Chemical Age

VOL. LXXIII

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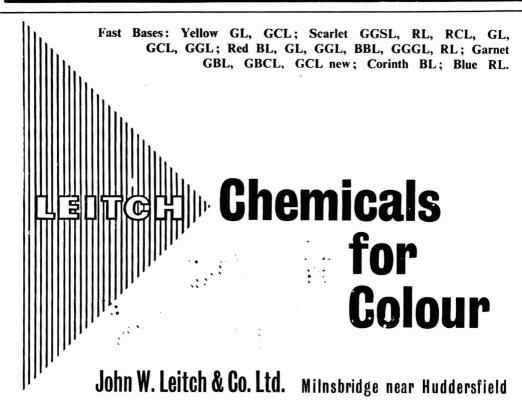
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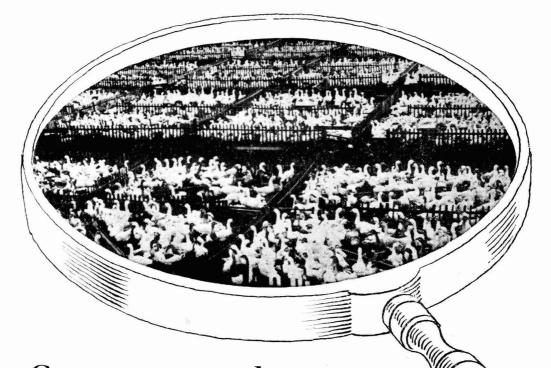


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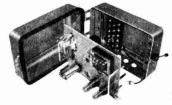
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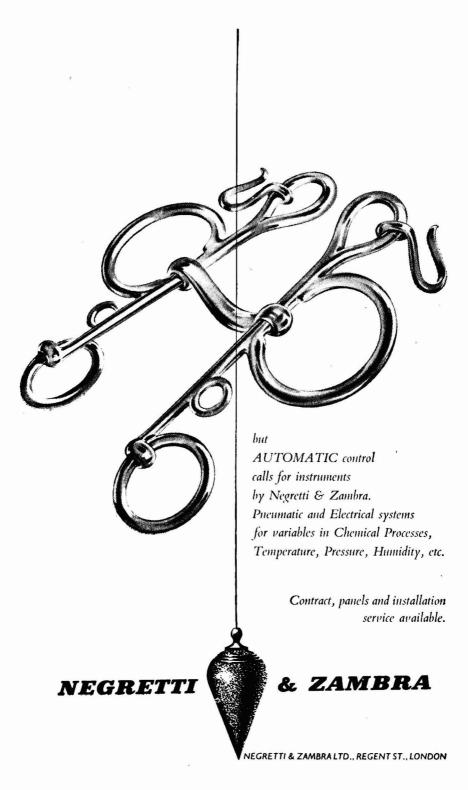
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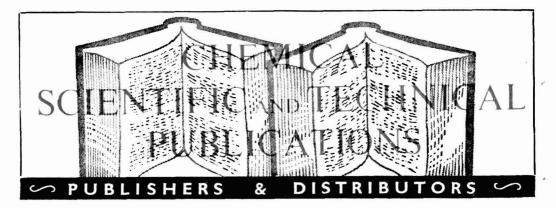
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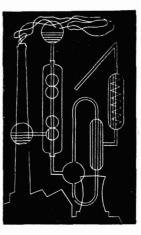
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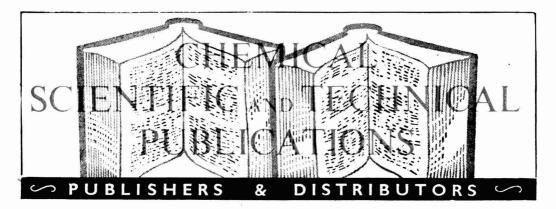
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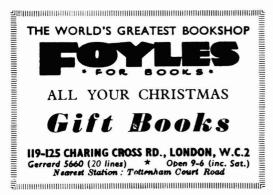
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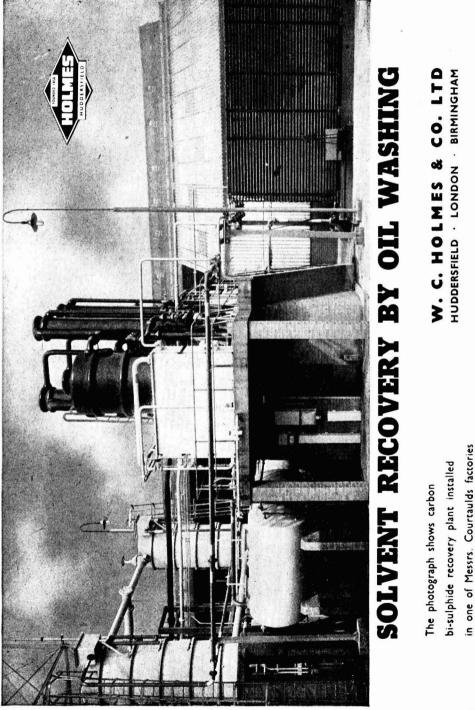
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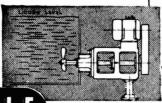


