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The
Chemical Age

VOL LXIX 15 AUGUST 1953 No 1779

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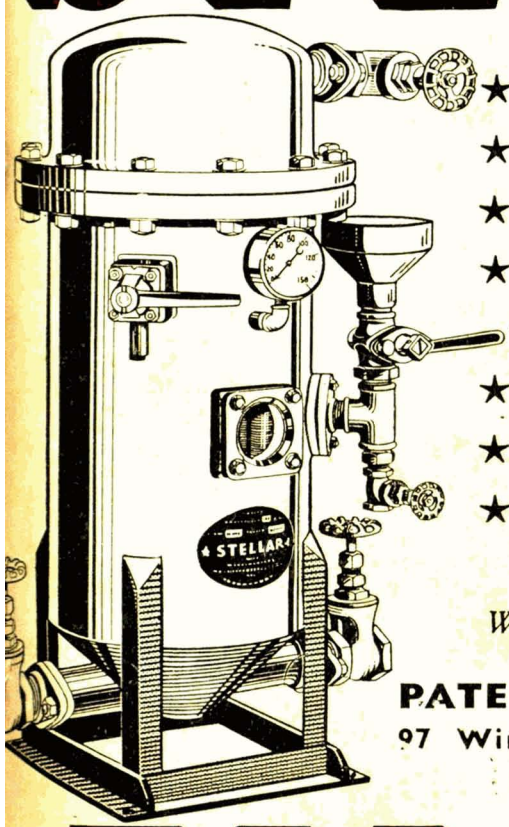

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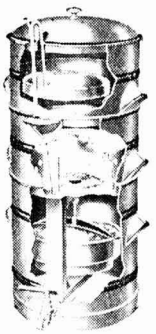

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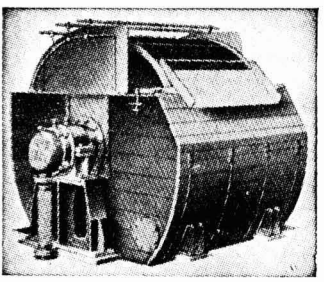

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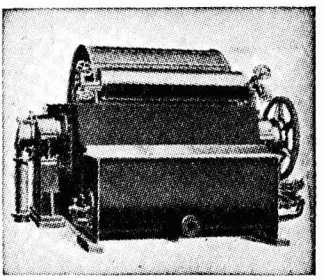

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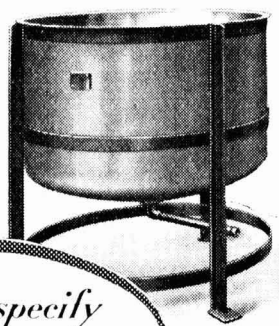

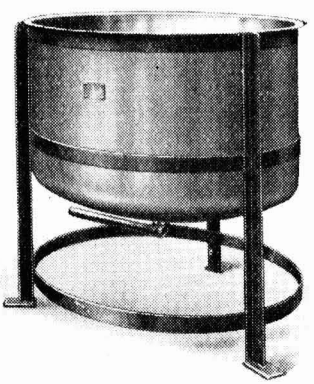

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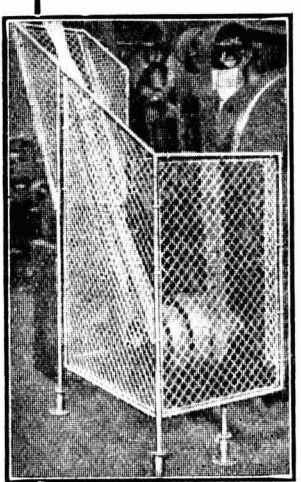

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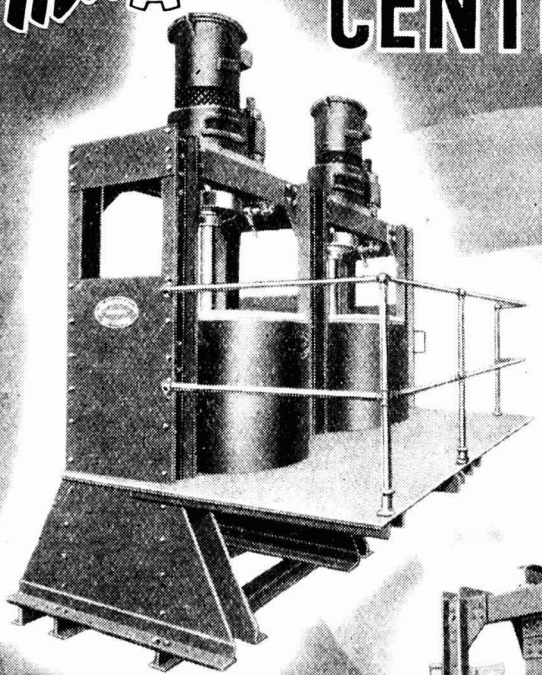

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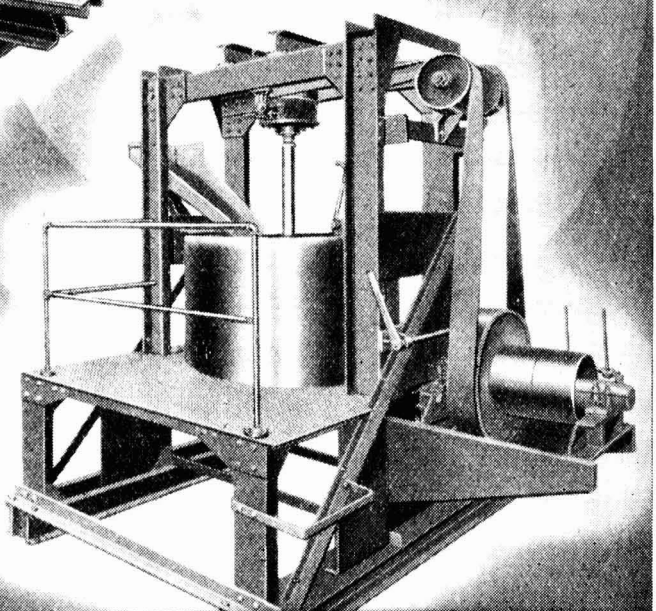

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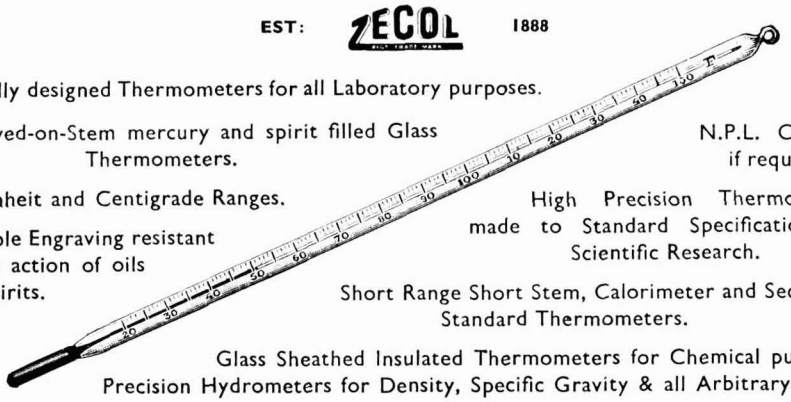

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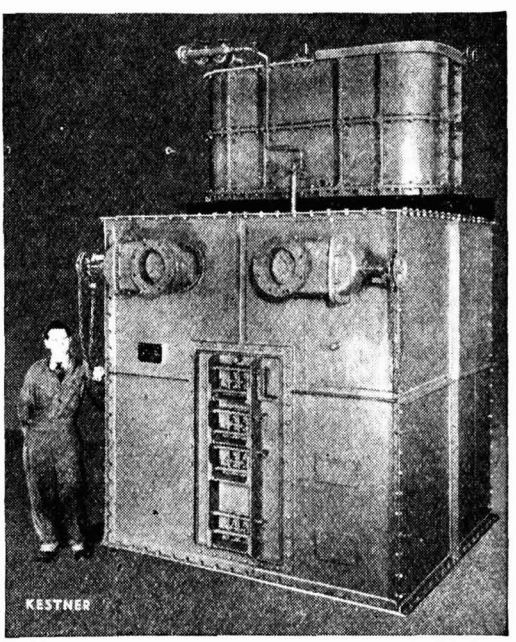

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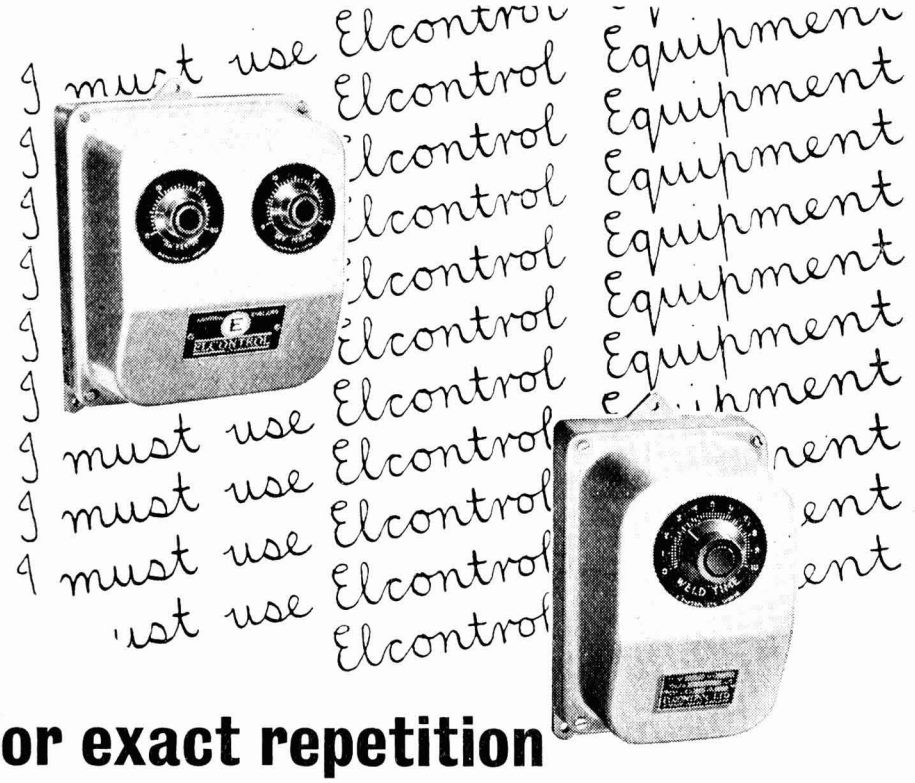

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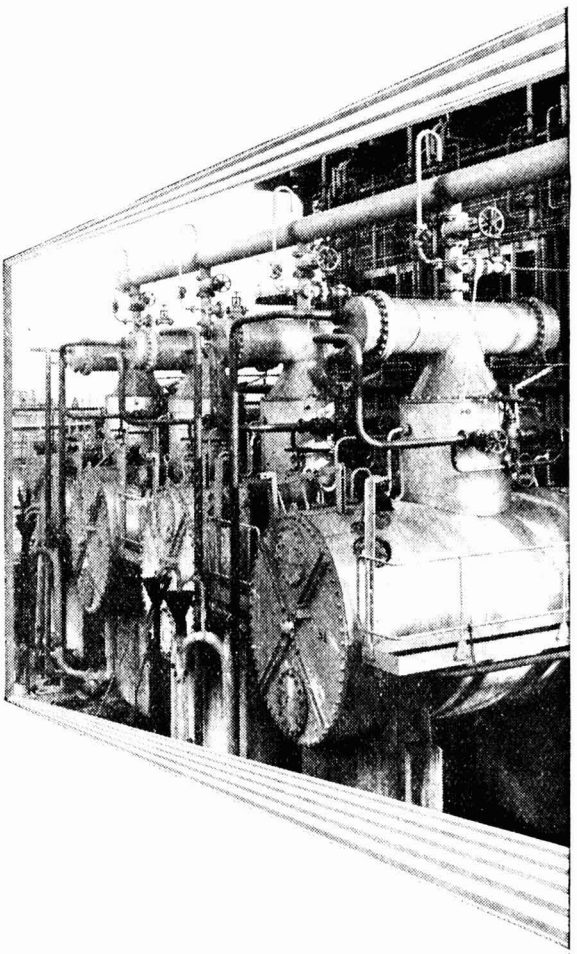

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Volume LXIX 15 August 1953 Number 1779

The Research Problem

SO much is said and written about research today—and commonly from a critical standpoint—that we are in some danger of forgetting that, like another modern shibboleth, 'productivity,' research is a sadly over-worked word. Of all aspects of the relationship between industry and science, it is certainly the most dramatic and colourful. Nothing succeeds like success, and naturally those research projects from which new and profitable processes or branches of industry eventually arise receive a much greater amount of publicity than the buried and sterile projects that produce mere costs and disillusionment. It has long been a standing indictment of British industry that it is reluctant to undertake an adequate amount of research. The more vigorous attitude of American industry is invariably cited in comparison.

The truth is that reliable and comparable figures for British and American expenditure upon research are not readily available. The industrial turnover of the United States of America is very much greater; so, too, is the cost of scientists. In consequence the sums reported to be spent there each year upon industrial research seem astronomical. We have never—at any rate in reasonably modern times—spent anything approaching the American amount upon research, and it is scarcely likely that we ever shall or should. Recently *The Economist* (1953, 5,735, 191-193) estimated that both countries 'devote to research just under 1 per cent of their gross national product.' But research costs are less in this country, so we should actually be getting a much better *pro rata* return. However, this is an inference that even the scientific Anglophile will find difficult to swallow.

The Advisory Council on Scientific Policy in their last Report (see THE CHEMICAL AGE, 18 July, p. 118) while stating that a much greater amount is spent by industry on research than before the war, said that this effort falls 'far short of the average level in the United States.' Here, however, the word 'average' is highly operative. For 'some firms in this country mostly among the larger ones, are quite as advanced and efficient in their application of scientific research as any in the world. The

trouble is that there are not enough of them . . . .' There is a point below which firms are not big enough to employ even one full-time scientist. This is not due to apathy, but to hard facts and common sense. Medium-sized firms have certainly not employed scientists sufficiently in the past and the same reluctance still exists fairly commonly, but, as has often been mooted in these pages, a wider use of experienced scientists as part-time advisers or consultants would be more fruitful than the accepted full-time employee system. Too many people whose experience is wholly of science in action within large organisations talk about the benefits of science in small organisations. When the turnover or volume of output is relatively small or medium-sized, the small scientific staff that can reasonably be afforded must waste time and potential ability upon many routine duties if full-time occupation is to be justified. Conditions and circumstances in, say, a company controlling four or five works from a single headquarters, are totally different.

To try to solve this problem we have, of course, the joint DSIR—Industrial Research Association system, an approach that has little counterpart in the USA. Splendid work at comparatively small cost has been done by the best, but it must be realistically admitted that the effectiveness of some of these research centres is seriously retarded by growing pains. Co-operative research by natural home competitors is a delicate plant to bring to harvest. The smallest supporting firms (who in theory should be the greatest beneficiaries) usually pay the least; the largest firms often have their own private research to finance as well and they nevertheless pay the most. The capacity of member firms to absorb benefits depends very considerably upon whether they themselves employ scientists. If it is true that research is a risky investment, it is a sound and attractive plan for the risk to be spread collectively; but when a research project succeeds, is collective exploitation as easy to arrange ? Or as attractive ? It is surely a basic fact that applied research is competitive.

