EXTENDING THE USEFULNESS OF MATERIALS

THE Materials Science Club held the third of its 1965 series of technical meetings on December 14 and 15 at the Atomic Energy Research Establishment, Harwell (see *Nature*, 206, 335; 1965). The meeting was divided into three sessions: the first was devoted principally to recent advances and future prospects in diverse fields such as ferrous and non-ferrous metals, ceramics, concrete, glass, paper, plastics and graphite. The second session covered general trends of development in materials science with particular emphasis on the inter-relationship between different fields. After a brief introduction covering the economics of new materials development, the final session was devoted almost entirely to a general discussion of the topics already covered during the meeting.

Recent advances in particular fields. The first material, graphite, was surveyed by Prof. A. J. Kennedy (the College of Aeronautics, Cranfield). Conventional polycrystalline graphites form a range of very varied materials the properties of which depend strongly on the origin of the cokes used as starting materials. The methods of preparation and purification of graphite can also profoundly affect the properties, as can be seen from the increase in density. That of conventional graphite lies in the range 1·5–1·9 g/ml.^{3}, recrystallized material is raised to 2·1, and the highly oriented pyrolytic form (deposited from the vapour phase) has a value of 2·2. The highest figure, that for a single crystal of graphite, is 2·26.

In principle, graphite is an outstanding material for high-temperature applications (with strength increasing to a maximum at about 2,700° C), and much effort has been devoted to developing new forms which might pay dividends in this respect. Graphites are characterized by a relatively high porosity—20–30 per cent is typical—and it is possible to achieve an improvement in mechanical and thermal properties by such techniques in impregnation and hot working.

The pyrolytic graphites are highly anisotropic, a feature which has both limitations and advantages. The fact that they possess high strength parallel to the crystal planes at 2,700° C, and that heat is rapidly and evenly distributed along the surface, makes their use in rocket engines and nose cones of considerable importance. The utilization of graphite in nuclear engineering and particularly in advanced gas-cooled reactors raises another singular feature of its behaviour, namely, its ability to reduce the internal stresses developed by irradiation through the mechanism of irradiation creep. The properties of graphite fibres, although they have only about half the mechanical strength of carbon fibres, are interesting, particularly at elevated temperatures.

In the discussion, attention was centred on the nature of the voids in graphites which apparently are normally small intercrystalline spaces (5–10μ). These, in addition to being able to be filled by impregnation, can also provide accommodation for creep. Delamination of graphite was found to occur because of the anisotropy of shrinkage resulting from the cone or circular type of growth exhibited in pyrolytic graphites. The high resistance to corrosion of graphitic materials was of value when used in the electrolysis of brine, and it was suggested that the particular orientation of graphite provided a system most capable of accommodating and resisting corrosion stresses.

The second paper, on "Steel Plant Refractories for High Temperature Processes", was given by Dr. J. N. Chesters (United Steels Co., Ltd., Rotherham). He began by summarizing the development of steel-making techniques from the original crucible process to the present-day electric arc furnaces. More than half the refractories produced in the world are consumed in steel-making furnaces. During the past decade there has been a radical change due in the main to the introduction of tonnage oxygen in the Bessemer-type of furnace. This has resulted in operating temperatures at least 100° C higher than normally used and concentrations of iron oxide many times those previously experienced. The use of high-current arc furnaces has also contributed to higher temperatures. Although material developments have arisen from work on atomic energy and rocketry, these have proved largely inapplicable to steel-making due to their instability under oxidizing conditions and their inadequate resistance to iron oxide. It seems that the development of suitable materials for furnace linings is very likely to come from the use of modified conventional materials; for example, in the use of denser materials of higher purity fired at unusually high temperatures. As a replacement for the conventional fire-clay and silica bricks, increased use has been made of basic refractories, particularly dolomite and magnesite. Many of these are used in the unfired state—frequently tar bonded—and it is increasingly certain that the carbon resulting from the tar plays a vital part in resisting slag attack.

Even the densest of these dolomite refractories are, however, proving inadequate for the worst conditions, and the latest swing is towards increasingly pure magnesia either in the fired or unfired state. Bricks made from relatively pure magnesia, fired at high temperatures, are also possessed of unusually high thermal shock resistance. A further decrease in porosity would be likely to improve this performance still more. Another major development has been the use of high alumina (85–98 per cent) bricks.