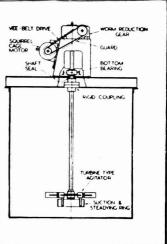
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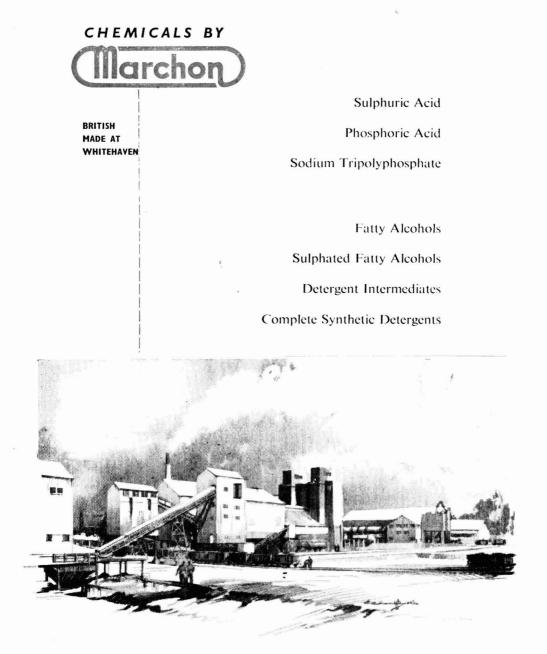
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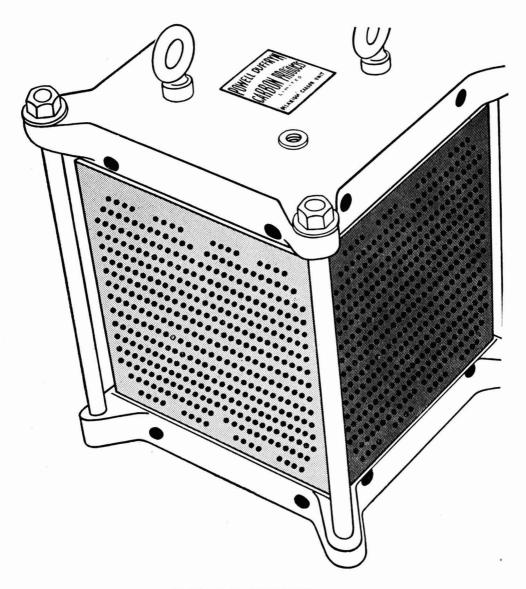
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Top Secret-Phosphate Rock

F ROM Australia on 26 October THE CHEMICAL AGE received word that important talks would soon be taking place in London to discuss the future of supplies of phosphate rock on Nauru and Ocean Islands. After wasting nearly two full days trying to obtain confirmation all we had was two emphatic denials and several inconclusive replies such as : 'We regret that we can neither confirm nor deny your report', 'We have heard nothing here' or 'No such meeting has yet been announced and so far as we know none is contemplated'.

On Monday of this week one of our national newspapers printed a dispatch from its Melbourne correspondent stating that Australian and New Zealand Government officials were on their way to London for joint discussions with the British Government on Ocean Nauru Island phosphate. Once again we started to try to obtain a confirmation or fuller details and for some time we received only confessions of ignorance. Australian and New Zealand Government officials were the only people who seemed to know anything. The office of the British Phosphate Commissioners, however, suggested that we contact the Commonwealth Relations Office for a statement. The statement we got, however, was that nothing could be added to the few words which had appeared in the newspaper in question. It was, we were told, merely another regular meeting of the Phosphate Commissioners. We were also told that it was unlikely that any official statement would be made at the conclusion of the meeting.

Phosphate is not available to any appreciable extent in either Australia

New Zealand and practically all or their needs come from Nauru, of Ocean and Christmas Islands. In 1954, 693,557 tons of phosphate rock Australia from were imported into Nauru, 159,021 tons from Ocean Island and 307,819 tons from Christmas Island. Christmas Island phosphate is owned jointly by the New Zealand and Australian Governments and reserves are not large. Nauru and Ocean Islands, on the other hand, are jointly held by the UK, Australia and New Zealand under a United Nations Trusteeship.