In any case, research expenditure is only the beginning of investment. If its results are to be utilised, far greater investment in new plant must usually follow. It is fair enough to say that taxation relief favours expenditure upon research here as reasonably as in America, but the equality comes to a sharp halt at the smallest pilot-plant stage. New plant in the USA can be both built and written off much more quickly than here, and at least the home market for its output is more assured. Anglo-American comparison must take this major difference of industrial climate into account.

On Other Pages

The annual subscription to THE CHEMICAL AGE is 42s. Single copies, 1s.; post paid, 1s. 3d. SCOTTISH OFFICE: 116 Hope Street, Glasgow (Central 3954/5). MIDLANDS OFFICE: Daimler House, Paradise Street, Birmingham (Midland 0784/5). LEEDS OFFICE: Martins Bank Chambers, Park Row, Leeds, 1 (Leeds 22601). THE CHEMICAL AGE offices are closed on Saturdays in accordance with the adoption of the five-day week by Benn Brothers Limited.

Notes & Comments

Very Civil Service

THE latest report from the DSIR includes special mention of publicity measures. There is undoubtedly some justification for believing that the technical services of the DSIR and its various establishments would be more widely utilised by industry if in fact more was known about them. Evidence that active steps are being taken to promote an up-to-date public relations policy can be found today at any branch of Her Majesty's Stationery Office. The buyer of a DSIR report will probably also receive a post-paid reply letter form inviting comments. Opposite the space for comments he will find this persuasive appeal for co-operation: 'You have bought this report. Is it what you need? DSIR wants to ensure that its reports meet the need of as many users as possible . . . How do you use this report? For background information in relation to your work? For specific information? As a reference to the original publications of the Station? Or merely for possible future reference? If this report is going on the shelves of a library, who will use it? And how much will it be used? The form, style and contents of this report could be altered in future years. Have you any suggestions for changes which would make it more useful for you . . . ?'

A Bouquet

IT is always easy to hurl brickbats at official organisations. Here is a first-class opportunity to present a bouquet instead. This frank and personal approach of the DSIR to one important aspect of the public relations problem—the presentation of published material—could hardly be bettered. It deserves a full response from the buyers of DSIR publications; certainly a 'can't-be-bothered-with-any-more-forms' reaction would be churlish indeed. For what our own opinion is worth, we do not think that DSIR annual reports are poor examples of presentation. Unless they can be enlarged without making them unduly expensive, only minor improvements seem possible, e.g., better lay-out might give a more readable appearance to a text that must inevitably be somewhat condensed. The greater need in our view is for *additional* publications, possibly for shorter and more simply written versions of the reports aimed at the reader who is technically-minded, but is not a 100 per cent scientist or technologist. However, the right spirit is obviously in possession at DSIR headquarters, and it is refreshing to find a Government department prepared to meet the public more than half-way. Nothing but good can come of it.

Tallow & Grease

WHAT are known as 'inedible fats' in world trade may, with a little exaggeration, become also known as unsaleable fats. Tallow and grease are steadily being produced in greater annual amounts than markets can absorb. That is the position at any rate in the USA, the major producer and exporter, and it is mainly caused by the decline of soap in the face of synthetic detergent competition. Production there is at least twice the pre-war level; it has risen at a much faster rate than meat production, recovery processes having increased in efficiency and points of application. Since 1947, however, the use of inedible tallow and grease in soap making has fallen by approximately 30 per cent, no easy change to assimilate when it is considered that this single demand formerly accounted for more than 80 per cent of total consumption. Nor is there any reason to suppose that future tallow production—or at any rate productive capacity—will not increase.

New Uses?

SO far, the problem of an annually rising surplus has been kept within bounds by increased exports. Outside America detergents have not yet made such large inroads into the soap market, and USA tallow is probably the cheapest fat available for soap manu-

facturers. Exports have risen from 70,000,000 lb. in 1947 to 750,000,000 lb. in 1952, but the maintenance of this unprecedented level depends upon dollar availability in foreign countries and (in the longer run) upon the ability of soap to withstand the threat from detergents. Yet tallow at 4 or 5 cents per lb. (at production points) is a cheap organic raw material. It is surely not beyond the powers of modern technology to develop new and possibly large-scale uses for tallow. What might be called chemical re-thinking seems urgently needed. Twenty years ago finding new outlets for a large-volume organic material might have been formidable, but today an expanding heavy organic chemical industry exists and creates a potential market for all cheap arrangements of carbon, hydrogen, and oxygen atoms.

Dollar Problem

FOR some years ahead, however, the primary trouble for USA tallow producers will be dollar availability abroad. However inedible American tallow may be, it makes a useful contribution to the world food situation; every ton of tallow used in soap releases its equivalent in vegetable oils that are edible or easily convertible into edible fats. The dollar gap creates a problem that would not otherwise exist, or, more accurately, a problem that could easily and smoothly be solved in a world of freely exchangeable currencies. And if USA tallow stocks accumulate, the price will fall lower still, making tallow and grease recovery much less attractive economically.

Rothamsted Reports

THE latest annual report from the famous Rothamsted Station mentions some preliminary tests with an ammoniacal by-product from gas-works which has been made by a new process. It would appear that there is much less removal of impurities for it is described as a 'sulphate of ammonia' in which 69 per cent of the sulphur is sulphate but 12 per cent is sulphite and 18 per cent thiosulphate. In most experiments the fertiliser behaved effectively as a nitrogen supplier, although in two cases there were respectively signs of poor early response and burning of foliage. The fertiliser was said to have had poor mechanical condition—and this is an important aspect of any fertiliser. To quote from the report itself: 'If by-product fertilisers, difficult to apply or containing materials likely to be harmful, are to be used in agriculture, they must be offered at greatly reduced prices.' It would seem that the new process, which is not described, will not be able to help in solving the still sizeable problem of ammoniacal wastes at small gas-works. Using such waste liquors to ammoniate superphosphate, as recently developed on a pilot-scale by Scottish Agricultural Industries Ltd., seems much more promising.

Soil Conditioners

ANOTHER chemical topic raised by the new report is that of polymeric soil conditioners. Tests on 13 farms in 1952 showed no significant benefits from the application of one of these new products. Nor were benefits produced in three tests in forest nurseries. The verdict given is that 'improved methods of preparation and application must be devised before synthetic soil conditioners can be expected to improve crop growth under normal agricultural or horticultural conditions.' Since these products are far from cheap—judging their economics by current market prices—this preliminary opinion from Rothamsted is hardly a happy augury for their future in Britain. Perhaps it is too early to reach too adverse a conclusion, but high-priced commodities in farming or market-gardening must offer certain and substantial benefits if they are to establish any real demand.

Union and Wage Restraint

The preliminary agenda for the annual conference of the Trades Union Congress at Douglas, Isle of Man, early next month, includes resolutions from several of the smaller unions condemning wage restraint. One from the Chemical Workers' Union calls for 'a legalised minimum living wage linked to a revised and accurate retail cost-of-living index.'

The Production of Germanium

Important Developments in Great Britain and the USA

MENDELEEF'S striking forecast of the properties of *ekasilicon*, fifteen years before its discovery in 1886 by Winkler (who christened it germanium), has become one of the legends of chemistry, but even he could not predict the wide interest which has been shown lately in this element.

Winkler's source of the metal was argyrodite, a rare mineral, 4 $Ag_2S.GeS_2$, containing 5-7 per cent by weight of germanium. In 1918, a further source was discovered in the copper mines at Tsumeb in South West Africa. This was germanite, $Cu_3(Ge, Ga, Fe, Zn)(As, S)_4$, containing 6-10 per cent germanium and up to 2 per cent gallium. The deposits were until recently believed to be nearly exhausted, but a report appeared in the *South African Engineering and Mining Journal* the other week, announcing that a germanium recovery research programme, supplemented by work on the part of three major metallurgical research groups in the USA, had been begun at Tsumeb; and it is expected that the pilot plant stage will be reached within the next few months.

Germanium also occurs in zinc ores, and when the roasted ore is leached with sulphuric acid, as for the electrolytic process, germanium, cadmium, gallium and indium also go into solution. It was formerly believed that the presence of these metals seriously affected the deposition of the zinc, and they are precipitated from the leach liquors as sludge, by the addition of more zinc in the form of the powdered metal. Recovery of germanium from this sludge is practised in the USA (Jaffee, McMullen & Gonser, *Trans. Electrochem. Soc.* 1946.)

Germanium in Coal

The occurrence of germanium in coal was first noted by Goldschmidt and Peters (*Nachr. Ges. Wiss. Göttingen*, 398 [1930]; 141, 371 [1933]) during the spectrographic analysis of a sample of coal from the Hartley Main Seam in Northumberland; further samples from the same district were also found to have an unusually high proportion of the element. Up to .003 per cent of germanium and almost as much gallium occurred.

The interest of the late Sir Gilbert Morgan, then director of the CRL, was aroused, and an investigation into methods of extraction of these two rare metals was initiated (Morgan & Davies, *Chem & Ind.*, 717 [1937]). It was found that as much as 60 per cent of the germanium content of coal was lost when it was completely ashed, and attention was directed to the flue dusts, in which some of the volatilised germanium might be expected to have accumulated. Nearly 20 years previously, gallium and an 'unidentified element,' among others, had been found in a spectrographic examination of flue dust by Ramage and Hartley. (Ramage, *Nature*, **119**, 783; *Trans. Chem. Soc.*, **71**, 533, 1897, etc.)

Ideal Conditions in Gas-Works

It was found that ideal conditions for the concentration of germanium exist in gas-works: beginning with a coal of content up to .003 per cent, a dust may be obtained from the flues of the producer system containing possibly more than 1 per cent. Ash from Lancashire boilers, on the other hand, contains only a low concentration of about .003 per cent, as in the original coal.

The rare metals occur in flue dust probably as the lower sulphides or oxides, which are very volatile above about 700°. The composition of dusts varies considerably, depending upon the coal being burnt: some contain a high proportion of sulphate and arsenic, others silica, ferric oxide and alumina.

Morgan and Davies set up a pilot plant capable of treating 28 lb. batches of material at a time. The flue dust was treated with sufficient HCl to ensure that, after all reaction had been completed, the remaining acid concentration would be slightly less than that of the constant boiling mixture. The germanium tetrachloride, boiling at 84°, was then distilled off. The distillate contained a considerable proportion of arsenic trichloride, but by means of a second distillation in a current of chlorine, much of the arsenic was induced to remain behind as the less volatile pentachloride. Gallium was extracted with ether from the acid residue.

It was not until 1945, following the

development at Purdue University of germanium high-inverse voltage rectifiers, that the firm of Johnson, Matthey & Co., well known for their experience in metallurgical technique, began research into the production of germanium in commercial quantities at the request of the General Electric Co., who by this time had fully appreciated the importance of flue dust as a source of germanium. In a survey made just before the war, of the trace elements in British coals, the Fuel Research Station had shown that, except in South Wales coals, there is no marked variation in the proportion of germanium. There was thus a substantial tonnage of potential germanium available in the flues of gas-works throughout Great Britain.

The method of Morgan and Davies was found not sufficient to extract all the germanium from dust in a number of samples. The process developed by Johnson, Matthey & Co. (Powell, Lever & Walpole, *J. Appl. Chem.*, **1**, 541 [1951]), who had already some knowledge of the extraction of germanium from germanite, involved, first of all smelting the dust with soda, lime, copper oxide and carbon. Silica and alumina are removed as slag by the action of the soda and lime; copper, released from the copper oxide, collects gallium; and iron from the flue dust collects germanium. The result is a copper-iron regulus containing 3-4 per cent of germanium and 1.5-2 per cent of gallium —a recovery of more than 90 per cent germanium and about 80 per cent gallium.

The regulus is taken into solution by treatment with a stream of chlorine under a dilute solution of ferric chloride. The liquid is then distilled, and the condensate separates into two layers: the lower layer consists of germanium tetrachloride containing up to 20 per cent arsenic trichloride, and the upper of constant boiling HCl containing large quantities of $AsCl_3$, but little germanium. The lower layer is run off for further purification.

Separation Difficult

Germanium tetrachloride boils at 84°, and arsenic trichloride at 130°, but separation by distillation is difficult, since the vapour pressure of $AsCl_3$ at 84° is high. By careful fractionation the arsenic content may be reduced to 20 ppm., but this is a level of impurity very much higher than can be tolerated. Oxidation of the trichloride to the pentachloride, as in the process of Morgan and Davies, is also insufficiently efficient.

The arsenic content is finally lowered to less than 0.1 ppm. by refluxing for 12-15 hours through a column packed with clean copper turnings. This figure for arsenic has been checked at the Chemical Research Laboratory, employing As-76 as tracer.

In America, the most readily available source of germanium is zinc ore from the Tri-State area (Missouri, Kansas and Oklahoma), which contains from 0.01 to 0.1 per cent. Information is not readily available on the production of the metal, but the prime producers appear to be the Eagle-Picher Co., who have long been concerned with the smelting of zinc ores.

Germanium is won from Tri-State ore as follows. After normal grinding, flotation and roasting, the ore is sintered with a mixture of coal and sodium chloride. Germanium and cadmium form volatile chlorides which are condensed and then separated by distillation. As in the British process, the germanium tetrachloride is subsequently distilled from strong HCl and refluxed through copper turnings (*Chem. Eng.*, **59**, (4), 158 [1952]).

Further Treatment

The further treatments leading to production of the metal are very much the same in both countries. Purified germanium tetrachloride is hydrolysed to the dioxide by pouring into water; very cold water must be used, or the dioxide is obtained in a highly refractory form. The dioxide is generally sold by the prime producers, and their customers prepare the metal with a controlled content of impurity.

The element is produced by heating the dioxide in pure graphite boats at 600°-650° in a current of hydrogen until no more water is given off; the temperature must be kept below 700°, however, to avoid loss of the volatile monoxide. To fuse the metal into an ingot, it is heated to 1,000° in a current of nitrogen; hydrogen cannot be employed at this stage, since the molten germanium will absorb it, giving rise to blowholes on cooling.

Further purification of the element may be carried out by allowing the molten ingot to cool slowly from one end to the other, when fractional crystallisation will occur. About 90 per cent of the metal may be obtained by this method before the concentration of impurities becomes too high; the residue is

returned to the metallurgists for re-purification. The germanium obtained in this crystallisation is multicrystalline; it is quite suitable for transistors employing the point contact principle (see below), but not for those of the junction pattern.

To obtain single crystals the metal is melted and held just above its melting point at 960°. The surface of the melt is touched with a single crystal, which is then slowly raised, drawing a monocrystalline rod after it. This process is carried out in vacuum or an inert atmosphere of helium or argon, and by its means carefully controlled proportions of impurities may be added to the melt and thus incorporated in the crystal. As little as 1 part in 10^{11} will detectably affect the electrical properties.

The properties of germanium which have been responsible for its sudden rise to fame are those of a semi-conductor, and, in order to appreciate the reasons—and the processes—for the incorporation of impurities into the crystal, it is necessary to appreciate the causes of semi-conduction. The same phenomena are explained in different ways on both the electronic and the quantum mechanical theories, but the former is perhaps the more readily grasped.