In the discussion which followed, Dr. Chesters instanced many cases of anomalous behaviour in refractory furnace linings, and pointed out that very little was really known about the atmosphere inside the furnace and the precise mechanism of attack. In one case, for example, a layer of special high-resistance material set in dolomite was found to have distorted considerably more than expected. The question of whether or not a better performance was obtained with fired or unfired materials probably depended on the relative conductivities and the penetration of the slag melt into the refractory. Graphite had been used as a furnace-lining material in the United States, but it was known that graphite burns rapidly in the presence of oxygen, and therefore some experiments had been done to reduce the oxygen concentration in the furnace atmosphere by replacement with nitrogen or steam. There was a suggestion that the considerable increase in temperature in certain areas of the lining leading to rapid breakdown was due to the influence of plasma jets which, of course, were capable of generating extremely high temperatures.

Mr. J. P. Proctor (Pilkington Brothers, Ormskirk) next gave a talk on "Glassy Materials". Three types of compound are found in normal glasses: (*a*) network-forming compounds which are oxides of such elements as silicon, phosphorus, boron and germanium; (*b*) network modifiers which are essentially alkali metals such as lithium, magnesium, caesium and barium; (*c*) intermediates such as the oxides of beryllium, aluminium, zinc and lead.

It was also possible to produce glasses containing fluorides, sulphides, tellurides, etc. In the laboratory at least, the properties of glass can be tailored to cover a wide range of specifications and the important factors in obtaining the desired result are: (1) chemical composition, and (2) the thermal history—important aspects of which are the transformation temperature, the strain release temperature and the annealing temperature.

Methods of improving the strength of glass include chemical toughening as exemplified by the replacement of small by large ions using diffusion processes. Strengths in the range 50,000–80,000 lb./in.^{2} can be obtained in this way. All glass-like substances are prone to the process of devitrification but, by the use of controlled nucleation with elements like gold, silver or copper, the crystallinity can be controlled, and this technique, together with chemical toughening, can provide glasses with strengths in the range 100,000–250,000 lb./in.^{2}.

Mr. Proctor showed how the use of silver halides in a glass of the sodium–boron–silicate type gave a photochromic substance which darkened on exposure to light.

Questions on the properties of different glasses revealed that it was possible to produce complex structures made up of layers of different glasses.

Some discussion followed on the use of slag/ceram compounds as building materials, and Mr. Proctor pointed out that it is necessary to control the crystallization in order to preserve strength. For this reason it was impossible to roll β-eucryptite glass without destroying the crystallites since any subsequent treatment always tended to alter the crystalline nature. It was apparently not possible to modify the surface properties of glass in order to reduce the amount of condensation which occurred in Britain's humid climate!

Mr. R. W. Nichols (Reactor Materials Laboratory, Culcheth), in his talk on "Recent Developments and Future Prospects for Ferrous Materials", said that a growing understanding of the mechanisms available for strengthening metals, coupled with new production processes which permit greater freedom of composition, is enabling the development of structural steels with higher strengths than the mild steel which they are beginning to replace. One method of strengthening steel is the introduction of additives such as carbon or other alloying elements which act as obstacles to fracture propagation. The proportions of these additives must, however, be kept to a minimum in order to avoid affecting the toughness and welding characteristics of the steel. A striking example of alloying developments is the series of maraging steels giving weld strengths of 90 tons/in.^{2} upwards with excellent weldability.

The important requirement of steels for high-temperature usage has involved work to determine the parameters controlling high-temperature strength and high-temperature oxidation resistance. In addition to employing alloying techniques, considerable gains can be made by the more efficient use of existing materials, as a consequence of a better understanding of the limiting failure processes. Control of the grain size can, for example, be achieved by the use of aluminium (as AlN) and niobium (as NbC). Further improvements can be made by increasing the purity of steel—most important is the reduction of sulphur—and also by the use of hot rolling techniques to break up the carbides into smaller particles.