Nauru is a small coral island fourfifths (or 4,116 acres) of which is phosphate bearing. Under the trusteeship, Australia is entitled to 42 per cent of the phosphate and New Zealand to 16 per cent. The UK is also entitled to take 42 per cent of the yearly production which is limited by agreement. The same arrangement governs the smaller deposits on Ocean Island. The phosphate on the two islands is controlled by the British Phosphate Commissioners which consist of one representative from each of the three countries. At the present rate of production it is estimated that reserves will be exhausted in less than 70 years. But if the demand for Ocean Nauru Island phosphate is doubled, which is possible, reserves would be depleted in 30-40 years. And if Britain were to draw her share, Australia and New Zealand would soon be forced to look further afield for supplies.

From 1919 until 1936 practically no Ocean Nauru Island phosphate came to Britain. In 1936, with ocean freight rates low, the British Phosphate Commissioners approached certain large manufacturers with a view to bringing the high grade (80/82 per cent phosphate)of lime) Ocean Nauru Island rock to the UK and over 150,000 tons were shipped here between 1936-1940. From then until three years ago Britain did not take any and her share was divided between the other two interested countries-apart from the years when the islands were occupied by Germany. In 1951/52, 1,328,950 tons were exported from Nauru and of this 57 per cent went to Australia and 43 per cent to New Zealand. Three years ago the British Phosphate Rock Agency Ltd. was persuaded by certain of its members to import trial amounts. In 1952/53 two cargoes totalling 20,000 tons were purchased and were tested to see if they suited modern British manufacturing methods. In the following year 160,000 tons were purchased but in 1954/55 much less came here. In each year the amount purchased was far below the 42 per cent that Britain is entitled to under the treaty and less than half of the amount offered to Britain by the Phosphate Commissioners. British officials state that UK manufacturers have no intention of ever buying large quantities of Ocean Nauru Island phosphate and that the recent purchases were merely a three-year experiment to see if the rock could be used in case of an emergency. British manufacturers of superphosphate require regular supplies and the long journey of 50-55 days from Nauru does not make this possible. Furthermore, freight costs are very high for such a low-priced commodity. Nevertheless. Australia and New Zealand manufacturers have been alarmed by the purchases and have brought pressure to bear on their Governments to get the original treaty altered or to obtain some assurance from the British that they will not raid what they now look upon as their private source of supply.

Another cause for worry in the two Commonwealth countries is that certain British superphosphate manufacturers have recently expressed anxiety over the fact that most of the fertilizer trade's phosphate comes from French Morocco. The recent political disturbances there have not only alarmed British manufacturers but Australian and New Zealand makers as well. There has been talk to the effect that the UK industry should

have an alternative source of supply and Australians and New Zealanders have assumed that this means that it intends to draw upon Ocean Nauru Island. has been known for some time that output of the Moroccan mines cannot be increased so that if consumption of superphosphate increases our Commonwealth partners fear that we will ask them for some of their phosphate. British officials, on the other hand, deny that there is any likelihood of this. America holds over 50 per cent of the world reserves of phosphate and prior to 1940 between one quarter and one third of the rock mined in the US was exported, mainly to Europe. If the dollars were available the UK would more likely buy Florida than Ocean Nauru Island phosphate. This would be a question of sound business practice and not merely one of brotherly love. Florida rock is only slightly lower in quality than the Moroccan (68/75 per cent as compared with 73/75 per cent phosphate of lime). the supply can be regulated more easily and shipping charges are much less. In view of the fact that 60 per cent of the recent increases in the cost of rock is due to heavier transport rates, it can be seen that this is a very important factor.

In spite of reassurances the Australians and New Zealanders are still unhappy. For the past three or four years they have been worried about the future of their raw material supplies and it is believed that they are now asking the British Government to waive its claims to a share of the Ocean Nauru Island phosphate. It is to be hoped that the fertilizer trade will be consulted before the Government makes any amendment to the original agree-ment. While the UK does not want to use Ocean Nauru phosphate unless forced to, it is comforting to know that in case of a temporary emergency it would be possible to import a few shiploads. It is also to be hoped that the CRO will issue an announcement following the conclusion of this week's talks, for the trade (as well as the public) has a right to know what the Government is doing. We sincerely hope that we do not have to refer to Australia the next time we wish to learn something about this rather unfortunate situation.

Notes & Comments

More Antibiotics

¬HE annual symposium-conference on antibiotics in US took place last month at Washington, and once again new antibiotics came forward like a shower of starlets at a film festival. Cathomycin by Merck, synergistin by Pfizer, thiostrepton by Squibbs, vancomycin by Lilly, albamycin by Upjohn, and rovamycin from France. Nor does this represent the full list of newcomers. The main characteristic common to almost all is comparative freedom from resistance-development. The quest for new antibiotics has obviously been influenced by the stubborn problem of bacterial resistance to older ones. Although promising clinical tests have been conducted with most of these prospective additions to the medical armoury, it is too early to attempt picking out winners. For what chance speculation is worth, albamycin seems an attractive starter it has a very wide spectrum, shows no effects of toxicity after very high dosage tests, and is said to give the highest blood level contents of any antibiotic yet studied. If fuller clinical testing fails to reveal snags, albamycin is due for rapid elevation.