The germanium atom is tetra-covalent and in a perfect crystal each atom is located in a regular lattice, with nearest neighbours at the corners of a tetrahedon, as in diamond. Normally, the covalent bonds keep each atom firmly in place, and the crystal would be non-conducting.

n- and *p*-Type Conductors

Suppose, however, that an atom of arsenic, with five covalency electrons, replaces an atom of germanium at one lattice point. Four of the electrons will form valency bonds with the four neighbouring atoms, but the remaining electron will orbit loosely about the arsenic, and may move easily under the influence of an applied electric field. This is the cause of an '*n*-type' semi-conductor, so-called because the current is due to the movement of the negatively-charged electrons.

If a tri-covalent atom, such as boron, is introduced, the result is the opposite. The boron effectively represents a 'positive hole' into which electrons will jump from neighbouring bonds, giving a movement of the 'hole' in the opposite direction to that in which the electrons move. This type of semi-conduction is therefore called '*p*-type.'

The successful explanation of the action of transistors requires the postulation of the existence of both *n*- and *p*-type conduction in the same crystal. The quantum mechanical theory, very briefly, is as follows.

For the treatment of electrical and spectroscopic properties it is sufficient to make sweeping approximations and plot only one potential energy curve for each dimension of the complete solid. If the ground states of the individual atoms are g-fold degenerate, then, on the approach and interaction of N atoms, g^N levels will be produced in the ground state and we shall get, in effect, not a single bonding curve, but an energy band, part of which will be antibonding.

Potential Energy Bands

In the case of non-conductors, some of the interactions are forbidden, and the result is that the potential energy curve is split into a number of narrower energy bands, some of which are bonding and some antibonding. In semi-conductors, the presence of impurities, cracks, edges or lattice defects will produce single isolated levels lying between ground levels and the conducting (antibonding) levels.

When the energy distance is not too great, excitation of an electron from the ground state to an impurity level, or from an impurity to a conducting level, may occur. In the first case, *p*-type conduction occurs in the ground state, and in the second, *n*-type conduction.

Thus impurity levels may be caused by purely physical effects: a *p*-zone may be induced in *n*-type germanium by the application of pressure—*ca.* 200,000 psi.—or *n*-germanium may be converted to the *p*-type by heating to above 500° and quenching.

Because of its typical semi-conductor properties, germanium was first used as an ordinary crystal rectifier or diode, a great advantage being the very high voltage which it would withstand in the reverse direction. It was not until 1948 that Bardeen and Brattain, of the Bell Telephone Laboratories (*Physical Rev.*, **75**, 1208 [1949]) discovered that if two metal points are arranged to contact a wafer of germanium, very close to one another, the current in one contact is affected by the voltage in the other.

If a negative voltage is applied to one of the electrodes (the 'collector') then the current to it can be varied by changing the

positive voltage on the other (the 'emitter'). The germanium, in fact, is operating as a triode valve.

Consider the positive contact. Electrons flow from the germanium to the electrode, but in addition positive holes flow from the electrode to the germanium. The total current flowing will be due to the sum of the two, and if the concentration of holes at the point of contact is greater than the concentration of conduction electrons, a hole current will predominate. This concept of the emission of holes by the electrode has given rise to the name of 'emitter.'

If a negatively biased electrode contacts the germanium at a point not too far distant from the emitter, then the positive holes will flow into it, and therefore it is called the 'collector.' Thus the current in the collector is directly dependent on the emission of holes by the emitter.

Junction Triodes

Several types of point-contact triodes (or 'transistors') have been evolved, but the latest design is the junction type. In this, a thin positive layer is produced between the two negative ends of a monocrystalline bar, by the introduction of a small amount of impurity. The impurity is diffused into the lattice at the required point by the application of heat and alloying action. The whole crystal is embedded in plastic, and contacts from the three regions are brought out as 'tails,' giving a very rugged construction.

Transistors have a number of advantages over triode valves. They are very much smaller — a junction type occupies about 1/400 cu. in., while the smallest miniature valve takes up about $\frac{1}{8}$ cu. in.—they are rugged, need no warm-up, require infinitesimal power—a valve requires 1 watt to amplify a signal of 10^{-6} watts, but a transistor requires only 10^{-6} watts—and there is no filament to burn out. On the other hand, transistors are noisier than valves, have very much lower upper frequency limits (junction: 3-5 Mc; point contact: 70 Mc; triode valve: 60,000 Mc), cannot operate at high ambient temperatures, and have outputs limited so far to about 2 watts

Production figures are, of course, difficult to obtain. In Britain, production of a suitable grade of germanium is not yet sufficient to meet domestic requirements. When sufficient is produced to enable exports to begin, the very favourable comments passed by American experimenters on the superiority of British germanium to that produced in the USA encourage the hope that a market may be found there.

It has been estimated that the demand for germanium in the USA will be 15,000-20,000 lb. a year by 1955, and 40,000 lb. by 1956. The production in 1948 was put at about 1,000 lb., and in 1951 at 5-6,000 lb., with an emergency potential of 15,000 lb.

A year ago, Eagle-Picher quoted prices of $340 per lb. for the metal, and $142 per lb. for the dioxide. Current British prices for the highest purity dioxide are about 3/1¼ per gm., which is just about the same as the American price, at current rates of exchange. American transistors cost $15-20 about a year ago, at a time when they were put together manually under a microscope, and production was 200-300 per day.

The demands for germanium will undoubtedly increase many-fold in the near future, and it is heartening to know that Britain is holding her own, both in the chemical and the radio-engineering aspects of the development of this new material of progress.

Oil Sulphur Removal

A NEW process briefly referred to in a recent issue (p. 185), for the 'hydrodesulphurisation' of oils, has been developed in the Amsterdam laboratories of the Royal Dutch/Shell Group. It differs from the present vapour-phase procedure in that hydrodesulphurisation is effected by the 'trickle' technique, so called because the feedstock trickles over the bed of catalyst in the presence of hydrogen under pressure.

The oil, after meeting a circulating stream of hydrogen, is heated and the mixture passed over a fixed bed of catalyst (cobalt molybdena on alumina). Reactor pressures are of the order of 300-750 psi. and temperatures 340-400° according to the nature of the feedstock. A sulphur reduction of 85-90 per cent can be effected with very little attack of the cracked or highly aromatic components present.

Since the feedstock does not have to be vaporised, the new process results in considerable economies in heat transfer equipment and fuel, as well as in hydrogen compressors, pipelines and valves. It is expected that regeneration of the catalyst will be only infrequently required.

Ultrasonic Testing of Chemical Plant

Methods Developed by W. J. Fraser & Company

IN the construction of modern chemical process plant the use of welding has become indispensable and its use steadily increases. In high-pressure vessels the strength of a weld is often a matter of life and death and both user and insurance company must be thoroughly convinced that the weld is completely free from flaws. Ultrasonics, as applied to welded pressure vessel inspection, has recently become a technique which is receiving serious attention from both manufacturers and inspecting authorities, but few people have devoted so much time and effort towards developing a suitable technique as W. J. Fraser & Company, Limited. This firm of chemical engineering contractors have carried out a considerable number of investigations in their own laboratory and works at Monk Bretton, near Barnsley, Yorkshire, and they have now developed a method which is capable of practical application.

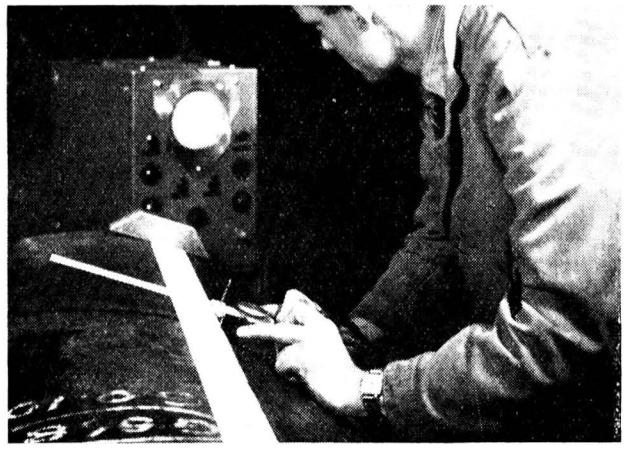
For some considerable time the X-ray testing of welds in plant designed to withstand pressure or vacuum has been fairly common practice. This method, however, involves a large number of X-ray photographs; is slow, relatively costly and does not readily lend itself to the complete examination either of plates before welding or of the welds themselves.

A number of years ago the application of ultrasonics resulted in a new technique, but progress has been almost entirely confined to the ultrasonics detection of cracks in shafts and bars and defects in simpler types of castings and standard forgings where a routine procedure can be laid down, suitable for rapid application by ordinary operatives after a period of training.

In the case of continuous plate and weld examination, however, conditions cannot be standardised and progress has been much slower. W. J. Fraser & Co. became convinced that the problem was not hopeless and their metallurgist at Monk Bretton, Mr. Richardson, began an extensive research programme under the direction of Mr. C. M. Auty, the director responsible for process development. As a result of their faith in the principle and their industry and perseverance, Frasers are today able to inspect, ultrasonically, inclusions of quite small size and thickness and also to detect rapidly any lack of continuous bonding in clad plates.

In addition, a considerable amount of work has been done on the continuous examination of welds and it has been possible to begin standardising a procedure for the rapid routine examination of certain classes of welds. This is a complicated subject and has necessitated the design of special equipment in many cases. In this connection Frasers have had the full co-operation of the designers of sets, W. S. Atkins & Partners, Kelvin Hughes, and Glass Developments.

Although most codes of practice do not



An operator making a routine inspection of a weld; a general view of the equipment involved

yet officially recognise the use of ultrasonics for weld and plate inspection, Frasers have been able to prove to both inspectors and customers that their ultrasonic method of inspection is a further safeguard of quality. In fact, during the visit by the scientific and technical Press on 17 July, flaws were quickly and definitely located, by means of ultrasonics, which were barely visible either under the microscope or in X-ray photographs.

Echo Pulse Reflection

The application of ultrasonic waves to flaw detection in metals is mentioned in a German patent as early as 1931, but present day methods working on the echo pulse reflection principle were developed about 1942 when Sproule, in this country, and Firestone in the USA, working independently, evolved this method in preference to the older 'Pohlman' or shadow techniques. The equipment at that time could be used for the testing of plates for various defects, but the examination of butt welds presented a number of serious drawbacks. The development of the 'Perspex' probe about 1948 made possible the use of shallow angle beams and is the basis of most butt weld inspection employing ultrasonics.

In the construction of modern chemical process plant, the use of welding as a method of fabrication continues year by year to grow and with the increasing complexity of materials and method incorporated, the demands made become more arduous. Unfortunately, more testing demands increased time with subsequent increases in overall cost and, therefore, before a new method of testing can be accepted as necessary, it must justify its use either by being unique in its application or easier, quicker and cheaper to apply, with results comparable to those obtained by other methods. Although ultrasonics cannot be claimed as a panacea for non-destructive testing, in recent years it has come to be regarded as an extremely useful tool in inspection processes. Its application as a quality-controlling device for steel plates and butt welds in chemical plant is finding increasing favour.

The presence of laminations and other planar flaws—e.g. rolled out slag inclusion of varying size in steel plate—is a relatively common defect found in works practice and when contained in the body of plate material is not easily detected. The application of ultrasonics to this type of defect has met with considerable success, particularly in the examination of plates thicker than $\frac{1}{8}$ in.

The principle of application using echo pulse reflection apparatus consists of using 30° transmitting and receiving probes on the same side of the plate under examination. As the pulse of sound is passed into the material, any difference in path length of the sound wave is noted by the cathode ray tube in an alteration of the position of the leading edge of the returning echo on the screen.

Care must be taken in adjusting the sensitivity of the receiver and the gap between the probes to give a sharp edge to the echo. If the power used is too high a shoulder will tend to form, giving the appearance of an echo from a flaw, and if too low the bottom edge will not rise sharply from the base line, making a shift of the echo or the formation of a shoulder difficult to see.

The use of a stepped plate giving areas of plate of varying thickness enables the position of each corresponding echo to be marked (a piece of gummed paper or a cardboard mask serves very well). The gap between the probes should be adjusted experimentally, so that the shift of the echo is clean over the whole range of depth at which faults are likely to be met in the plate under test.

Choice of Frequency

The practical application of this principle shows that although frequency (within limits) has little effect on the ability of the beam to detect planar flaws, it has considerable effect on the screen picture obtained. The 'trigger blip' and 'echo blip,' are pulses or bursts of short duration, each being an envelope of energy from the high frequency oscillator, and the number of peaks visible in the sawtooth edge of the echo by the high scanning speed depends on the frequency. At a frequency of 1 Mc. the peaks are few and give a ragged edge to the extended blip, whereas at 5 Mc. the picture is a fine sawtooth and conducive to clear reading of the screen. For this reason Frasers favour the use of 5 Mc. frequency together with appropriate coaxial cables for this frequency.

If the defects considered detrimental are relatively small (say $\frac{3}{8}$ in. dia.) then considerable time is spent inspecting a large plate and although rolled out inclusions of this size can be detected, it is doubtful in

many cases if their presence can be considered unduly harmful. Generally, the larger the acceptable fault, the quicker the inspection can be carried out. Inspection of important parts of the plates is generally more thorough, e.g. clad plate, where knuckle radius of a dished end would occur, or the areas immediately adjacent to weld preparations.

Size of Fault

The size (plane surface area presented to the beam) of a fault has an important effect on the form of the signal received. The large echo received from the bottom of the plate is the result of reflection from a surface, effectively that of the cross sectional area of the beam. A small planar fault, say circular and about $\frac{1}{4}$ in. dia. close to the bottom surface of a 1 in. plate, and parallel to it, would intercept the beam and return a portion of it to the receiver crystal sooner than would the bottom of the plate, but the magnitude of this portion of the beam is considerably less than that from the bottom. The size of the small flaw may thus be estimated.

The closer the fault lies to the bottom of the plate, the less the relative shift of echo along the time base and the extent of this shift gives an indication of the depth of the defect.

Over the whole depth range the method is not consistently accurate since there is a triangular blind spot between the probes in which a small fault could lie and not be reached by the 'beams.' As the probes slide over a fault which is small and too close to the surface to give a distinct reflection, it does cut off part of either the transmitted or reflected beam. This has the same effect as loss of probe contact, but rescanning with extra pressure will soon ascertain the presence of a defect. In practice it has been found that faults less than $\frac{1}{8}$ in. from the surface must be located by this means.

The method developed at Monk Bretton is applicable in straightforward manner to composite plates (e.g. stainless clad plates). Where lack of bonding exists, the very thin discontinuity acts as a reflecting surface, as in the case of laminations and inclusions. In testing clad plate, probing is best effected from the mild steel side since bonding is then furthest from the probing surface and small patches (for instance $\frac{1}{4}$ in. dia.) of unbonded plate are easily detectable. Equipment which has recently become available enables scanning to be done from the clad side. This is an important development, as the clad side is in better condition for obtaining good contact.

Fully comprehensive scans of plate where all sizes and depths of defects are noted is very tedious and rarely called for in practice, but once the tolerance, size and distribution of plane defect has been established, a probing sequence or pattern can be adopted where the location of faults of that size can be assured. On a good surface, moving in zig-zag pattern, a square yard of plate can be inspected in about 15-20 minutes, dependent on the size of defect sought and the ease with which the set can be moved over the job.