After this paper it was decided to proceed without discussion to the joint contribution by Dr. A. R. Harding (Aluminium Laboratories, Ltd., Banbury) and Mr. R. W. George (Royal Aircraft Establishment, Farnborough) entitled "Aluminium and its Alloys". This paper, incidentally, provided an encouraging example of a joint approach by supplier and user. Dr. Harding described the present stage of development of aluminium alloys, showing how their properties and application are already well exploited, and pointing out the importance of the economic factors in deciding what materials should be developed and how they should be produced. He then showed how the present development of conventional alloys is governed by the need to satisfy conflicting requirements. Mr. George then discussed the improvement of fatigue properties in aluminium and its alloys and showed how increases in tensile strength obtained in the development of aluminium alloys have not been accompanied by proportionate increases in fatigue strength. From various metallographic studies it became increasingly clear that the plastic deformation resulting from cyclical loading can break down or destroy those micro-structural features which confer tensile strength. In attempting to develop an improved aluminium alloy, a material containing 5 per cent magnesium was selected, since this alloy, although of low tensile strength, has a relatively high fatigue strength. Improvements in tensile strength were attempted by further alloying without interfering with the intrinsic fatigue-strengthening mechanism of the aluminium–magnesium matrix. Various aluminium–magnesium–copper and aluminium–magnesium–zinc compositions have been made and tested and an aluminium–zinc composition was chosen as a working basis for further development.

Discussion on improvements in ferrous metals showed that again developments centred on the principle of limiting fracture by the use of additives, and by careful attention to control of grain size. The use of carbon, for example, could be tolerated provided the carbides formed were small and uniformly distributed; weaknesses were often encountered due to the lath-like shape of certain carbides. It was also observed that inter-metallics can occur as small brittle particles in the matrix, and this leads to fracture. Pearlite colonies can also contribute to fracture as these show weakness across the lamellae.

There was a suggestion that in view of the endless series of papers describing work on aluminium containing 4 per cent copper, very little progress has been made in commercial aluminium alloys over the past 10 years or so. Dr. Harding said that this surely indicated that aluminium–copper alloys cannot be extended into the more sophisticated requirements range and he felt that we had reached the maximum degree of improvement which can be achieved by purely empirical methods. It was time for science to have a look-in. Improvements in materials can be obtained by the trick of keeping some of the additives in solution so that they would be available for precipitation when the appropriate conditions were encountered.

The first day's discussions ended with a dinner attended by about fifty delegates at the Shillingford Bridge Hotel. After the meal the meeting was addressed by Dr. P. Murray (assistant director of the Atomic Energy Research Establishment).

Dr. Murray summarized the advances made in reactor technology at Harwell since its formation. The increasingly severe operating conditions demanded by these advances had made necessary the creation of a whole range of new materials with highly specific properties. These developments had been shared by a number of the divisions at Harwell and had, in fact, represented a truly 'materials science' approach to the solution of problems.

A vigorous discussion followed Dr. Murray's talk in which the economics of development on such a scale was often mentioned even to the extent of one member suggesting that the country and the tax-payer would be better served by closing down Harwell altogether!

The theme of "Recent Advances in Particular Fields" was continued at the start of the second day by a lecture from Dr. K. Newman (Imperial College of Science and Technology) on "Concrete". He remarked that the most useful properties of concrete are its ability to be moulded easily into any shape in the fresh state, and its high compressive strength and durability in the hardened state. Its main disadvantages are its low tensile strength, which has led to the use of mild and high tensile steel as reinforcement, and the considerable shrinkage it undergoes on drying out. Despite its widespread use little was known until recently about the structure of concrete and how this affects its physical and mechanical properties. The internal structures of hardened cement pastes, mortars and concretes were briefly described and their influence on stiffness, strength and the mechanisms of shrinkage and creep were explained. It was then apparent that considerable advantages can be gained in the use of concrete as a structural material by providing even a moderate, reliable tensile strength and a significant reduction in shrinkage. Several methods of improving tensile strength were proposed, including the use of aggregate of similar stiffness as hardened paste to reduce stress concentrations, the formation of chemical bonds at the aggregate–paste interface, and the introduction of small pieces of steel wire, glass or other fibres to arrest crack propagation. Most of the adverse effects of shrinkage can be overcome by more sensible construction techniques. However, drying shrinkage can be reduced considerably by sealing the concrete in impermeable surface finishes, by the use of expanding cements, or by steam curing at temperatures more than 180° C. A range of materials with properties between those of concretes and ceramics could be produced by autoclaving low water content, pressure compacted, concrete products.

