wavelengths.

| For example | | Asymmetrical NO ₂ stretching | Symmetrical NO ₂ stretching | |
|-------------|----------------------|--|---|-------------------------------|
| | | | | |
| aliphati | .c C-NO ₂ | 1565 - 1545 cm ⁻¹ | and | $1385 - 1360 \text{ cm}^{-1}$ |
| aromatic | C-NO2 | $1550 - 1510 \text{ cm}^{-1}$ | and | $1365 - 1335 \text{ cm}^{-1}$ |
| nitrates | 0-NO2 | $1665 - 1610 \text{ cm}^{-1}$ | and | $1300 - 1255 \text{ cm}^{-1}$ |
| nitramin | les N-NO2 | 1585 - 1530 cm ⁻¹ | and | 1300 - 1260 cm ⁻¹ |

Propellants and explosives of unknown composition are extracted and examined qualitatively by infra red spectroscopy.

Recently quantitative gas analysis has been performed on reaction mixtures of $SiCl_4 + H_2$ by observing the appearance of absorbtions due to $SiHCl_3$ and SiH_2Cl_2 . Results have agreed closely to those predicted by thermodynamic computations.

Quantitative analysis by infra red is carried out on esters, plasticisers, stabilisers and nitro compounds extracted from propellant and explosive compositions. A recent example is the determination of diethyl hexyl adipate and nitroglycerine in exudates from mortar-bomb cartridges.

Ultra Violet and Visible Spectroscopy

An ultra violet or electronic spectrum is the energy absorption pattern obtained when a compound is subjected to ultra violet radiation in the region $10 - 380 \text{ m}\mu$. The near ultra violet, accessible with the quartz spectro-photometer extends between $200 - 380 \text{ m}\mu$ and it is this region that is of most interest to the organic chemist. The wavelength of maximum absorption (λ_{MAX}) and the molar absorptivity (ϵ_{MAX}) are usually recorded and often presented in tabular form.

/Unsaturated

Unsaturated covalent structures such as ketones, aldehydes and aromatic compounds all show characteristic ultra violet absorptions. Nitro benzene for example shows absorptions at 252 m μ , 280 m μ and 330 m μ .

The use of ultra violet and visible spectroscopy in this laboratory is mostly restricted to quantitative determination of known compounds in solution. The determination of 2-nitro diphenylamine and lead dinitro resorcylate at 425 m μ and 395 m μ respectively being two of the main compounds investigated. Quantitative determination of styrene has also been carried out in connection with polymer degradation work.

Unknown materials are sometimes examined but as a general rule ultra violet spectra reveal fewer structural features than infra red.

Mas Spectrometry

Mass spectrometry involves the ionization and fragmentation of compounds, usually by bombardment with high energy electrons. The molecular ions and ion fragments obtained are accelerated by a high electrostatic potential and a separation according to the mass to charge ratio achieved by a magnetic field.

The information obtainable is of wide application and can be summarised into four main types:

1. Identification and determination of mixtures by comparison of the fragmentation patterns with known compounds.

In this way complex mixtures of gases and volatile compounds may be resolved. This technique has been used for the analysis of gases from propellant storage trials, the thermal and radiation degradation of polymers the volatile products from reaction studies and the combustion products of flames.

2. Identification and structure of compounds.

The molecular weight and molecular formula may be uniquely determined by an accurate mass measurement of the molecular ion. This is of great use in the identification of unknown compounds separated by the various chromatographic techniques.

A study of the fragmentation pattern will also help in the elucidation of molecular structure since the way in which a molecule is broken leads to an indication of the way in which the molecule is built.

A recent example of this use was in the identification of the suspected dimer of α -methyl styrene. An accurate mass measurement of the molecular ion gave the correct molecular weight and formula and the fragmentation pattern was consistent with the expected structure.

3. Isotopic analysis.

Because any molecules containing atoms of a higher isotopic weight will give peaks of a higher mass than the normal peaks the mass spectrometer is an ideal instrument for the detection and measurement of isotopic enrichments.

An example of the use of this type of work is afforded by the study of reaction kinetics of nitrodiphenylamine stabilisers using [¹⁵N] labelled compounds.

Staff

Mr. G.C. Bromberger Mr. N. Paul Laboratories and Exhibition Building L. 145 Bay 6 Building L.153 Bay 5

Ministry of Aviation

Explosives Research and Development Establishment

General Chemistry and Thermochemical Analysis (Analytical Services Branch)

Heat Flow Calorimeter

Low rates of heat evolution may be measured by determining the heat flow across a disc of low thermal conductivity. In our apparatus, developed by van Geel in Holland a teflon disc is sandwiched between a crucible containing the sample and a large aluminium block which acts as a heat sink. Embedded in the teflon is a 2000 junction thermocouple with cold junctions at one face and hot junctions at the other face of the disc.

The whole system is immersed in a thermostat. To reduce stray thermal effects a dummy sample holder and thermocouple are incorporated into the system so that only the differential e.m.f. between sample and reference is measured. The signal is amplified by a Keithley Model 149 high gain amplifier and fed into a recorder.

Our apparatus is now almost complete and with it we hope to be able to measure heat evolution from explosives and propellants at temperatures as low as 40°C. From these figures can be calculated safe storage times and radii for different propellants.

Differential Scanning Calorimeter

Differential thermal analysis depends on heating a sample and an inert reference in the same furnace at a constant rate and measuring any temperature difference between the two as a thermal change takes place. During the period of a change the sample temperature is of course not increasing in the same way as the reference.

In the Perkin Elmer Differential Scanning Calorimeter this difficulty is overcome by heating the sample and reference separately and varying the heat input to each to maintain a linear increase in temperature. The differential heat input is displayed on a recorder on which the peak areas are direct measures of the heat involved in the changes.

The equipment is being used to study the stability and compatibility of explosives and propellants, polymer glass transitions, determination of purity of some crystalline solids and the thermal breakdown of various organic and inorganic materials.

/General

General Chemistry

Work is being done on the degradation of stabilisers, in particular the oxidation of nitroso- to nitro- aromatics using [¹⁵N]labelled nitric acid. The reaction of diphenylamine with simple nitric esters is also being investigated as a prelude to more complex esters and stabilisers. Analytical problems posed by new oxidisers and fuels are being considered.

Chemical Hazard Appraisal

Attempts are being made to determine the degree of hazard of various chemical processes. An investigation has just finished into the preparation of the perchloric acid/acetic anhydride reagent used to determine the hydroxyl content of polyglycols and polyesters.

Micro Analysis

A microanalytical service using modified procedures suitable for explosive materials is provided for the establishment. In connection with this a thermometric titration apparatus is being developed for improved end point determination on the micro scale. Initially it is hoped to determine halogens by this method but it should be possible to extend it to other titrations which are difficult on the micro scale.

Staff

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