Where loose scale adheres to the plate it must be removed, but firmly adhering scale or simple rusting presents little difficulty. Normally a thick grease which forms a tough film and allows the probes to ride easily over the surface is used, but easier sliding can be obtained by using sheet plastic in suitable thickness with light oil on both sides.

The application of ultrasonics to butt weld inspection, viewed from a theoretical standpoint, would appear to offer an extremely valuable and ready method of inspection. However, it is impossible to generalise on the value of ultrasonic weld inspection as a testing technique.

Limits of Accuracy

The exact nature of the inspection to be performed and the limits of accuracy required must be defined. Failure to do this results either in dismissal of the technique as being erratic or the excuse that only with further development should the method prove useful. Two of the more important factors which retard the acceptance of ultrasonic weld inspection are (a) it relies on the integrity and skill of the operator and is therefore an applied art; (b) the difficulties of maintaining a constant and reliable surface contact are considerable.

In general, ultrasonic weld inspection involves the use of two probes. One probe transmits a beam of high frequency mechanical vibrations which, reflecting at an acute angle between the surface of the plate, irradiates all the material in its path. Homogeneously fine grained materials will not

cause it to deviate appreciably but a discontinuity, such as a fault, will reflect the sound just as a mirror would reflect a beam of light.

The detection of faults depends upon the reception of this reflected part of the original beam by a second probe and its electronic conversion to a visual 'blip' on the cathode ray screen. The shape and position of the blip on the screen give some indication of the size and form of the fault behaving as a reflector, and the distance of the fault from the probe. A knowledge of the direction at which the beam enters and reflects within the boundaries of the plate enables a variation of signal caused by a given probe movement to indicate to some extent the position and shape of the inspected fault.

Taking into account the simplest law of reflection, the planar fault can reflect the beam in almost any direction, depending on its orientation, so that it may emerge from the surface over an area relative to the transmitting probe. If the fault is not to pass unnoticed, the receiver probe should be moved over this area, giving what is called a 'kidney scan.'

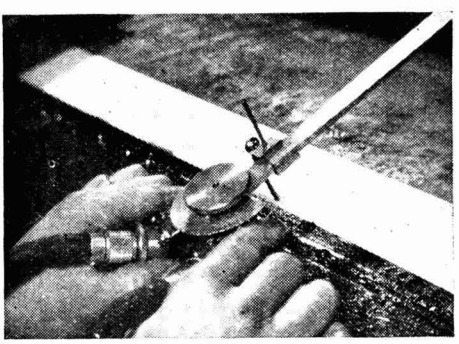
Approaching a weld of unknown quality and without subsidiary knowledge, this type of procedure is ideally required, but the time involved detracts seriously from the advantages to be gained, since all scanning must be carried out with care, bearing in mind the difficulties of surface contact.

Based on this reasoning, together with the fact that few flaws are truly planar, there is quite a good case for a systematic examination using a single probe or two probes fixed relative to each other. The relative drop in efficiency of the method is usually outweighed by the advantages gained, the chief of which is that fewer variables are left to the skill of the operator. The success of fixed probes relies on the relative size of flaw acceptable in the work under examination and however orientated (since few flaws, even cracks, are planar in practice) will usually give ultrasonic evidence of their presence.

In applying ultrasonics to their own inspection problems (up to 1½ in. thick plates) Frasers set out to systematise the scanning, using one scan either side of the weld at a fixed distance and one straddle scan (probe either side of the weld at an acute angle to it) for welds up to ¾ in. thick and five scans (two either side and one straddle) for thicker welds. Later work, however, showed that a wriggling motion along the path parallel to the weld not only improved the reception of echoes, but assisted in the maintenance of good surface contact. The slight additional movement did not slow down the inspection appreciably since the probes were moved as a unit by means of a protractor holder.

The identification of types of defect as planes, cylinders and spheres in theory was found to be relatively easy, but when applied in practice defects found are, more often than not, a combination of these geometrical shapes and, as such, extremely difficult to identify exactly.

Closer identification of the nature of defects can sometimes be made radiographically, but where this is not convenient Frasers recommend that a series of standard welds incorporating various defects should be prepared in an appropriate set of thicknesses. This may be done over a period of time until a comprehensive 'library' is built up. Each weld should have a radiographic



Two views of the scanning unit, showing (left) the protractor holder, and (right) the two probes

record and a print or sketch kept for shop reference during inspection. By inspecting these plates ultrasonically under laboratory conditions the best instrument settings can be obtained and noted on the radiograph.

After repeated runs on any one plate an operator will soon recall the fault associated with the present working conditions and the response of the equipment at any time while working on an unknown job can be assessed. This point is important. In actual testing it is the instinctive desire of an operator to do a run on similar, but known welds before translating signals in terms of faults on an unknown job. This procedure, although empirical and having some disadvantages, is probably one of the best ways of ensuring that variation of working conditions as a whole does not result in failure to record a significant fault.

The angle at which the beam enters the plate is usually known and its path through the plate can be drawn geometrically, taking into account reflection between the plate faces. The distance of the fault from the probes measured along the calibrated screen and marked on the diagram gives in theory the exact position of the fault. In practice this can be done by eye with quick reference to a diagram of the plate under inspection, but more easily assessed in thicker plates where the beam does not cover the whole of the weld from any one scanning position.

Lack of weld interpenetration can usually be distinguished by the obtaining of a maximum echo when the probe assembly is perpendicular to the centre line of the weld and a very rapid falling off of the echo as the assembly is turned through a small angle. A slag line on the other hand is often composed of a number of individual particles which reflect radially and the falling off of echo is not so pronounced. The signal should also, by theory, tend to become ragged due to difference of path length from the probe to the individual particles.

W. J. Fraser & Company have always proudly claimed that throughout the whole of their 120 years of existence they have constantly kept ahead in the development and use of new techniques. With their departure into the field of ultrasonics they are living up to this boast. They are ensuring that the high quality level of their products is maintained while at the same time keeping the costs of production down. Already ultrasonic testing has saved the firm considerable money on two large contracts and has saved the customers concerned lengthy delays in delivery. Mr. Autry and other directors of Frasers are enthusiastic about the technique they have developed and are anxious to prove its advantages to all those interested.

Special Sugar Allocation

THE Ministry of Food has announced that manufacturers may apply for an allocation of certain imported direct consumption sugars over and above their allocation of sugar for the present period. The sugar consists mainly of BWI crystallised, but there is also a small quantity of Muscovado sugar. Manufacturers who are interested should apply forthwith to the Ministry of Food, Sugar Division, Great Westminster House, Horseferry Road, London, S.W.1, for the necessary permit. They should state: (1) the quantity and type of sugar required, and (2) the name of their registered supplier of imported direct consumption sugar. Manufacturers may apply for whatever quantity of these special sugars they can take up within a month of the date of application. The usual terms for the sale of imported direct consumption sugars will apply.

Titanium for Defence

THE Ministry of Materials announces that it has recently concluded a contract with Imperial Chemical Industries Limited under which the company will at its own expense erect capacity to produce 1,500 tons of sponge titanium a year. The company will also install capacity for converting this metal to ingot form, and it expects production to begin in two years' time. The Ministry has undertaken to buy directly up to three-quarters of the company's output of sponge titanium over the first four years of production if the metal is not otherwise sold in various forms for use by Government contractors, and the Ministry has an option on the full output if needed for defence purposes. Apart from this project, I.C.I. already has in course of erection pilot plants which will begin to produce at the rate of 150 tons per annum early in 1954.

The British Association

Forthcoming Annual Meeting at Liverpool

THE British Association for the Advancement of Science has issued advance copies of the programme for the 115th annual meeting, which is to be held in Liverpool, 2-9 September.

The programme shows that the customary wide range of subjects will be discussed at meetings of the 13 sections. Altogether, it is estimated, there will be about 320 speakers, including about 180 from Universities, 50 from Government establishments and 30 from industry.

Details are also given in the programme of about 100 excursions and visits to works, which will enable members to visit all the main places of interest on Merseyside. Some of the excursions will go as far afield as the Lake District and North Wales.

The president is Sir Edward V. Appleton, vice-chancellor and principal, University of Edinburgh, who was Nobel Prizewinner (Physics) in 1947. 'Science for its Own Sake' is the subject of his presidential address, which he will deliver the first evening in the Philharmonic Hall.

The officers of the Chemistry Section are as follows:—President, Professor G. R. Clemo; vice-presidents, Professor C. E. H. Bawn, Dr. J. Ferguson, Professor T. P. Hilditch, Professor R. A. Morton, Professor A. Robertson, Professor E. C. Rollason, Professor W. Wardlaw and Dr. C. G. Williams; recorder, Professor L. Hunter; secretaries, Dr. J. Dewar and Dr. D. C. Martin; local secretaries, Dr. A. Hickling and Dr. W. B. Whalley.

Meetings of the Chemical Section will be held in the Gossage and Muspratt Chemistry Laboratories. Professor Clemo will deliver his presidential address, 'A Suggestion and Some Consequences,' on the morning of 3 September.

At subsequent meetings of the section, speakers and their subjects will be as follows:—Sir Robert Robinson, 'Structural Relations of Natural Products'; Professor E. D. Hughes, 'Reaction Mechanisms'; four addresses on 'The Heavy Chemical Industry of Merseyside': 'A General and Historical Survey,' by Dr. J. Ferguson, 'The Alum and Aluminium Sulphate Industries,' by Mr. J. H. Harwood, 'The Development of the Soap and Allied Industries of Merseyside,' by Dr. T. Kennedy, and 'Recent Developments in the Petroleum Industry,' by Mr. A. J. Carter; four addresses on 'Chemotherapy and Antibiotics': 'Introduction,' by Professor A. Robertson, 'The Chemistry of Antibiotics,' by Dr. B. A. Hems, 'Chemotherapy,' by Dr. F. L. Rose, and 'Biological Problems of Chemotherapeutic Research,' by Professor B. G. Maegraith; three addresses on 'Transmutation (Modern Alchemy)': 'Transmutation and Chemistry,' by Dr. R. Spence, 'Transmutation by Means of Particle Accelerators,' by Dr. K. F. Chackett, and 'Synthetic Elements Produced by Nuclear Reactors,' by Dr. R. Hurst.

Places to be visited during excursions by the Chemical Section are as follows:—Shell's Thornton Research Centre; W. & R. Jacobs; Joseph Crosfield & Sons; I.C.I. Research Laboratories, Widnes; Shotton Ironworks; Bibby & Sons; Tate & Lyle's Refinery; and Lever Brothers, Port Sunlight.

Social arrangements for the Association as a whole include a civic reception, a University reception, 13 dinners, two complimentary lunches, two conversaziones and various other entertainments. There will be an official service in Liverpool Cathedral on 6 September, at which the sermon will be preached by the Archbishop of York.

Terylene Council Move

THE I.C.I. Terylene Council, at present operating from Welwyn Garden City, is gradually being transferred to new headquarters on a large estate on Hookstone Road, Harrogate, which was purchased last November and includes Crimple House. The first contingent, with Dr. E. B. Abbott, technical service representative to the Council, arrived in Harrogate on Monday, 10 August, and established themselves in the house which will serve as temporary headquarters pending the completion of new buildings next year on the adjacent land. It has been made clear by the I.C.I. that these buildings will be devoted to research in the development of Terylene.

Glaxo Development at Ulverston

Rapid Growth of a Vital New Industry

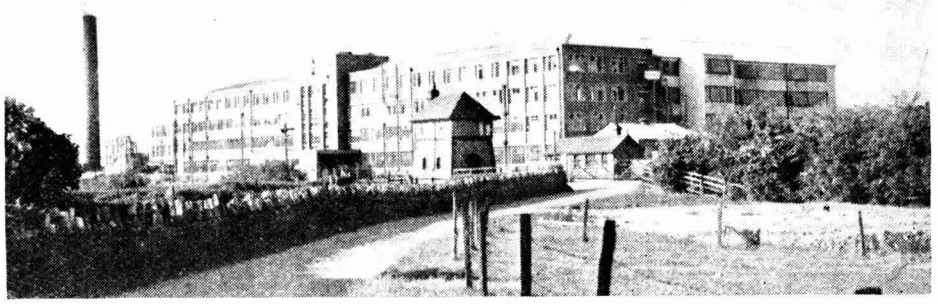
ONE of the largest projects carried out by Glaxo Laboratories, Ltd., in post-war years has been the construction and subsequent development of the company's antibiotics plant at Ulverston, in north-west Lancashire. This enterprise has been in continuous production since 14 April, 1948, and is making a substantial contribution to home and overseas supplies of penicillin, streptomycin and vitamin B_{12}.

It was early in January, 1945, after the new factory project had got well beyond the planning stage, that the company applied to the Board of Trade for a building site. After surveys in South Wales, Lancashire and Co. Durham had been carried out, the North-West Region of the Board of Trade suggested a site, a former ironworks, at Ulverston. Triangular-shaped and of an area of 140 acres, it had much to commend it. There was direct access to the sea along the east side of the Furness Peninsula; the Bardsea branch line of the Midland Region ran alongside the derelict ironworks which itself had 8,000 yards of sidings; there were ample water supplies from the neighbouring canal; and gas and electricity supplies were adequate. The manpower situation in the town and outlying district was entirely favourable to the setting up of a new industry, while the comparative nearness of Ulverston to the company's other antibiotics plant at Barnard Castle (Co. Durham) was a further advantage.

Towards the end of 1946 the necessary building licences were obtained and by the following January work had begun. It was destined to be one of the fastest building operations in the history of the company. The work commenced at a time when the whole country was in the grip of an arctic winter and when fuel shortages were acute, but despite the many difficulties the project went ahead with astonishing speed. Only sixteen months later, on 14 April, 1948, the first fermenter was seeded and a few days later the inital batch of penicillin had been produced.

The fermenters were installed at the rate of one every two weeks and initially were devoted to penicillin manufacture. At the same time the second fermenter hall was rapidly nearing completion and by November, 1948, half of the fermentation plant was installed and working. In 1949 the second fermentation hall was completed and turned over to large-scale manufacture of streptomycin. Today one hall is devoted to penicillin manufacture and the other mainly to streptomycin.

Much depends on continuous provision of power supplies and in the event of a grid failure diesel driven high-speed alternators are on stand-by to take over part of the



A general view of the Glaxo penicillin and streptomycin plant at Ulverston

electrical load. The fermenter halls, which are steel framed and clad with corrugated asbestos and asbestos roofing, are ventilated by large mechanically-driven extraction fans.

Most of the multi-storey main building is devoted, in broad terms, to pencillin and streptomycin extraction processes and to various departments ancillary to the fermentation operations.

On the ground floor of the central part of this main block are the loading bays, despatch section, plant-rooms and offices. The first floor is devoted to the storing of raw materials and to the batching areas. Inoculum production (for the seeding process), and the chemical control and biological control laboratories are sited conveniently near operating levels on the operating floor. During each fermentation run four-hourly tests are carried out in these control laboratories to check sterility, *p*H, etc. Liquid raw materials (acids, corn steep liquor, etc.) are stored on the third floor.