In considering methods of reinforcing concrete, Dr. Newman during the discussion indicated that a large number of different additives had been put into concrete, including organic reinforcement; the only criterion to emerge from these trials was that for satisfactory performance the reinforcing material should be stiffer than the material into which it is placed. The presence of a large number of small cracks also served to increase the strength of concrete by acting as fracture terminators.

After this, Dr. H. Corte (Wiggins Teape, Research and Development, Beaconsfield) gave a talk on the development of paper and board. The early use of paper was confined almost entirely to writing and printing and it was only the application of mechanization, leading to increased production of cheap paper, which opened up new fields of application. These come into four main categories: (*a*) Packaging paper (such as disposable milk containers and multi-wall corrugated containers). (*b*) Sanitary papers (such as disposable incontinence pads). (*c*) Communication papers (such as '*NCR*' paper and electrographic copying paper). (*d*) Special technical papers (such as the eighteen different functional papers which are used in and for the motor-car).

In attempting to improve the performance of paper, three essential properties of the material must be borne in mind. These are: porosity, sensitivity to water, and strength.

Paper has a random porosity with a considerable variety of pore sizes. This scatter can be a disadvantage in such applications as oil filters, etc. Control of porosity can be done by various techniques including coating, which is often done for other reasons and also by the closure of pores as in the preparation of vegetable parchment by sulphuric acid or chloride ions. The mechanism of this latter technique is not yet fully understood but is being investigated. It is also possible to reduce porosity by means of resin impregnation. Multiple spark techniques can also be used to impose a controlled porosity, and, finally, the porosity can be further reduced or eliminated by lamination with plastics or aluminium foil. The sensitivity to water depends mainly on the presence of hydrogen bonds, and two techniques are available for increasing wet strength. Either additives which form bridges can be used, or alternatively, the incorporation of plasticizers (as in the wrappings of sweets) can be effective. Paper tears essentially by delamination, and improvements to tear strength are achievable by coating with latices or by impregnation with epoxy resins. A more advantageous fibre distribution (felting) can also be used but the technique is difficult.

Dr. J. P. Berry (Rubber and Plastics Research Association) next gave a talk on plastics. He began by indicating the close relationship between some of the materials called plastics impregnated papers by Dr. Corte, and visualized as paper reinforced plastics by himself! Plastics have many attractive properties but their complex mechanical behaviour poses problems in design. Inadequate understanding of these problems has tended to restrict the use of plastics to non-critical applications or has resulted in over-conservative and hence uneconomic design. In selecting a plastic for a particular use it is necessary to establish the factors which will render it unserviceable. After this the mechanical properties will determine the dimensions of the fabricated component. By achieving an understanding of the properties and limitations of plastics, and in particular the effect of time and temperature, more efficient use can be made of existing materials in present applications, and extensions can be made to new applications. There also will exist the possibility of developing materials with special improved properties.

In reply to a question on the use of paper fabrics, Dr. Corte pointed out that the principal difficulty was to achieve a satisfactory drape, and also indicated that the paper fabric-like non-woven materials were mostly composed of non-cellulose materials and were therefore expensive. Cheapness in 'paper making' depended on long runs, and the type of run used in textile production would be quite uneconomic. There was, however, a development in the United States of such disposable items made from paper as laboratory coats and hospital sheets.

Several members raised the question of ageing, odour absorption and unattractive appearance associated with plastic domestic utensils. Dr. Davies pointed out that articles which were cheap were often 'nasty' and both he and Dr. Berry emphasized that improvements were continually being made to plastics, and that it was also necessary to educate consumers in the choice of suitable materials. There was some discussion on the similarity of paper and wood, particularly in the case of some resin-reinforced products which Dr. Corte showed. The difference between wood and paper depended on whether or not the lignin was left in or removed from the product.