A Flash in the Pan?

EANWHILE we might reasonably wonder what is happening here in the home-country of the first antibiotic. Where are the hopeful starlets from British research? Is our principal government effort still limited to the able but far too miniature research station on the Somerset the coast. Medical Research Council unit housed in a building that was once a small private school? We were in at the start. but now it seems difficult to estimate how far we are behind the rest of the field. Is it too critical to say that we should still be limited to penicillin if overseas producers of other antibiotics had not been willing to set up subsidiary manufacturing units in Britain?

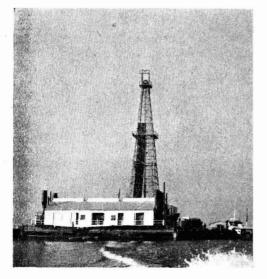
Synthetic Rubber

NY doubts that still lingered on the synthetic rubber project in Britain have now been dispersed—a plant to produce 50,000 tons a year will be in operation by 1958. The International Synthetic Rubber Company—which at present includes Dunlop, Michelin, Goodyear, and Firestone-proposes to raise £7-9,000,000. This is bound to be another large-scale development that links itself with the post-war expanded oil-refinery industry. The site of the new rubber plant is expected to be close to one of the larger refineries, and Fawley is being tipped fairly confidently. Malaya may feel dubious about this threat to her market, but there are good reasons for believing that the threat is nebulous. The demand for rubber steadily advances and the natural supply cannot expand at the same pace. For that reason the price of natural rubber far exceeds that of synthetic rubber. Every synthetic rubber plant that opens up is undeniably a threat to high prices for natural rubber, but that is hardly the same thing as a threat to natural rubber's share of the market at a reasonable price. Secondly, synthetic rubber has certain properties that suit specific tasks more effectively than natural rubber.

Far from Happy

THE argument that Malayan sales are one of the sterling area's best dollar-earning operations is genuine enough. Inasmuch as British synthetic rubber production helps to reduce the Malayan price, this source of dollar income will be slimmed. But for Britain herself-and the British dollar-sterling situation is the vital plank on which the whole sterling area's prospects must stand—the present rubber situation is far from happy. The year 1956 will see purchases of up to 50,000 tons of synthetic rubber from North America, all of it dollar expenditure. The British demand for the synthetic material must increase. For some needs synthetic rubber is technically superior; and it is cheaper. Manufacturers of rubber articles, especially motor tyres, cannot produce the best possible product at a worldcompetitive price if they are unable to procure the styrene-butadiene man-made polymer. So long as it remains unmade here it will have to be imported.

World's Deepest Well



A new world's well depth record is being set by this floating drilling rig in the Mississippi River marshland on the edge of the Gulf of Mexico. The wildcat oil well being sunk by Freeport Sulphur Company of New York and two independent oil producers has already reached a depth of 22,000 feet, more than 500 feet deeper than the 21,482-foot record established in California in 1953. and is still going down. Located six miles east of Freeport's large Grande Ecaille sulphur mine, the well is in a remote area accessible only by boat or plane. Thus far is has taken nine months to drill and has cost approximately \$1,500,000. While indications of oil have been encountered, no production has as vet been obtained

Colonial Research

THE latest report, for 1954-55, from the Colonial Research Council (1955, HMSO, Command Paper No. 9626, 9s) is glittering or depressing according to the reader's sense of financial perspective. Grants to a total value of £533,548 were approved for 52 new schemes of colonial research and for 48 supplementary schemes. Funds allocated for the period 1955-60 amount to an annual expenditure of £1,600,000, whereas expenditure in 1954-55 was £1,173,381. The question is whether these sums are large (a) when set against the size of the British annual budget, or (b) when set against the size of the colonies. Things may be getting better but are they yet good enough? The development of the colonies, better utilization of their resources and improvement of conditions of living for their populations, is our responsibility, and the whole 'colonial idea' stands or falls by it. Either we encourage progress and increase its pace or we might as well discard all higherminded philosophy of leadership and example. In these days sums of money that seem immense to the individual are not recognized as being in hard fact niggardly and inadequate. Yet even these new figures may be less than real, for it is revealed that £3,976,000 is an unspent balance from previous allocations to colonial research. The new £8,000,000 (5 years at £1,600,000) for 1955-60 includes this balance from the past! It remains to be seen, therefore, how much of the £8,000,000 is unspent in 1960 and handed on once again. The current annual cost of the subsidy for British farmers' purchases of fertilizers amounts to about £16,000,000, i.e., in one year twice as much as we now propose to devote to colonial research in five years. The only justification for so cautious and limited a policy is the fear that much of the money spent upon colonial research would be wasted, that it is more likely to be a poor investment than a good one. Surely investment in colonial research need not be as frugal and fearful as this? Research pays better than subsidies and emergency grants-in-aid.