Elsewhere in the main block are sections carrying out extraction processes on both penicillin and streptomycin. The similarity between the production of penicillin and streptomycin ends at the fermentation stage. In penicillin manufacture, batteries of centrifuges are used in the solvent extraction and purification of the crude drug from the broth, followed by its precipitation as a crude salt. The crude penicillin is converted to crude sodium or potassium penicillin which, as solutions, are passed through Seitz filters into stainless steel crystallisers where every precaution is taken to ensure sterility of both plant and product. To these crystallisers, appropriate additions of solvent or procaine hydrochloride are made, under sterile conditions, according to whether the product required is crystalline sodium, potassium or procaine penicillin.

Streptomycin Separation

In streptomycin manufacture the drug is separated from the mould by vacuum filtration, adsorbed by means of an ion-exchange process and subsequently eluted with acid. The solution is converted to a crude but crystalline salt, calcium chloride complex. This complex can, in turn, be converted to streptomycin sulphate by precipitating the calcium present, as insoluble sulphate, and preciptating the streptomycin sulphate from methanol.

By means of catalytic hydrogenation, and similar removal of the calcium, calcium chloride complex can also be converted to dihydrostreptomycin. Solutions of streptomycin, either the complex or sulphate, or of dihydrostreptomycin, in water, are subjected to Seitz filtration and freeze-drying operations before the drug emerges in its finished state as either sterile streptomycin, or dihydrostreptomycin.

New Unit for Final Operations

A new unit has been erected on the north side of the main installation to deal primarily with finishing operations on penicillin and streptomycin. This is a three-storey building, of brick exterior, commenced in 1949, and now in full commission. Sterile re-crystallisation processes on penicillin, finishing processes on streptomycin and dihydrostreptomycin and streptomycin freeze-drying are some of the operations conducted in this block. Final tests on the finshed products are carried out in extensive modern laboratories in the same unit.

The ground floor of the unit is devoted to sterile crystallisation of penicillin. It comprises a crystalliser room, with a battery of stainless steel crystallisers and accompanying Seitz filters; a sterile offloading section, where the containers from the crystalliser section are transferred to trays; a drier room where solvent is removed from the penicillin by drying under a vacuum in electrically heated driers; a sterilising section; and a sieving room, where, under sterile conditions the crystalline penicillin is reduced to a uniform smooth flowing powder.

The first floor is devoted to the streptomycin finishing and freeze-drying sections. After the drug has undergone final purification tests in the finishing room, it passes to the freeze-drying unit, where a battery of glistening drying units, lofty ceilings, excellent natural and artificial lighting, tiled walls and gowned operatives make an impressive picture Also in this sterile area is a cold room, milling room, and wash-up and sterilisation room, and, under the control of the analytical department, a sampling department. Distilled and demineralised water is supplied by this section to the plant.

Two departments, Analytical and Process Investigation, are housed in the top storey, the former carrying out the analytical requirements on all finished products and certain raw materials and the latter charged with the investigation of process problems.

The Analytical Department includes two chemistry laboratories, a biological assay laboratory, media preparation and sterility testing sections, balance room and a control laboratory. One of the features of the biological assay laboratory is a plate reading projection section where projectors can handle large assay plates involving 200 samples per day. The laboratory is equipped with several incubators in which automatic temperature recorders give continuous day and night readings.

The refrigerator unit is an interesting installation. Adapted to the company's own specification, the refrigerators are low built and give bench space along their whole length.

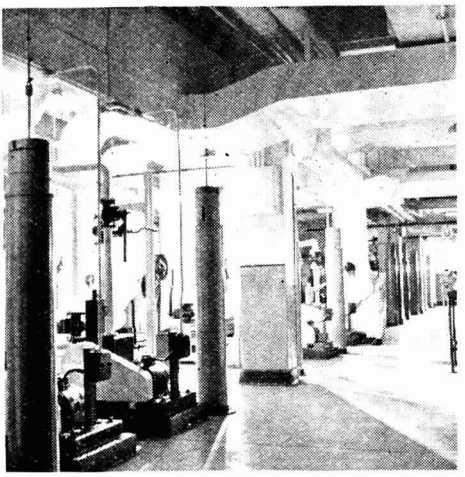
In the air-conditioned sterility testing room samples from every batch of penicillin and streptomycin are examined for freedom from contaminating moulds and bacteria. The two chemistry laboratories separately handle penicillin and streptomycin. Distilled water on tap is a special feature of these laboratories.

The function of the Process Investigation Department is to suggest improvements in the production processes and to investigate process problems. It comprises four laboratories, Biochemistry and Biological; Physical Chemistry; Organic and Analytical; and Experimental The section includes a darkroom with polarimeters for penicillin assay and a statistical section.

Administration Building

The administration centre for the plant is located in a country house over 125 years old, formerly occupied by the North Lonsdale Iron and Steel Company. In this suite are the director in charge of fermentation operations and his staff; administrative departments; accounts, costing and wages sections; and the teleprinters linking the plant with the company's headquarters at Greenford and its other northern antibiotics plant at Barnard Castle.

In a well-equipped power house are steam turbine and electrically driven air compressors supplying filtered air to the fermentation units; diesel driven reserve compressors; electrical switchgear and distribution equipment for supply of current and diesel alternators for load shedding and for use in the event of power failure. During the winter months these diesel alternators supply part of the factory's daytime requirements.



Part of the refrigeration plant for the streptomycin freeze-drying unit

An efficient boiler house installation includes Lancashire oil-fired boilers, supplied with water taken from a neighbouring canal. Adjoining the boiler house is a reservoir of 1,250,000 gallons capacity, used as a spray cooling pond.

During the first five years of the factory's history, the Engineering Department was housed in old buildings, hardly suitable for efficient operating. In 1952 a new building was completed which houses the central engineering workshops, engineering stores buying and stores department offices. The buying and stores departments and a conference room are located in the same building.

Since the company took over the site in 1948 a number of exterior improvements have been carried out. Cultivation of a slag heap, estimated to hold over 6,000,000 tons of slag, and a legacy of the old ironworks days, has started. Several hundred sycamores, elders and limes have been planted and are beginning to stimulate vegetation. Technical development at Ulverston is proceeding hand-in-hand with site improvement.

Pharmaceutical Society Relief Fund

The Pharmaceutical Society has now closed, at more than £12,000, its fund for the relief of pharmacists who suffered in the East Coast flood disaster.

Sulphuric Acid Returns

A SUMMARY of monthly returns of production and consumption of sulphuric acid in the UK from 1 April to 30 June, issued by the National Sulphuric Acid Association, Ltd., shows that stocks increased during that period from 59,020 tons to 63,076 tons.

The following tables have been abstracted from the summary:—

SULPHURIC ACID AND OLEUM
(*Tons of* 100 *per cent* H_2SO_4)

Data referring only to acid makers' returns	Chamber only	Contact only	Chamber and Contact
Stock, 1 April, 1953 ..	26,417	32,603	59,020
Production	161,120	298,739	459,859
Receipts	31,992	22,642	54,634
Oleum feed	—	1,212	1,212
Adjustments	−116	−119	−235
Use	99,843	115,799	215,642
Despatches	89,195	206,577	295,772
Stock, 30 June, 1953 ..	30,375	32,701	63,076
Total capacity represented	200,080	365,400	565,480
Percentage production ..	80.5%	81.8%	81.3%

RAW MATERIALS
(*Tons*)

Data referring only to acid makers' returns	Pyrites	Spent Oxide	Sulphur & H_2S	Zinc Concentrates	Anhydrite
Stock, 1 April, 1953 ..	130,762	214,597	51,327	86,246	550
Receipts ..	94,102	66,408	57,334	37,504	45,965
Adjustments	−1,072	+612	+1,223	−37	—
Use	95,922	68,712	61,845	39,998	45,635
Despatches*	309	17,379	105	—	—
Stock, 30 June 1953 ..	127,561	195,526	47,934	83,715	880

* Including uses for purposes other than sulphuric acid manufacture.

CONSUMPTION IN THE UNITED KINGDOM
(1 *April*–30 *June*)

Trade Uses	Tons 100% H_2SO_4
Accumulators	2,347
Agricultural purposes	450
Bichromate and chromic acid	3,052
Bromine	4,120
Clays (Fuller's earth, etc.)	2,226
Copper pickling	911
Dealers	2,755
Drugs and fine chemicals	3,234
Dyestuffs and intermediates	15,752
Explosives	7,761
Export	542
Glue, gelatine and size	134
Hydrochloric acid	13,035
Hydrofluoric acid	2,708
Iron pickling (including tin plate)	22,818
Leather	882
Lithopone	3,068
Metal extraction	996
Oil refining and petroleum products	16,054
Oils (vegetable)	2,558
Paper, etc.	1,403
Phosphates (industrial)	399
Plastics, not otherwise classified	5,336
Rayon and transparent paper	55,322
Sewage	2,766
Soap and glycerine	7,088
Sugar refining	171
Sulphate of ammonia	70,617
Sulphates of copper, nickel, etc.	5,935
Sulphate of magnesium	1,344
Superphosphates	119,740
Tar and benzole	4,705
Textile uses	5,540
Titanium oxide	40,994
Unclassified	34,532
Total	461,295

Colchicine Changes Hands

WE are informed by Metallurgical Chemists Limited that they have recently acquired from Universal Crop Protection Limited, agents for Pal Chemicals Limited, the stock in trade and the manufacturing plant and rights for colchicine. Production has now been resumed in the works of Metallurgical Chemists Limited and any commercial quantity of colchicine or colchicine salicylate can be supplied from stock. After the colchicine has been extracted and purified to the standards of the British and United States Pharmacopœias, it is submitted to an extensive chromatographic purification developed from that described by Ashley and Harns (*J. Chem. Soc.*, 1944, 677) and by Horowitz and Ullyot (*Science*, 115, 216 [1952]). This removes bright yellow impurites which occur to the extent of 4 per cent in material complying with the requirements of the BP and USP. The main impurity was first described as Substance 'C' by Santavy and Reichstein (*Helv. Chim. Acta*, **33**, 1606 [1950]) and has now been shown to be desmethylcolchicine where one methoxy group in Ring A of the formula of colchicine is demethylated. (Horowitz and Ullyot [*loc. cit.*]). The other impurities which occur in lesser amounts are removed at the same time.

New members of the Federation of British Industries include Fisons Chemicals (Exports), Ltd., London, N.W.1; the Smith Chemical Company, Guiseley, nr. Leeds; and Stockport United Chemical Company, Ltd., Stockport, Cheshire.

Development, Principles & Applications of Interrupted Quench Hardening

by RICHARD F. HARVEY*

THE hardening of steel is generally accomplished by one of the following methods: (*a*) continuous cooling from above its critical temperature to room temperature, usually in air, oil, or water; or (*b*) interrupted quench hardening, variously termed step quenching, Martempering, or Marquenching.

Scientific heat treatment is a recent development which has its roots in the fundamental knowledge of the changes which occur when steel is quenched or otherwise cooled from above its critical temperature to room temperature. The commercial application of this fundamental knowledge has enabled us to obtain and control desired combinations of hardness, structure, and properties which a few years ago were not possible. Our knowledge of heat treating has been clarified through research and several new treatments have been developed. One of the most useful of these new treatments, now known as Martempering, is important commercially because it minimises stress concentration and distortion and avoids cracking.

The Martempering treatment is so well known today that a detailed description of the process is hardly necessary. Suffice it to say that the method involves quenching the heated steel from above its critical temperature into a hot bath maintained at about 400°F. or above or within the range of martensite formation. The work is then held sufficiently long to equalise in temperature but not long enough to permit transformation at that temperature level. This is followed by slow cooling in air through the range of martensite formation to room temperature.

Early Attempts Empirical

The earliest attempts at interrupted quench hardening may be traced back to 1879 when Richard Akerman[1] dipped the austenitised steel into a lead bath followed by air cooling. Other early investigators including D. Lewis[2] and O. C. Trautman[3] might be cited. In the light of our present knowledge these early attempts appear to be largely empirical and limited in usefulness.

The writer is more than grateful to the kindly fates that brought early acquaintanceship with Martempering. Insofar as it is known, the treatment was first developed and brought under scientific control through the efforts of the writer starting in 1938 while serving as a hardening apprentice at the Simmonds Saw & Steel Co. The treatment, which was originally termed Step Quenching, was first applied to the hardening of hacksaw blades of the 1¼ per cent carbon-1½ per cent tungsten type, resulting in straighter, tougher blades which cut 20 per cent better than regularly hardened saws. These efforts culminated in a US Patent Application, Serial No. 320,998 filed 27 February, 1940,[4] which was eventually disallowed by the Patent Office. Figure 1 illustrates schematically three heat treatments: Conventional hardening, Austempering, and Step Quenching, with reference

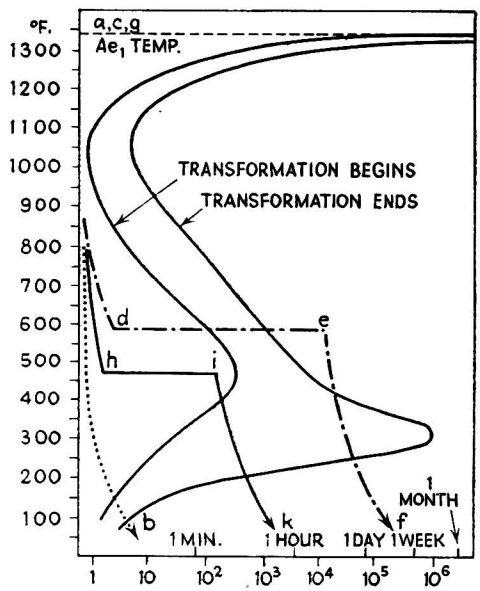


Fig. 1. Reproduction of Fig. 5 of US Patent Application, Serial No. 320, 998, filed 27 February, 1940

* Reprinted from *Journal of The Franklin Institute*, 255 (2), pp. 93-99, by permission of the author.

to the transformation curves taken from the patent application.

Austempering is an isothermal treatment which consists of quenching rapidly through the pearlite—forming temperature region down to a temperature above the range of martensite formation and then holding for a sufficient length of time to complete the transformation at that temperature level to a structure termed Bainite. This treatment results in moderate hardnesses generally about Rockwell C 50 to 55 which is appreciably below that obtainable on interrupted quench hardening.

The method previously used to harden the hacksaw blades consisted of heating in a hearth type furnace, followed by oil quenching and clamp straightening. The blades were placed in the furnace one at a time by means of a hooked poker about 6 ft. long. After heating through, the hardener had to hook the small hole about $\frac{1}{8}$ in. in diameter at one of the ends of the saw blade with a poker to withdraw the blades from the furnace for quenching in oil. It was a difficult task to hook the small holes, as the heated blades and furnace all appeared the same shade of red when the hardening temperature was attained. While I was a failure production-wise on this assignment, it did leave me with a strong desire to develop a better method of hardening hacksaw blades.

There were many difficulties and progress was slow as the new treatment I developed was too far ahead of its time to be appreciated. My formal training and degree were in chemistry and much of my time had to be devoted to learning the fundamentals of metallurgy. Also, I was assigned to production problems and routine laboratory work rather than research, so that the constant interference of other projects, many of an entirely different nature, became a major problem. However, a clearly conceived idea of the new quenching method and what it was expected to accomplish kept me on the right track. Working in a new field, there are no established directions to follow and many cross roads appear which can only lead to blind alleys and negative results.

There were many other obstacles to overcome. The transformation curves constituting the fundamental technical knowledge which led to the conception of Martempering, were misleading insofar as the transformation of austenite to martensite is concerned.