General trends of development in materials science. This session began by a survey of 'Fracture' by Mr. S. F. Pugh (Metallurgy Division, Harwell). Mr. Pugh plunged straight into classical materials science by observing that Galileo was wrong in assuming that the strength of materials was independent of dimensions, and remarked that Leonardo da Vinci found after experiments with iron wires of different lengths that tensile strength was dependent on the length of the specimens. It was suggested that a longer specimen contained more dislocations. A knowledge of fracture mechanisms may help both in the development of materials with improved fracture resistance and in improving engineering design techniques. Similarities and differences between the fracture characteristics of materials with as wide a variety of structures as mild steel and magnesium oxide were discussed. In developing a coherent science of fracture of materials, the subject can be divided in different ways according to the criterion adopted. So far there is no way of predicting which types of fracture a new material will suffer, because the critical factors are not known. Further investigations of fracture mechanisms are required, perhaps with more attention being paid to comparison of a given type of fracture in materials of widely different structure and physical properties. A complementary problem is in finding the relevance of the various fracture models to each particular type of fracture in each type of material. Mr. Pugh gave examples of the phenomenon of ductile fracture in glassy polymers subjected to prolonged stresses and said that a typical instance was to be found in stainless steel. He also dwelt on the use of reinforcing agents and, like Dr. Newman, indicated that it was often effective to set a crack to stop a crack, and instanced the poor bond between the reinforcing material and the resin in fibreglass as a means of providing a strong composite.

In the discussion it was observed that as materials were produced which had greater strength, the problem of brittleness became more acute and much of the work on improvement of materials lay in the reduction of brittleness. A technique was the provision of a multitude of crack-inhibiting centres. These can be either voids (as in the case of graphites) or particles and voids as produced in refractories resistant to thermal shocks by the incorporation of ground-up bricks in the clay matrix. Soft reinforcements in materials like concrete would also achieve the same end.

Dr. C. Edeleanu (Imperial Chemical Industries Materials Group, Billingham) then spoke on the development of engineering materials. The factors which have to be considered when making the critical decision of what material to use in an engineering application are: (1) What materials are available. (2) Costs—not of the material itself, but of the whole job, including processing and fabricating. (3) The time limit—whether or not the material can be developed and processed in the time available. (4) Human factors which include whether or not the material is pleasing in the particular application.

All these factors, but particularly the last, entail close co-operation between the designer, the fabricator and the user. It is also essential to understand the real and not the alleged requirement of a product, since often the customer does not really know what he wants. It was pointed out also that in many cases important developments occurred not in the material but in the development and use of economical ways of fabrication and production. Examples of this type of improvement emerged in the discussion particularly with reference to the manufacture of aluminium and the continuous process for steel fabrication.

Mr. J. M. Hutcheon (Reactor Materials Laboratory, Culcheth), in his talk entitled "A Plea for the Humdrum", said that although the development of new materials to the full scale in a very short time is possible, it is very expensive, and can only be undertaken under very exceptional circumstances. In most countries, for most of the time, the requirement is that the investment in research and development be kept to a reasonable proportion of the gross national product. There is, however, still ample scope for the materials technologist to use his influence with the engineer to ensure that materials specifications are rationally related to the proposed use; to investigate also the causes of variability within what is nominally the same material, and to seek means of improving existing materials by relatively small changes in the manufacturing route. This shifts the balance of research and development responsibilities away from the user laboratories into those of the manufacturer. Rapid test procedures are required, together with the detailed examination of every stage of the manufacturing process, and it is possible that some central organization such as the Ministry of Technology should finance developments.

The speaker also pleaded that university graduates should work in operating plants. He also pointed out that the statements made in his talk underlined the necessity for co-operation between user and manufacturer at the earliest possible stage of a project.

The final paper in this session was given by Mr. J. E. Gordon (Explosives Research and Development Establishment, Waltham Abbey). The speaker wondered whether the practice of cheapening a material in order to extend its market was making the most economic use of Britain's scarce research resources. Bessemer was said to have reduced the cost of steel by a factor of 5, but it is unlikely that the cost of mild steel could be reduced in the future by so much as 20 per cent. If this did happen, the value to the motor-car user, and perhaps the economy as a whole, would be no greater than a reduction of 1 per cent on petrol consumption. Reductions in the region of 25 per cent in petrol consumption could clearly be achieved by cutting the weight of the car. It is therefore arguable that steel is too cheap and if it were more expensive manufacturers would produce more efficient designs. Cheap materials may be as doubtful a blessing as cheap labour. Improved materials development could produce two benefits: (*a*) a more efficient product by weight saving, or (*b*) cheaper fabrication, since the cost of fabrication is nearly always much greater than that of the material.