The Preparation & Industrial Utilization of Hydrazine

by H. G. EMBLEM

HYDRAZINE has been known as a laboratory curiosity for many years, but has only recently become available in quantity. It was first obtained as hydrazine hydrate. and hydrazine salts, by Curtius (1) from the hydrolysis of triazo-acetic acid (CH N_2 .COOH)₃. Methods for the preparation of hydrazine include :—

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lysis or by photo-decomposition. (c) Oxidation of ammonia, or ammonia

derivatives such as urea.

A commercial method of preparation due to Raschig (2) is by the oxidation of ammonia with sodium hypochlorite. Chloramine is formed as an intermediate.

 $NH_3 + Na OCl \rightarrow NH_2 Cl + Na OH$ $NH_2 Cl + NH_3 + Na OH \rightarrow$

 H_2O + Na Cl + N₂ H₄.

The resulting dilute solution of hydrazine may be concentrated by distillation or by forming the insoluble sulphate. In this preparation nitrogen and ammonium chloride are formed through side reactions which are catalysed by metallic ions, particularly by copper ions (3). The addition of glue or gelatin, which increases the viscosity of the reaction mixture, greatly increases the yield of hydrazine (4).

Other large scale methods have been employed in Germany (5) (6) (7). In one process the reaction between ammonia and sodium hypochlorite was carried out under pressure, eliminating the necessity for a glue or gelatin inhibitor. In another process urea was reacted with sodium hypochlorite and the hydrazine produced was isolated as hydrazine sulphate. This was converted to the hydrate by reaction with caustic soda.

American Processes

Processes used in America for the large scale production of hydrazine have been reviewed by Mooney (8). One plant operates the modified Raschig process using urea. The formation of hydrazine from urea has been studied by Audrieth, Colton and Jones (9). In this country hydrazine is available commercially as hydrazine hydrate and hydrazine salts.

The insolubility of monohydrazine sulphate N₂H₄.H₂SO₄ is the basis of recent proposed for the isolation of methods hydrazine from the dilute solutions obtained in the Raschig synthesis. In one process (10) this solution, after removal of sodium chloride, is introduced as vapour into a saturated solution of monohydrazine sulphate. Sulphuric acid is added to maintain the pH < 3.5 to give more monohydrazine Monohydrazine sulphate may sulphate. also be formed (11) from dihydrazine sulphate by passing the dilute solution obtained from the Raschig synthesis, as vapour, into a solution of dihydrazine sulphate and maintaining at pH 3.5 to 5.5 by adding sulphuric acid. Monohydrazine sulphate is precipitated.

Anhydrous Hydrazine

Anhydrous hydrazine may be prepared by refluxing hydrazine hydrate with alkaline dehydrating agents such as calcium oxide, barium oxide, potassium hydroxide and sodium hydroxide. In this connection, the ternary system N₂H₄-H₂O-NaOH has been studied by Bonnemann and Audrieth An alternative method is to distil (12). hydrazine hydrate from calcium oxide under reduced pressure (13.) Hydrazine may also be dehydrated by fractional distillation of the hydrazine-water azeotrope with aniline (or a similar entrainer such as benzene) in an atmosphere of nitrogen, then stripping aniline from the hydrazine-aniline distillate (14).

Anhydrous hydrazine may also be prepared from hydrazine sulphate by ammonolysis with liquid ammonia (15). In a commercial method (16) dihydrazine sulphate is maintained mainly in the liquid phase by dissolving it in hydrazine, and treated with ammonia above atmospheric pressure. Anhydrous hydrazine is distilled from the reaction mixture.

Anhydrous hydrazine is a colourless fuming liquid, freezing at 2°C. It boils at

113°C at atmospheric pressure, and has a density of 1.005 at 15°C.

The most important chemical properties of hydrazine are its ability to form a hydrate N₂H₄.H₂O and its very powerful reducing action. It is a di-acid base forming two series of salts most of which, with the exception of the monosulphate, are readily soluble in water. Hydrazine is an electronpair donor and readily forms co-ordination compounds. Hydrazine salts, therefore, form well defined addition compounds with In a number of ways many metal salts. hydrazine may be compared with ammonia but is distinctive as regards its reducing ability. The relationship between hydrazine and ammonia has been discussed in detail by Audrieth and Mohr (17). The toxicology of hydrazine has recently been reviewed by Krop (18). Anhydrous hydrazine is much more toxic than hydrazine hydrate.