Contrary to accepted opinion at the time, this change is not an isothermal transformation as shown in the transformation diagram of Davenport and Bain, but is a change which depends on a lowering in temperature for its continuation. The classic investigation of Troiano and Greninger[5] conclusively proved this point.

Over traditional hardening, interrupted quench hardening offers a number of important advantages: (1) less distortion, (2) less internal stress, (3) less cracking, (4) greater toughness, (5) greater strength, (6) greater fatigue life, (7) savings in man hours through elimination of unnecessary operations such

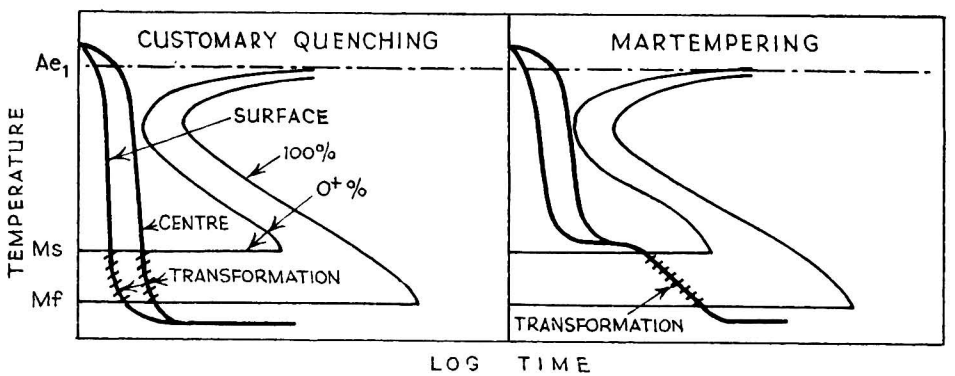


Fig. 2. Comparison of regular hardening and Martempering, showing difference in cooling rate of the outside and centre. It will be noted that large temperature gradients may exist in steel as it hardens on conventional quenching, whereas in Martempering the temperature is nearly uniform throughout all sections as hard martensite forms. The tempering operation is not shown as this is the same for both treatments

as straightening and grinding, (8) less rejects, and (9) higher hardness and wear life on case hardened work as the distortion on regular hardening necessitates removal of the hardest part of the case in grinding.

The reason for internal stresses is that the outside or surface hardens first on continuous quenching to room temperature, resulting in an expansion. The interior hardens afterwards when it reaches the hardening range (below about 400°F. for many steels). As the interior hardens it tries to expand but is resisted by the hardened surface. Also, simultaneously, there is a thermal contraction taking place as the steel changes from above its critical temperature to room temperature. The net result of this is a tug-of-war between the inside and outside and between thick and thin sections.

Warning of Cracking

These internal stresses are often greater than the inherent strength of the hardened steel, which may be as high as 500,000 psi. for tool steels. When this happens the hardener will hear that familiar 'ping' which tells him his work is cracked. Even if the part does not crack it is common for the disruptive internal stresses to amount to as much as 90 per cent or more of the total strength of parts. This means that only a small fraction of the calculated strength of hardened parts may be available to do useful work. Since we cannot conveniently measure the concentration of stress in hardened parts by non-destructive tests, it is often a gamble as to whether such parts will stand up or fail in service.

The interrupted quench hardening treatment attempts to equalise the temperature within parts before hardening proceeds on slow cooling in air. In this way the hardening proceeds uniformly throughout all sections of the part thus avoiding undesirable concentrations of stress. The treatment is, therefore, of particular value on parts of intricate shape or where the ultimate in accuracy, physical properties, and uniformity is desired.

Since my original investigation on hacksaws, other applications followed and a thesis[5] on the treatment was prepared at the Worcester Polytechnic Institute during 1940. Today the treatment is widely used commercially by many concerns engaged in the hardening of steel.

A large manufacturer of farm implements reports use of the treatment on an extensive basis for transmission gears, differential gears, pinions, shafts, etc. The use of quenching presses, plugs, and special fixtures has been eliminated. Straightening and the amount of finish grinding has been reduced. The company found residual stresses reduced to such an extent that service life of gears is increased 100 to 200 per cent and load carrying capacity is increased from 15 to 20 per cent.

The following laboratory test results for drive gears is cited:

Die quenched: Load, 260 lb. Results: Pits after running 18 hr.

Hot quenched: Load, 300 lb. Results: No pits after running 100 hr.

An automotive manufacturer reports the use of interrupted quenching in hot salt on transmission gears where involute tooth distortion is held to plus or minus 0.0002 in. Tooth grinding has been eliminated.

While no attempt will be made to cite a complete list of uses of the treatment, some other applications are: 20-mm. machine gun parts, 40-mm. Bofors gun parts, tank engine camshafts, tank armour plate, tank torque converter parts, tank tread parts, aircraft clutch rings, armour piercing shot, bearings, races, spindles, clutches, meat slicing knives, raceways, outboard motor

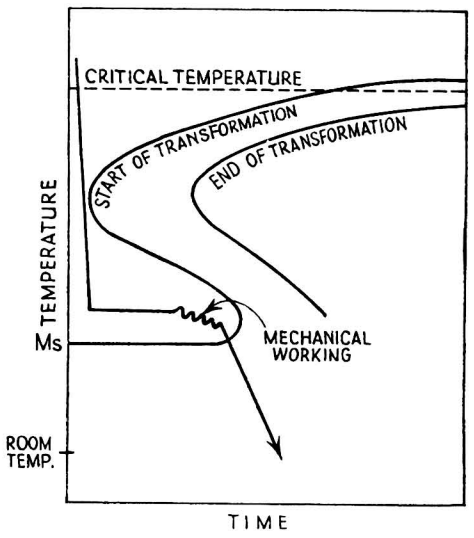


Fig. 3. Diagrammatic representation of the Step Quench Hot Working Method. Shot peening is an effective hot working method

crankshafts, gears, springs, dies, knives, etc.

The hot quenching bath may be molten salt or hot oil. Hot salt has the advantage of a faster quenching rate and is more effective for large sections. It is necessary that the work be cooled rapidly, at a rate greater than its critical cooling rate, for full hardening. Salt pumps or motor driven agitators are used to circulate the salt to provide a more rapid and uniform hot quench. Separating chambers are provided on many new installations to separate contaminants which reduce the cooling power.

One field where commercial applications of the treatment will probably increase involves the hardening of brazed parts. The use of assemblies brazed with silver solder or copper has replaced expensive machining operations on many industrial applications. The brazed joints, however, are very weak at the temperatures used for hardening and may easily be ruptured by the severe temperature gradients encountered in traditional hardening practice. The interrupted quench, on the other hand, permits hardening of brazed assemblies which would otherwise be most difficult if not impossible.

Latest Development

A new modification[8] of the interrupted quench treatment which I recently developed involves a combination of step quenching and mechanical working above or within the range where hardening occurs.

Shot peening is a common means of cold working metal parts to introduce beneficial surface compressive stress to increase fatigue life. Shot peening the metal while hot and still austenitic after removing from the low temperature bath above or at the Ms point results in a greater degree of compressive stress at the surface than may be imparted by conventional cold peening methods. For example, Almen test strips 0.051 by $\frac{3}{4}$ by 3 in. shot peened to an arc height of 0.016 in. by conventional cold peening, showed an arc height of 0.029 in. when hot peened by the new method described and using the same intensity of peening. The greater curvature of the hot peened specimens is due to the greater degree of surface compressive stress.

In addition to the higher compressive stress and fatigue life the hot peening results in higher hardness and transformation of an undesirable constituent, retained austenite, from the surface. It has been found that mechanical working of the subcritical austenite results in a greater conversion to hard martensite than would result without the mechanical working. X-ray determinations of the volume per cent of retained austenite on specimens peened and unpeened show a conversion of up to about 80 per cent of the retained austenite as a result of hot peening.

Other forms of mechanical working other than shot peening may be used with beneficial results. While the step quench hot working method is still in the experimental stage it is expected that commercial applications of this treatment will follow.

The trend of hardening in modern industry is toward more extensive use of interrupted quench hardening and many feel that it will ultimately be more widely used than other methods of hardening steel. The use of Martempering—or step quenching as I still like to think of it—has enabled us to obtain greater strength and toughness from hardened parts together with a minimum of distortion, internal stress, and cracking.

Over the years the combined efforts of many interested in the science of metals and heat treating have transformed an age of stone into one of brass and later to one of iron and steel.[9,10] To prophesy the future is an uncertain and thankless task. However, it is to be expected that the interrupted quench principles will be one of the fundamental building blocks on which much of future hardening progress will be based.

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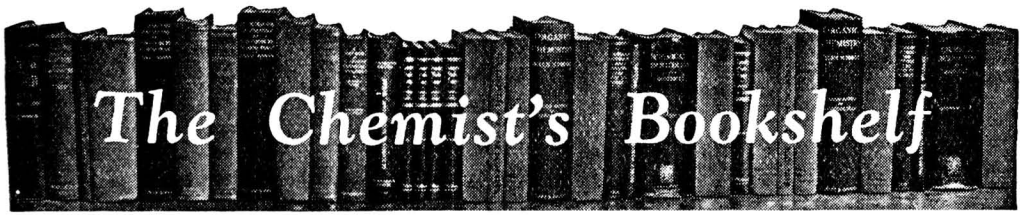
USA Rubber Plants Sale

A Bill setting up a three-man commission to sell the USA Government's synthetic rubber factories to private enterprise has been signed by President Eisenhower.



ELECTRON DIFFRACTION. By Z. G. Pinsker. Butterworth Scientific Publications, London. 1953. Pp. xiv + 443, with numerous line diagrams and plates. 63s.

Professor Pinsker, Institute of Crystallography of the Academy of Sciences of the USSR, is a leading Russian authority on electron diffraction. He and his colleagues have exploited the method of obtaining important information on the structure of materials suitable for examination in the electron diffraction camera. This includes structural examination of materials which exist in forms not readily amenable to X-ray diffraction. Unit cells may be determined from diffraction patterns obtained with particles about 5×10^{-6} cm. across. Complete structure analysis has been made with substances such as barium chloride monohydrate available only in very small crystals. The structure of this compound was previously unknown. Among the methods of treating the diffraction information is that of Fourier synthesis carried out with derived structure factors as coefficients. The resulting maps do not give electron density distribution. The electrons are diffracted not by the electron clouds, as in the case of X-rays, but by the potential of the crystal lattice. This potential, like the electron density, is a continuous periodic function of the co-ordinates and the maxima of this function are closely connected with the centres of gravity of the atoms. It is pointed out that the method can reveal details which remain unresolved in X-ray work. This includes determination of the positions of hydrogen atoms. Since the book was written the author has published work on electron diffraction determination of the position of hydrogen in the lattice of paraffin wax, and, more recently, Australian workers have made similar determinations and have examined the positions of hydrogen in the hydrogen-bonded crystalline boric acid. There are many important applications of the method to technical problems in metallurgy, electrical engineering, chemical technology, plastics, and the study of molecular interaction on crystal faces, this last application being of importance for an understanding of catalysis, adsorption and associated matters.

The book opens with sections on wave theory, wave mechanics and diffraction of waves by crystals. There follow chapters on experimental technique and the theory of interpretation of electron diffraction patterns by reciprocal lattice methods. Effects with both fast and slow electrons are considered. Atomic scattering and the dynamic theory of scattering are next described and the applications of these methods in structures of thin films, crystallisation processes, orientated deposits and sub-microscopic crystallography are presented. Among fine-structure problems considered are those concerning the crystalline and amorphous structures of elements and certain alloys, the structures of films formed on metals or alloys by oxidation, and the nature of the disorders in layer lattices. Application of electron diffraction to the structure of molecules is considered briefly.

The text is arranged so that the sections on experimental technique and on the results of the electron diffraction method do not require a detailed knowledge of the theory. The theoretical parts require some moderate knowledge of relevant physics and mathematics. There is one mathematical appendix.

This valuable work will enable those who do not read Russian to become more familiar with extensive Russian advances in the subject and is likely to stimulate further applications of the method. The translators, in addition to doing their difficult work well, have added a number of references and footnotes on more recent publications and supplied alternative plates for one or two which could not be reproduced from the Russian edition.—H.M.P

NATURFORSCHUNG UND MEDIZIN IN DEUTSCHLAND, 1939-1946. Verlag Chemie, Weinheim, 1953. Volumes 34 and 35: Theoretische Organische Chemie, Parts I and II by W. Hückel. Pp. 145 and 160. DM.10.

Volumes 37 and 38: Präparative Organische Chemie, Parts II and III by K. Ziegler. Pp. 300 and 352. DM.20.

These volumes are reprinted from the 'FIAT Review of German Science' which was published in 1947 in a strictly limited edition under the auspices of the Military Government for Germany. According to the preface of the 1947 edition, the FIAT review was produced 'in the hope that it will assist in informing international science of research done in Germany during the war years.' In the six years which have elapsed, many of the periodicals and books published in Germany during the war have become generally available, and the volumes under review have therefore lost much of their value. Furthermore, the general perspective in many fields of research has altered considerably, and some of the sections, especially those in which the emphasis is on interpretation rather than evidence, are now out of date.

The volumes on theoretical organic chemistry contain much which would have been radically changed or even left out if a new edition had been prepared. Of the nine articles in these two volumes, five are written by the senior author, W. Hückel (general structural chemistry and chemical bonds, stereochemistry, reaction mechanisms, association and surface tension, constitution and physical properties). The remaining articles are by F. Seel (free radicals), G. Scheibe (absorption spectra), J. Goubeau (Raman spectra) and G. V. Schultz (mechanism and kinetics of polymerisation processes). In at least one instance the literature is misquoted (Part I, p. 7—racemic diphenylglycol forms an acetone compound more readily than the *meso*-form and not vice-versa) and the reviewer had some difficulty in finding the original paper as the volume number and year given in the reference did not correspond. However, many of the sections are excellent and it is interesting to see how German scientists have tackled many of the problems of theoretical organic chemistry in a way which is different from (and to some extent complementary to) the approach used in England and America. There is a useful index covering both volumes at the end of part II.

None of the sections in parts II and III of preparative organic chemistry are written by K. Ziegler himself, but he has collected a number of expert collaborators who have given us valuable and authoritative articles. Part II includes chapters on organometallic compounds, synthetic dyes, sugars and their derivatives, strychnine and related alkaloids, diene-syntheses (an excellent article by K. Alder), chromatographic analysis (this contains sections dealing with many important fundamental considerations) and automatic methods for the microdetermination of the elements in organic compounds. Part III is devoted to compounds of high molecular weight and will be of special interest to chemists in industry. It contains chapters on the theoretical and practical aspects of cellulose chemistry (by H. Staudinger and G. Jayme) and a number of articles on various aspects of radical polymerisation and polycondensation which include discussions of emulsion polymerisation and copolymers.

It is unfortunate that the publishers have reprinted the original edition instead of producing a new one. There are considerable overlaps between some of the reviews, very few cross-references, many references to 'unpublished work' which has since been published and many misprints. Certain paragraphs in the reviewer's copy contain enough incomplete words to make them interesting as crossword puzzles. It is a pity that such useful volumes should have so many faults.—J.C.P.S.

Benn Brothers Ltd.