The former point is receiving much attention, but the latter needs further consideration. Mr. Gordon believes that we may be nearing the end of the road with regard to cheapening existing metal-working processes, and we must begin to think more about moulding and casting processes. Some attention should also be paid to two-component systems, and here one can either increase the strength while leaving the density constant, or leave the strength unchanged and reduce the density. This could be achieved with steels, but not with some of the intrinsically lighter materials. An example would be the reinforcement of soft steel with hard ceramics; also, the quantity of wood consumed per year is about 10^9 tons compared with 4×10^8 tons of steel. Wood is cheap and has a similar modulus to steel, it has low density but does have other disadvantages. Could not more use be made of it? Plastics have too low a modulus to be used much as engineering materials, but might be modified. The use of very small quantities of reinforcement—less than 2 per cent—was found to give startling increases in mechanical properties. The discussion included remarks on the cost of training graduates, the type of training normally given to Ph.D. students and a suggestion that universities are far from being bands of learned men training intelligent machines. The only reason for research is to keep the teachers' minds alert in their task of machine training. It was also suggested that different fabricating techniques should be combined to provide new components based on design requirements and it was also contended that the policy of planned obsolescence, so prevalent in motor-cars, should be extended to things like houses so that the owner of a plot of land can replace his house easily and cheaply every 10 years or so.

Economics of new materials development. It was intended that most of this session should be devoted to a general discussion of topics raised in the Conference as a whole, and by way of a curtain raiser Mr. F. Roberts (Chemical Engineering Division, Harwell) gave a short talk on the economics of new materials development. He emphasized the importance of correctly allocating costs in any manufacturing operation and suggested that the important factors were: (1) raw materials, (2) power consumption, (3) capital costs, (4) reject rate and recycling costs, (5) degree of quality control (inspection), (6) scale of production (that is, (*a*) bench scale; (*b*) experimental or pilot scale; (*c*) large scale), (7) research and development charges.

In considering raw material costs it is necessary to consider the economic advantages of either starting with pure materials and ensuring they are kept clean during processing, or by starting with cheaper and less pure materials, and incorporating a refinement stage during the process. Turning to the question of continuous or batch production, Mr. Roberts emphasized that if continuous production were used it must be really continuous, since stoppages would inevitably affect the whole production system.

Finally, he emphasized the advantages of costing as early as possible in the development of the process.

Dr. Chesters began the discussion by pointing out that although labour costs were often the target for economy they were in many cases a small fraction of the total bill. For example, in the production of a ton of steel the total cost was £25, of which the main single item was £14 for scrap and only 7*s.* for labour costs. It is, therefore, better to improve techniques or to improve the quality and lasting power of such things as electrodes than to attempt to economize on labour costs. It was suggested that the number of different types of steels could profitably be reduced, but against this there was the position that the customer insisted on a variety to meet his own needs, and if this were not supplied would simply go to someone who was prepared to make it for him. There was agreement, however, that often the customer did not know what he wanted, and that there was a need for a closer investigation of requirements. Also, such matters as methods of test could easily be undertaken by joint user co-operation. If this co-operation were further extended and the users could be persuaded to do more research on requirements, it would then be possible to bully suppliers more successfully, and oblige them to provide new materials with properties which were really needed.

Some discussion ensued on the differences between the development of a new material and a new product. In the former case the development was usually for a specific purpose, whereas in the latter, having made the development, it was often necessary to search for a market. There were dangers attending on the change of manufacturing processes from, say, batch to continuous operation, since the product often possessed different properties, and it had been found in the past necessary in some cases to start from different raw materials.

Some surprise was expressed at the lack of satisfactory composite materials, and this was attributed to the fact that scientific method had only recently been devoted to the development of composite materials. These were very often very expensive in the early stages and also the techniques were often time consuming. However, it was felt that considerable advances in composite materials would occur over the next few years.

It is impossible to sum up in a few words a conference which covered such a wide field of materials and ideas, but a point which emerged very strongly—and which was commented on by a number of speakers—was the very keen interest displayed by lecturers and contributors to the discussion alike in economic matters. Even during a discussion on what the organizers felt would be purely technical matters, the conference was continually being reminded of the need to consider economics.

W. A. HOLMES-WALKER

IDENTIFICATION OF ELUTED COMPONENTS IN GAS CHROMATOGRAPHY

AN informal symposium with "Identification of Eluted Components" as the main theme was held by the Gas Chromatography Discussion Group of the Institute of Petroleum on September 23, 1965, at the Bodington Hall residence of the University of Leeds. The members were officially welcomed to the University by Prof. H. M. N. H. Irving.