Hydrazine hydrate is miscible in all proportions with water. It is a corrosive, fuming liquid, attacking glass, rubber, and cork. Wherever practicable hydrazine should be used as an aqueous solution of hydrazine hydrate.

Industrial Uses

A number of the industrial uses of hydrazine depend on its reducing ability. An early example (19) is the use of hydrazine sulphate and alkali, or hydrazine hydrate and ammonia, to deposit copper on nonconducting surfaces. Hydrazine salts may be used in the separation and precipitation of metals. For instance, hydrazine sulphate can be used as a flux in rare metal refining (20) (21). Because of their reducing properties hydrazine compounds are also included in solder flux formulations (22), examples of suitable hydrazine compounds being the hydrohalides (23) and the phosphates (24). The reducing ability of hydrazine has been used as the basis of a method of silvering glass surfaces (25). Some attention has been given to the use of hydrazine and its derivatives in photography (26). Considerable quantities of hydrazine were used in Germany during the war for torpedo fuels and in rockets (27). Hydrazine salts may also be used in the application of certain azo dyes (28), and many organic nitrogen compounds are best prepared from hydrazine or hydrazine salts.

The reducing ability of hydrazine has been applied in the de-oxygenation of boiler feed water. Examples of this application include the use of a cation exchanger saturated with hydrazine solution to de-oxygenate and soften water (29), and the use of salts of hydrazine containing anions giving insoluble salts with lime and magnesia (30). One salt of this type is sodium hydrazine phosphate, Na₂ (N₂H₄)HPO₄.¹/₂ Na₃ PO₄, another being hydrazine oxalate which may be used in conjunction with trisodium phosphate.

Hydrazine is a very useful starting material for the preparation of complex nitrogencontaining organic compounds. These are frequently heterocyclic, many having been found useful as dyestuff intermediates and in the preparation of pharmaceutical special-The applications of hydrazine in ities. organic chemistry have been reviewed in detail by Byrkit and Michalek (31). Anhyhydrazine drous appears to be a much more vigorous reagent than hydrazine hydrate which is normally used. In general organic reactions involving hydrazine are performed in solution using hydrazine hydrate solution, or a hydrazine salt.

Alkyl and aryl halides react to give derivatives in which one or more hydrogen atoms are replaced by an organic grouping. Aryl hydrazines are well known as analytical reagents. Some have recently been found to possess insecticidal activity. A number of organic hydrazines are suitable for use as rubber plasticizers.

Hydrazine is di-functional, and can condense with poly-functional compounds to give resins. By condensing with adipte acid and heating the reaction product, a clear, stable resin can be obtained (31). Other resins may be prepared by condensing hydrazine with methyl methacrylate.

Semicarbazides

Urea or cyanates react with hydrazine or substituted hydrazines to form semicarbazides. Thiocyanates react similarly to give thiosemicarbazides. The semicarbazides. and particularly the thiosemicarbazides, are strong reducing agents and have been employed as antioxidants. Semicarbazide resembles hydrazine in its reactions with esters, acid chlorides and amides, and is used identify aldehvdes and to ketones. Many of the resulting semicarbazones show useful insecticidal properties. The thiosemicarbazone of acetyl-aminobenzaldehyde, and similar thiosemicarbazones continued on page 1268

Ion Exchange Resins

Joint Meeting Hears of Industrial Applications

A T a joint meeting of the London section of the Royal Institute of Chemistry and the Woolwich Polytechnic Scientific Society, held on Monday 14 November at the Woolwich Polytechnic, Dr. T. V. Arden of the Permutit Co. Ltd. delivered an address entitled 'Ion-Exchange Resins' before an audience of almost a 100, which included members, registered students and visitors.

Certain natural minerals with ion exchange properties had been used commercially for water softening for almost 100 years, said Dr. Arden. The discovery of the ion exchange properties of sulphonated coal in Germany in 1934 and the cation exchange properties of sulphonated phenolformaldehyde resins in England by Adams and Holmes in the following year gave a tremendous stimulus to the study of this With the development of the property. sulphonated cross linked polystyrene resins as cation exchangers and the corresponding aminated compounds as anion exchangers, agents of great chemical stability and commercial value were produced.

Polystyrene Resins

Polystyrene resins with SO₃H as the functional group (strong acid resins) exchanged cations for H⁺ irrespective of the anions present, but with COOH (weak acid resins) the exchange only occurred with weak acid anions present. Corresponding strong base and weak base resins were also used. The exchange capacity of such resins could be as high as 1 gram-equivalent per bulk litre of resin.

Applications of these resins were manifold. In the wine industry there were two main uses. By exchanging the K^+ by Na⁺ the wine was rendered more stable since in the form of Na salt precipitation of the tartrate did not occur. Excess organic acid could also be removed with a suitable resin. These processes were widely used in Australia and South Africa.