The directors of Benn Brothers Limited, publishers of THE CHEMICAL AGE, recommend the payment of the following final dividends less tax for the year ended 30 June, 1953:—3 per cent on preference shares, which with the interim dividend of 3 per cent paid in February makes 6 per cent for the year; 11 per cent, plus 2½ per cent special Coronation Bonus, on ordinary shares, as increased by the issue of bonus shares in December, 1952, which with the interim dividend of 4 per cent paid in February makes 17½ per cent for the year on such increased capital; and 3s. 6d. per share on the deferred shares.

More Uses for Plastic Materials

Applications in the Fitting-Out of Boats

THE 11-ton gaff-ketch 'Tango' is owned and has been converted by Richard Levin, O.B.E., M.S.I.A., Head of BBC Television Design. A converted Looe Lugger, of 32 ft. length overall, the 'Tango' now has wheel-steering and a new wheelhouse. At the same time the main cabin top has been raised to allow more head-room within.

Chief alterations, however, have taken place in the 4-berth cabin itself, which has been completely re-designed and furnished, and here great use has been made of Warerite Laminated Plastics for panelling and surfaces of all kinds.

The main fittings in the cabin are:—(1) a galley-unit consisting of concealed sink with a cover which provides a work-top and inverted, a chopping-board; a central cooker with cover which can be removed for use as a tray; and a concealed anthracite heater. Behind is a storage unit for food and, below, another for Calor-gas, etc. All the panelling of this unit is provided by Warerite veneered board in a grey weave design.

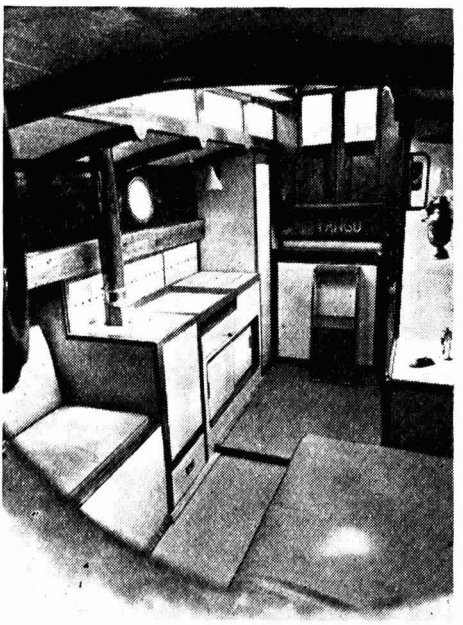
(2) A full-length, hanging cupboard built into the bulkhead and surfaced with Warerite materials.

(3) A water-tank, sited amidships. This is equipped with polyethylene plastic piping and concealed by a Warerite panel, which is hinged for rapid conversion into a lift-up chart-table. When used for this latter purpose, the steps leading into the cabin are employed as a pedestal, and they in turn incorporate a cold-box panelled with Warerite Red Stardust.

(4) The general-purpose table, surfaced with a combination of red and green Warerite, which is cantilevered from the main-mast tabernacle support and can easily be either folded away or removed altogether.

(5) In addition Warerite grey weave has been used to panel all the bulkheads, sides, bunk-fronts, drawer-units and cabin-top, the latter being also lined with Fibreglass insulation mat and topped with plywood, both of which are bonded with Bakelite and similar-type synthetic resins.

(6) The three bunks, one of which is double, are upholstered in leathercloth supplied by the Greenwich Leathercloth Co. and made from vinyl resins also supplied by Bakelite Limited.



This photograph, taken by natural lighting through the forward porthole of the cabin, shows the considerable improvement over the traditional mahogany panelling. Lined with Warerite, the interior is very much lighter and longer-lasting

Plastics have therefore played a major part in this conversion, not counting numerour minor accessories such as light switches, etc., and have helped to provide an interior which is both permanently colourful and exceptionally hard-wearing.

Free Trading in Copper

After a lapse of nearly 14 years, the London Metal Exchange began dealings in copper again on 5 August. Copper valued at more than £500,000 was traded, although there was a sharp decline in prices compared with the final Government selling price of £252 a ton. Closing prices were £210 buyers, £212 10s. sellers, for cash copper; and £196 buyers, £198 sellers, for three months metal.

• HOME •

Killed in Explosion

Only three days after starting work at the chemical works of L. B. Holliday & Company, Ltd., Deighton, Huddersfield, a man named Wilson was killed in an explosion there last week. He leaves a widow and seven children.

Factories Need More Water

The new I.C.I. factories at Wilton are provided with 6,000,000 gallons of water daily from a pumping station specially installed by the Tees Valley Water Board three years ago beyond the tidal reaches of the Tees. I.C.I. have now asked for an increase to 9,000,000 gallons a day. This means that the present machinery will have to be stripped and replaced and the pumping plant kept working during the change-over. The estimated cost of £37,000 is to be borne by I.C.I.

In Private Ownership Again

A £1,000,000 deal, transferring a steel company back to private ownership, has been completed by the Iron and Steel Holding and Realisation Agency. British Ropes Limited and William Cooke & Company, Ltd., have each bought 72,001 shares in Templeborough Rolling Mills at £7 5s. a share, cum dividend, thus regaining their former holding. The take-over value was £7 a share. This is the first sale effected by the Agency since it took over the £300,000,000 capital of the former nationalised steel companies.

Light Metal Statistics

Statistics just issued by the Ministry of Supply show that in May the UK production of virgin aluminium totalled 2,693 long tons; imports came to 20,071 long tons; and 10,383 long tons were despatched from Government holdings. Production of secondary aluminium in the same period was 6,912 long tons, the virgin content being 337 long tons, and despatches (including virgin content) from secondary smelters totalled 7,148 long tons. Fabrication of magnesium and magnesium alloys in May was as follows (the figures representing long tons):—sheet and strip, 17; extrusions, 10; castings, 360; forgings, 1.

Unrefined Oils and Fats

The Ministry of Food has announced that no change will be made in the prices of unrefined oils and fats and technical animal fats allocated to primary wholesalers and large trade users during the four week period ending 5 September.

Import Licence Changes

The Board of Trade has announced that the Open General Licence which came into force on 2 May, 1952, has been further amended with effect from 1 August, 1953. Additions to those goods which may be imported into the UK under licence (from countries other than those specified, as in Notice to Importers No. 492) include dipentine, raw casein, dissolved bone fertiliser, bone powder and steam bone flour.

Starch & Glucose Imports

As already announced in THE CHEMICAL AGE (1 August, p. 234), Government control of starch, starch products and glucose will end on 27 September. Traders were warned not to enter into commitments until the Board of Trade issued details of import arrangements. The Board of Trade has since announced that with effect from 28 September maize starch, farina (potato starch), dextrine and glucose may be imported on private account. Full details are given in Notice to Importers No. 579.

British Standards Institution Moves

By Monday, 17 August, the British Standards Institution will be in full operation at its new premises at No. 2 Park Street, London, W.1. (Telephone No. MAYfair 9000). The removal was planned to extend over the present week and although during this period some disturbance of day-to-day activities was inevitable, the BSI had arranged that services to its members and committee members would be fully maintained. As already announced, the new building will contribute to more efficient working by concentrating the BSI's scattered departments under one roof. It will also provide increased and more convenient accommodation for the 13,000 specialists who attend the 3,500 BSI Committee meetings held during the course of each year.

• OVERSEAS •

Chile Produces More Copper

Copper production in Chile for the first seven months of this year totalled 217,912 metric tons, compared with 210,633 metric tons during the corresponding period of 1952.

More Tin from Malaya

Exports of tin from Malaya in July totalled 5,257 long tons, as compared with 3,897 long tons in June, according to the Registrar of Statistics. The July shipments comprised 2,675 tons to the USA, 632 tons to the UK, 802 tons to Europe, 443 tons to British possessions and 615 tons to 'other destinations.'

Uranium from Granite?

Lecturing on 'The Future of the Atomic Age' in Bulawayo recently, Professor A. E. H. Bleksley, head of the Witwatersrand Faculty of Applied Mathematics, envisaged that the most important source of uranium in the world would probably be the ancient granites, 'such as the great granite domes of the Matoppos.'

India and Edible Oils

There is reported to be a shortage of edible oils in India and anxiety has been caused by price increases totalling about 30 per cent during the past six months. Exports of groundnut oil have been banned, while imports of coconut oil and palm oil have been encouraged by free licensing and a cut in import duty. The soap industry has been directed to use palm oil instead of groundnut oil.

Iron Ore in Guiana

In a pamphlet published by the British Guiana Department of Geological Surveys it is stated that iron ore deposits of some 46,000,000 tons have been discovered in the Blue Mountains on the left bank of the Essequibo River. An important feature of the discovery is the accessibility of the location via the Essequibo River, which is navigable to small ocean-going vessels. The Government is steadily pushing ahead with its programme for the systematic survey of the country's mineral-bearing hinterland.

Iron Ore Pelleting Plant

The Sydvaranger iron ore mines near Kirkenes are to have a pelleting plant installed this winter. It is hoped that as a result of the installation of this plant—the first of its kind in Norway—about half of the mines' output of 1,000,000 tons of ore a year can be turned into pellets by 1957. Pellets command a better price than ordinary iron ore.

Sulphur Pyrites in New Zealand

A deposit of sulphur pyrites, a quarter of a mile wide, has recently been found near Thames, on the Coromandel Peninsula, New Zealand. The sulphur pyrites is in similar concentration to workable deposits overseas, and the resources of the area are being investigated by the New Zealand Mines Department and the Department of Scientific and Industrial Research.

Canadian Titanium

Canada will be a major producer of titanium if further researches develop a cheaper processing method, according to Dr. W. J. Kroll, who is reputed to have developed the only known production process for recovering pure titanium from titanium ore. Huge quantities of ilmenite have been discovered in the Lake Allard region of Northern Quebec and a company at Sorel, Quebec, produces titanium slag as a by-product from iron ore.

Swiss Chemical Production

Current records show that during the first five months of 1953, Switzerland's chemical industry exported a total of 267,000,000 francs' worth of goods, compared with 258,000,000 in the same five-month period of 1952. The country's exports of pharmaceutical products likewise increased from 128,000,000 to 129,000,000 francs in value, while the export of dyestuffs rose from 74,000,000 to 86,000,000 francs. While business in the textile industry has improved since last year, exports by the Swiss dye industry have encountered increased competition in foreign markets, it is reported, because most countries have increased their own production capacity and some of them still restrict imports of dyestuffs.

• PERSONAL •

SIR ERNEST GOODALE has become president of the British Colour Council and MR. L. W. HANCOCK has been elected chairman.

SIR WALLACE AKERS, formerly a director of Imperial Chemical Industries Limited and Director of Atomic Research from 1941-1946, has been appointed a part-time member of the Southern Gas Board.

MR. SYDNEY N. DUGUID has resumed private practice in connection with fuel efficiency and smoke abatement problems. His business address is Brookfield Works, Elm Tree Road, Lymm, nr. Warrington, Lancs. (Tel.: Lymm 133).

MR. G. S. J. WHITE, chief colourist and head of the dyehouse department of the Dyestuffs Division of Imperial Chemical Industries, Ltd., has been appointed Dyestuffs Division director in charge of technical service departments.

MR. R. HUGH COLE, managing director of R. H. Cole & Company, Ltd., wishes through the medium of THE CHEMICAL AGE to thank all who wished him well during his recent illness. He is now making a good recovery following an operation for a perforated ulcer.

MR. K. A. KERRIDGE, a B.Pharm. of London University, has been awarded for two years the Pharmaceutical Society's Research Scholarship of £300 a year. He will work in the Department of Pharmacy at Chelsea Polytechnic on the aspects of trace metal chelation in relation to bacteriostasis.

DR. F. J. SIDDLE, hitherto director in charge of technical service on the board of the Dyestuffs Division of Imperial Chemical Industries, Ltd., has been appointed managing director of the Terylene Council. He has been a part-time member of the Terylene Council since it was set up in 1951.

MR. K. H. PALMER, a B.Pharm. of Nottingham University, has been awarded for two years the Pharmaceutical Society's Ransome Fellowship, which has an annual value of £300. He will work in the Pharmacy Department of the University on research in plant chemistry, with special reference to the separation of naturally occurring mixtures of alkaloids.

MR. C. C. BATES, M.I.W., A.M.I.P.E., has been appointed chief welding engineer of Costain-John Brown, Ltd., South Audley Street, W.1. He will also be responsible for the technical supervision of Welding Supervision, Ltd., their associate company. Mr. Bates, who is 39, joined Costain-John Brown, Ltd., from the British Welding Research Association, where for the past four years he was one of the senior development engineers responsible for helping industry to apply the results of research, and member firms with design, technique, metallurgical and inspection welding problems. Before joining BWRA, Mr. Bates was with Under Water Welders and Repairers, Ltd., and previously spent eight years in the Ministry of Supply. He served an indentured apprenticeship and received his initial training with Harland & Wolff, Ltd.

Obituary

The death occurred recently of MR. WILFRED FREDERICK DARKE, chairman of Price's (Bromborough), Ltd., since 1948. Mr. Darke, who was 62, had been associated with the Unilever Group since 1920, when he joined Christopher Thomas & Brothers, Ltd., Bristol. Later he went to Port Sunlight and was engaged in research before becoming chairman of the Bromborough company.

Taken suddenly ill on returning home from a cricket match, MR. STANLEY ARMSTRONG (48), an analytical chemist, of Saltburn, died in hospital 24 hours later. At an inquest, Dr. T. Skeoch, who made a post-morten examination, said death was due to acute enteritis from toxic absorption of an unknown origin, but nothing had been found to reveal how this occurred, although it was probably from something that had been swallowed.

Publications & Announcements

THE principal article in the latest number of 'BASF' (Vol. 3, No. 3, July) is concerned with the final release of the Badische Anilin und Soda Fabrik from Allied control on 28 March, and with their hopes for the future. Other articles deal with the advantages of synthetic over natural indigo, the uses of indigo in West Africa, the grave-cloths of Pope Clement II, industrial psychology, the testing of herbicides, and the making of a colour film about them. As usual, the journal is very well produced and illustrated.

* * *

ISSUED by the Mond Nickel Company, Limited, Sunderland House, Curzon Street, London, W.1, the latest (June) number of *The Nickel Bulletin* includes an article which gives the composition, physical and mechanical properties of the alloys of the Hastelloy type. These casting alloys (nickel-molybdenum-iron, nickel-chromium-molybdenum-iron and nickel-silicon) are employed principally in chemical engineering and oil refining for their high corrosion-resistance. Abstracts of recent literature on electrodeposition include a critical survey of substitute finishes which have been tried in the USA during the period of restricted availability of nickel, a description of a method for plating manganese-base materials, and an explanation of the mechanism of black nickel plating.

* * *

INCLUDED in the summer issue of *Progress,* the magazine of Unilever Limited, Unilever House, Blackfriars House, London, E.C.4, is an article on 'The Task and Methods of the Monopolies Commission.' This has been written by Dame Alix Kilroy, Under Secretary, Board of Trade, who was secretary of the British Monopolies Commission from its establishment early in 1949 until March, 1952. The article is most interesting, particularly in view of the Commission's recent report on matches and match-making machinery. Dame Alix points out that the Commission's reports so far have not produced the hostile comments that were anticipated; such criticism as there has been has concentrated on the alleged slowness of the Commission and on the policy of the legislation under which the Commission works.