The first paper, presented by Dr. R. P. W. Scott (Unilever Research Laboratories, Colworth House), summarized recent work on the development of a chromatograph that would operate intermittently, allowing a 15-min time interval to elapse after the elution of each peak.

Experience in the separation of complex mixtures had shown that simultaneous low-resolution mass and infra-red spectra were needed to assign identities unequivocally to peaks. This being so, a single chromatographic run extending to as long as 24 h with each eluted component being given the attention it deserved could result ultimately in a net gain in analysis time, particularly if compared with duplicate runs, one for high-speed low-resolution mass spectrometry and the other for trapping and regeneration of samples for infra-red examination. Tailoring the gas chromatographic and infra-red equipment to meet the preferred approach was the subject of the presentation.

Because of the time needed to make a normal infra-red scan, it was essential to be able to stop the chromatographic run for a 15-min period without adversely affecting the chromatographic resolution of components still in the column. This was found to be possible using a set sequence of stopping the carrier gas flow at the exit of the column, directly after trapping out a peak and releasing the column pressure at the inlet. Elution was re-started when required by opening the inlet and outlet valves and re-applying the column pressure evenly over a period of about 30 sec. This procedure has found extended applicability for the analysis of flavours using a 60-ft. packed column having a performance of 30,000 theoretical plates.

To obtain the desired mass of component in the infra-red cell a concentration step was required; this has been achieved by condensing each fraction on to a 10-cm column packed with 40 per cent 'Carbowax 200 *M*' at ambient temperature, from which it is automatically eluted rapidly at high temperature into the infra-red cell. A sixty-fold concentration is possible by this means. Simultaneously with the infra-red examination, a mass spectrum would also be obtained using a low-resolution mass spectrometer, such as '*M.S.* 10', which would give similar resolution to the normal fast-scan mass spectrometers but at less than one-fifth of the cost. For infra-red spectra, a normal '*SP*.200' infra-red spectrophotometer with a 15-min scan has been used for which 25 μg of sample was adequate, provided the cell was designed so that the sample was situated wholly in the light path. A suitable heated cell, plated internally to minimize adsorption of sample and with a total volume of 25 ml., was described and the determination of the spectrum of eugenol which has a vapour pressure of 30 mm at the cell temperature of 150° C was cited as an example of its use.

The whole sequence of events has been automated to such an extent that injection of the sample in the first instance is the only manual operation required.

The next paper, presented by Mr. L. Davies ('Shell' Research, Ltd., Woodstock Agricultural Research Centre, Sittingbourne), dealt with the problems of identification encountered in the determination of pesticide residues in crops, soils, etc., where the solvent extract can often contain very much less than 1 p.p.m. of pesticide together with large amounts of co-extracted material.

The electron capture ionization detector, which possessed selective response to certain halogenated compounds at levels below 10^{-9} g, had been used successfully with both planar and radial arrangements of anode to cathode using a d.c. mode of operation with oxygen-free nitrogen as carrier; undesirable ionization effects had been observed when using argon. The halogen sensitive leak detector in its several forms (Associated Electrical Industries, Ltd.) was also useful as a gas chromatographic detector at the nanogram level although to achieve this sensitivity with acceptable base-line stability the electrode potential required optimizing and the cell (types *HA* and *J* had been used) should be operated in a stream of nitrogen. The electron capture and leak detectors were used to complement each other, in so far as when tied in series they exhibited differing relative responses to certain halogenated compounds. This enabled some degree of certainty to be attached to identification and considerably reduced the possibility of mistaking a product of low electron affinity present in gross amounts for a trace of chlorinated compounds, as was possible when electron capture was used alone. Chromatograms illustrating this type of application were shown. While the linearity of response of the leak detector cell was about three times that of electron capture and the elements are inexpensive, a degree of electrical instability and the need for conditioning are features that have to be contended with. Mr. Davies pointed out, however, that the cells were being used for an application for which they were not designed.

For non-halogenated organo-phosphorus pesticides a sodium thermionic detector, adapted from a flame ionization detector by fusing sodium sulphate to a platinum wire collector electrode, had been the subject of investiga-