Crude glycerin could be purified to BP standard and a reduction of metal ion content to 5,000 ppm effected, thus avoiding vacuum distillation with its attendant difficulties.

Sugar refining was carried out in the US

with mixed cation and anion resins on the same bed. By working at 50°C with these resins inversion was reduced to 1-2 per cent. Treatment with a strong base anion resin and then weak acid cation resin avoided inversion but led to choking of the anion resin due to precipitation of metal hydroxides.

Streptomycin could also be purified by a suitable technique. The broth was passed through a weak acid resin which retained the streptomycin. This was then eluted with weak HCl and passed through a cross linked strong acid cation resin. The metals passed on to the resin but the streptomycin was unable to do this on account of the size of the molecule which could not pass through the pores.

The resins could thus be 'tailored' to meet particular purposes. The non-crosslinked resins were water soluble, but with increased cross linking they became harder, less soluble and of smaller pore size. Thus the pore size could be adjusted to prevent certain large molecules or cations from penetrating them.

Recent declassified work concerned the use of these agents to extract uranium. This use, in fact, now represented one of the greatest applications apart from water softening. The crude liquors, might contain only 0.1 g. per litre of uranium with other metals present in amounts more than 100 times this concentration. This element, however, readily formed very stable anion complexes with SO₄. Only iron formed such complexes and these were far less stable. When the uranium liquors passed through an anion exchange resin, only the uranium was adsorbed. The uranium was then completely eluted by acidified nitrate or chloride. This elegant process was now universally employed throughout the world for extracting this element.

Second World Atomic Fair

The second World Atomic Trade Fair will be held in Geneva during August 1957. The Swiss organizing committee said that the financial success of the first fair would enable them to reimburse 32 per cent of the stand rent paid by exhibitors.

Preparation of Hydrazine

continued from page 1266]

are of interest through their application in Another hydrazine tuberculosis therapy. derivative arousing interest in tuberculosis therapy is isonicotinic acid hydrazide together with related compounds (32).

Aliphatic azo compounds may readily be prepared in good yield from hydrazine. Among these may be mentioned azoisobutyric dinitrile (AZDN), prepared by reacting acetone cyanhydrin with hydrazine hydrate, followed by oxidation of the resulting hydrazo compound with chlorine. This compound and others prepared similarly are used as foaming agents for plastics and rubber (33). Acid azides may also be prepared from hydrazine. Some are useful as antiseptics and disinfectants (34).

A large number of other reactions of hydrazine and its organic derivatives have been reported. Those described above are typical and show the industrial possibilities of some of the reaction products.

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USSR Scientific Training

PROFESSOR W. F. K. Wynne-Jones, professor of chemistry at Durham University, one of the British scientists who visited Russia recently as a guest of the Soviet Academy of Sciences, declared at a meeting in Edinburgh that if Great Britain was to have any chance of remaining in the foreground of modern civilization she would have to put an effort into scientific training and research at least five times as great as at present.

He had been staggered by the equipment he had seen in teaching and research laboratories in the USSR and by the rate at which Russia was producing scientists. Russia was producing something of the order of 350,000 trained people a year, and these people were being sent where the State wanted them-into industry, schools, and research institutes.

'In Great Britain as a whole I do not think we produce 30,000 trained people a year,' said Professor Wynne-Jones. 'Their population is about four times ours, therefore per head of population they are producing two or three times as many trained people as we are.'

'I am convinced that the future lies with the countries that are prepared to regard scientific work as of absolutely vital national importance,' emphasized Professor Wynne-Jones. 'We are very far from doing so in this country. At the very time when other enlightened countries are trying to get considerable development we, if anything, are cutting back.'

US Sulphur Output

US sulphur industry The produced 498,591 long tons of native sulphur and 30,800 tons of recovered sulphur during Sep. This compares with 500,710 and tember. 34,500 tons respectively in August and 439,244 and 27,300 tons respectively in September 1954. Producers' stocks of native sulphur at the end of September totalled 3,000,346 tons, compared with 2,943,466 tons at the end of August and 3,229,407 tons at the end of September 1954. Stocks of recovered sulphur at the end of September amounted to 106,729 tons as against 112,837 tons at the end of August and 97,339 tons at the end of September 1954.

The Analysts' Dilemma

Paper Read on Colorimetric Determination of Elements

A^T the October meeting of the Midlands Section of the Society for Analytical Chemistry held in the University, Edmund Street, Birmingham, Dr. R. J. P. Williams of Wadham College, Oxford, read a paper entitled 'The Analysts' Dilemma—Colour or Stability?'*

In his introduction, Dr. Williams said that the determination of both large quantities and traces of elements was often most readily performed colorimetrically. The analyst would like a specific colour reagent for each element but the colour must be stable and proportional to concentration. Unfortunately some of the best colourproducing reagents did not form compounds which behaved in this way. Thus, the accurate determination of ferric ions by thiocyanate was made difficult by the slow decomposition of the ferric thiocyanate complex; again the instability of the complex made it unwise to use simple phenols, e.g. resorcinol, in the quantitative determination of titanium, despite the strong yellow colour which was produced from the reagent and the cation.