A BOOKLET just published at 3s. 6d. by the Federation of British Industries, 21 Tothill Street, London, S.W.1, reproduces the introductory papers given at the Third Conference of Industrial Research Directors and Managers held under the auspices of the FBI at Ashorne Hill, Warwickshire, earlier this year. Summaries of the discussions are also included. The theme of the conference was the vital need to make full commercial use of research and discovery. The principal speakers included Mr. G. S. Samways, joint managing director, the Metal Box Company, Ltd; Dr. N. P. Inglis, director of research, Metals Division, I.C.I., Mr. L. H. Williams, deputy commercial manager, Shell Petroleum Company, Ltd.; and Mr. H. W. G. Highnett, superintendent, Development and Research Laboratory, Mond Nickel Company, Ltd.

* * *

IN 'Scientific Research as it Proceeds in Mellon Institute' the description of work in progress in the 73 separate Fellowships is necessarily brief. Mellon Institute, Pittsburgh, Pennsylvania, is an endowed, non-profit making, corporate body for the carrying out of comprehensive investigations on important problems in pure and applied science, for training research workers, and for providing technical information. Among the many research projects at present in operation, there are investigations into chain and arc-welding, the properties of glass, the chemistry of yeast, the status of piperonyl insecticides, new organic coatings, and the control of moisture in dry cleaning—a typical selection of the wide range of interests covered by the Institute.

* * *

FLUSH doors are gaining in popularity, but it may not be generally known that synthetic resin adhesives enable them to be used for exterior as well as interior purposes. The July issue of *Aero Research Technical Notes,* published by Aero Research Limited, Duxford, Cambridge, gives details of the processes in which 'Aerolite' KL, a urea-formaldehyde resin adhesive, is used for flush door manufacture by an Uxbridge joinery company. It is claimed that apart from lower cost, 'Aerolite' has been found to offer other advantages over casein as an adhesive.

Law & Company News

Commercial Intelligence

The following are taken from the printed reports, but we cannot be responsible for errors that may occur.

Satisfaction

BRADITE, LTD., Bethesda, cellulose manufacturers. (S., 15/8/53.) Satisfactions 9 July, of mortgage, and charge and a mortgage, both registered 9 February, 1949.

Changes of Name

The following changes of name have been announced: A. Boake, Roberts & Co. (Manufacturing), Ltd., to A. Boake, Roberts & Co., Ltd., on 3 July, 1953. National Titanium Pigments, Ltd., to Laporte Titanium, Ltd., on 20 July, 1953.

New Registrations

Firesnow, Ltd.

Private company. (522,147.) Capital £100. Fire, mining, hydraulic, mechanical, electrical and chemical engineers. Directors: C. Morris, Mrs. L. Morris. Reg. office: Withyfold Drive, Hurdsfield, Macclesfield.

George Mayor (Pharmaceuticals) Ltd.

Private company (522,382.) Capital £1,000. Manufacturers of and dealers in chemicals, gases, drugs, medicines. Directors: N. L. Allport, W. A. Ward, C. M. Edridge. Reg. office: 325 Kennington Road, S.E.11.

Chlorine Bleach Products Ltd.

Private company. (522,403.) Capital £1,000. Manufacturers of and dealers in chemicals and chemical substances, liquids and gases of all kinds. Directors: E. Smith, G. Smith. Reg. office: Lower Nuttall, Ramsbottom, Manchester.

Topmark Ltd.

Private company. (522,445). Capital £1,000. Manufacturing, pharmaceutical, analytical, photographic, advising and dispensing chemists and druggists. Directors: W. Seach, Mrs. D. M. Seach. Reg. office: 95 Second Avenue, Newcastle-on-Tyne, 6.

Whitehead Chemical Co. (Waxes), Ltd.

Private company. (522,249.) Capital £100. Manufacturers and merchants of chemicals, and all types of waxes, natural and synthetic and petroleum residues. Directors: A. Whitehead, R. W. Hague, J. Waldie. Reg. office: Holt Mill Road, Waterfoot, Lancs.

Claflin Chemical, Ltd.

Private company. (522,215.) Capital £500. Manufacturers of and dealers in substances used in medicine, pharmacy, perfumery and toilet articles of all kinds. Subscribers: P. J. Gaynor, Ann K. Hutton. First directors are to be appointed by the subscribers. Solicitors: Mckenna & Co., 12 Whitehall, S.W.1.

Increase of Capital

The following increase of capital has been announced: AJAMA CHEMICAL CO., LTD., from £1,000 to £10,000.

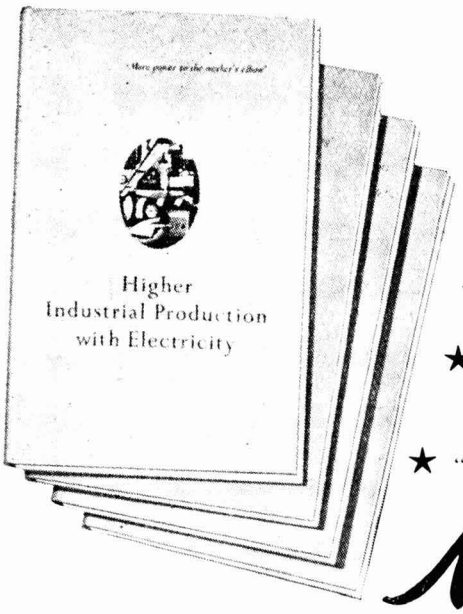
Company News

The Fullers' Earth Union Limited

To meet the cost of additions to the company's factory at Cockley, so that the output of activated bleaching earth can be extended by about 10,000 tons a year, the directors of The Fullers' Earth Union Limited are making a rights issue to ordinary shareholders of 266,934 5s. ordinary shares at 10s. 3d. each in the proporton of one for every three shares held on 4 August. The final date of acceptance is 25 August, when payment in full is due.

Hardman & Holden Limited

Trading profit earned by Hardman & Holden Limited during the year ended 31 March last was £145,987, as compared with £238,933 for the previous year. The dividend of 22½ per cent for the year compares with 27½ per cent for the previous year. The annual meeting, to be held in Manchtester on 1 September, will be followed by an



★ "HIGHER INDUSTRIAL PRODUCTION WITH ELECTRICITY"

★ "LIGHTING IN INDUSTRY"

★ "MATERIALS HANDLING IN INDUSTRY"

★ "ELECTRIC RESISTANCE HEATING"

New BOOKS ON Industrial Productivity

PRODUCTIVITY is one of the vital problems affecting Britain today. One way to increase productivity is to put POWER at a man's disposal; and the best way to do this is with electricity.

As a contribution to the solution of this problem, the British Electrical Development Association is now publishing a new series of books for management and executives in Industry. The first four are now available: "Higher Industrial Production with Electricity" describes a wide variety of modern production methods; "Lighting in Industry" shows how lighting can affect individual output, how its effectiveness can be assessed, and how improvements can be made; "Materials Handling in Industry" shows the way to increased productivity by improved handling; and "Electric Resistance Heating" indicates where, and how, this unique method of producing heat without combustion can be applied.

The post-free price of each of the books is 9/- and copies can be obtained from the British Electrical Development Association, 2 Savoy Hill, London, W.C.2, or from your Electricity Board.

The Association has produced a film called "A Case for Handling" which illustrates by practical demonstration the vital part that improved materials handling can play in all industries. It runs for 32 minutes, and is available on free loan.

Electricity for PRODUCTIVITY

extraordinary meeting to consider a proposal to capitalise £250,000 and distribute one new 5s. share for each 5s. unit held, as known.

The Mirrlees Watson Company, Limited

Satisfactory demand for sugar, chemical and allied machinery is reported by the Mirrlees Watson Company, Ltd., of Glasgow. Several very large sugar contracts were completed during the past twelve months and new orders booked include a 15 roller 36 in. by 78 in. turbine driven mill for St. Kitts, and a complete new mill to handle 2,500 tons of cane per day for Venezuela. On the chemical and distilling side substantial Canadian orders have been booked for evaporators for corn-steep liquor and a grain distilling plant for potable spirit. Production of tanks for storage and transport of liquid oxygen has increased and steam ejector air pumps for use in the chemical industry have also been a feature of this group's activities.

Oldham & Son Limited

In a statement circulated in connection with the annual meeting of Oldham & Son, Limited, on 27 August, the chairman, Mr. John Oldham, points out that the company's ability to cater for the changing requirements of industry has been further expanded during the year by extensions to the Research and Technical Development Section, which would accelerate the development of new lines over the wide field of the company's battery, mining and electrical interests. The consolidated profit and loss account shows a net profit for the year to 31 March amounting to £262,541, a decrease of 18 per cent compared with the figure for the previous year. The proposed final dividend of 10 per cent on the ordinary shares makes 17½ per cent for the year.

Market Reports

LONDON.—Quiet conditions prevail in all sections of the industrial chemicals market, but the outlook is brighter than for some time past, and rather more activity is looked for in the autumn markets. Price changes have been few and quotations generally display a firm undertone. The basis price for lead oxide and litharge is now £126 15s, the last change being 11 August. On the same date the basis price for dry white lead was increased to £144 10s. There has been little activity in the coal tar products market, but contract deliveries are being taken up with fair regularity and conditions are unlikely to change just yet.

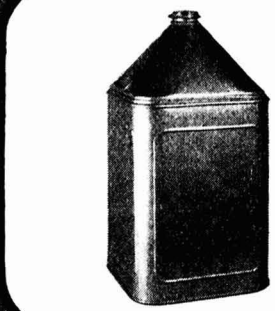
MANCHESTER.—The Manchester market for heavy chemical products during the past week, although slightly more active than a week ago, continues under holiday influences. These have affected particularly the movement of supplies of textile chemicals, for a large number of towns in the Lancashire cotton and Yorkshire woollen areas are now enjoying their annual stoppages. The prospects when conditions get back to normal are reasonably bright, however, and a steady flow of supplies to the textile and other leading industrial outlets is looked for. Among the tar products there is a steady call for creosote oil and, in the light distillates, for benzol, xylol and toluol.

GLASGOW.—Due to the large number of factories still closed owing to holidays, the past week generally has been somewhat uninteresting. However, next week should show a difference owing to the large number of orders on hand awaiting delivery.

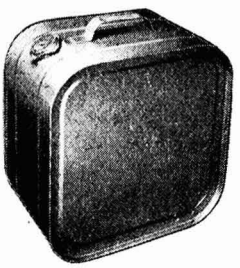
Mathieson Chemical Corporation

SALES and net profit of Mathieson Chemical Corporation, Baltimore, reached a new high figure for the six months ended 30 June. Net for the period, after taxes and preferred dividends, was $8,828,633 compared with $7,077,172 for Mathieson and Squibb on a proforma combined basis in 1952, and $5,135,516 for Mathieson alone in 1952. The merger with E. R. Squibb & Sons took place on 1 October 1952. The $5,000,000 cost of rehabilitating the company's Morgantown, W.Va., plant has been fully repaid, and earnings from the operation are now accruing to the corporation. The plant has been producing at a higher rate than at any time since its construction during World War II. Completion is also announced of the first successful natural gas well on lands leased by the corporation in East Texas. Additional wells are now being drilled in the area, with chances of successful production viewed as good.

Additional foreign agents recently appointed by Styrene Copolymers Limited are: Holland and Indonesia, Messrs. L. J. Volkers, Sarphatistraat 62, Amsterdam C; India, P. S. Daftary, Abdulla Building, D'Silva Road, Bombay 28.



The SQUARE TAPER



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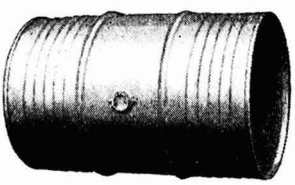

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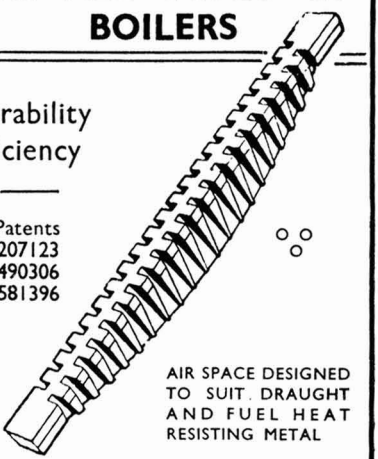

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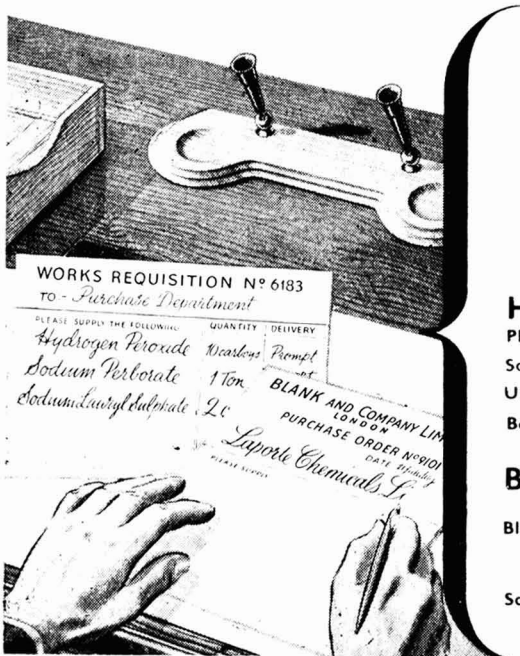

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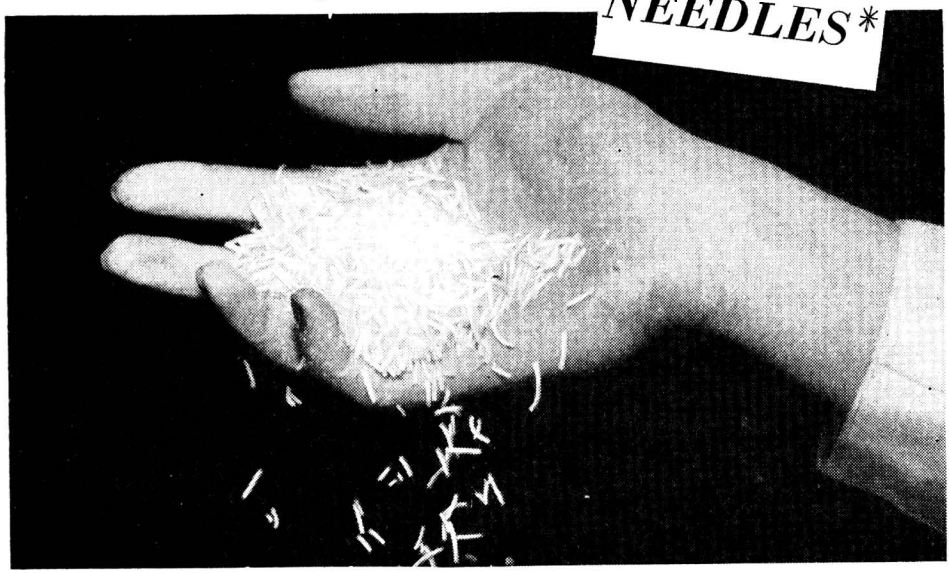

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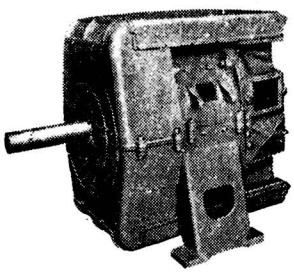

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