Reference to Feigl's monograph (1) showed that many of the hosts of colour spot tests for elements could not be turned to quantative use because of the transient character of the colour reaction. Noting this correspondence elsewhere in chemistry, it was reasonable to ask if there was any theoretical reason for connecting these quantities.

Colour & Stability

At this stage Dr. Williams thought it would be helpful to say a word or two about colour on the one hand and stability to chemical change on the other.

Chemical reaction (he said) required the uptake of energy normally called the energy of activation. Molecules did not decompose unless they were activated. It had to be remembered that chemical reactions as studied in solution only involved re-arrangement or exchange of electrons and not their total removal. The simplest reaction one could describe was that of the oxidation or reduction of a monatomic ion. Such reactions as:

 $Fe^{II} \rightarrow Fe^{III} + e$, $Ce^{III} \rightarrow Ce^{IV} + e$ when they took place in the gas phase, were simple ionizations and required large energy changes because the electrons were totally removed and not restabilized by interaction with another ion. The coupled reaction in water,

Fe^{III} + Ce^{III} = Fe^{II} + Ce^{IV} however, required less than one tenth of the energy of either of the above ionizations because the electron which was exchanged was always in a relatively stable state. The exchanges of electrons in solution between different atoms usually required but small energies. In fact ultraviolet light of wavelength around 100- $200m\mu$ was of sufficient energy to change the ionic state of most stable cations in solution and simultaneously to produce free radicals, the radiation activating the hydrate complex so that it decomposed (2): $M^{III} - OH_2 \rightarrow M^{II} - .OH_2 \rightarrow chain reactions.$

Irradiation with ultra-violet light was a common method for the production of free radicals. One way could now be seen in which the absorption of radiation (for example, the appearance of colour) might be linked with chemical change, the light absorbed activating the decomposition of the absorbing compound. If instead of the hydrate $M^{III} - OH_2$ we considered the series of complexes $M^{III} - I^-$, $M^{II} - Br^-$, $M^{III} - Cl^-$ then we would expect that the process:—

 $M^{III} - I^- \rightarrow M^{II} - X.$

 \rightarrow Decomposition with the production of free radicals

 \rightarrow Chain reaction

would require less activation energy the more readily the halide X^- gave up its electron, i.e. the more readily the halide was oxidized. The analyst used this difference in ease of oxidation in a number of reactions. For example, the chloride of cupric copper was stable but the iodide decomposed giving a method for the estimation of copper. The addition of ferric ions to iodide also produced some iodine but there was no corresponding reaction with chloride. These reactions were oxid-

^{*}Dr. Williams wishes it to be known that in this article the views of Dr. L. E. Orgel and himself are expressed.

ation-reduction reactions in which an electron was transferred from an anion to a cation. Such reactions formed the basis of many spot tests. Here the analyst had the opportunity of using unstable reaction products as he required qualitative reactions only. A few examples would illustrate the main argument.

There would be no objection to using the reaction:—

 $2 \text{ Cu}^{II} + 2 \text{ I}^- \rightarrow 2 \text{ Cu}^{I} + \text{ I}_2$

as a spot test for copper. The stages of this reaction were:-

(1) $Cu^{II} I^- \rightleftharpoons Cu^I I^-$

(2) $Cu^{I} I \rightarrow Cu^{I} + I$

(3) $\mathbf{I} + \mathbf{I} \rightarrow \mathbf{I}_2$

The formation of iodine would be the basis of this test.

As an intermediate in the reaction there was a radical I. Now many radicals are highly coloured so that their production could be made the basis of other spot tests.

TA	BLE	I
C		Tanta

	Some Spot Tests	
Reagent	Cation	Class of Reaction*
Resorcinol	Mo Fe Ti Ce	R
Strychnine	v".	R
2-Benzylpyridine	Sn	0
Phenylhydrazine	Mo	R
Phenylenediamine		R
Aniline	v	R
Cacotheline	Sb Sn Fe	0
Rhodanine		R
Benzidine		R
Diazine Green	Sb Sn	0
Thiocarbanalide	Os	R
Carbazide	Hg	R
Oxime and Ammonia	II Fe the reduction of the cat	0

* R is used to signify the reduction of the cation and O its oxidation.

Moreover, it was possible to obtain moderately stable free radicals by suitable choice of reagent. The triphenyl methane dyes formed one group of substances in this class. A similar compound to them was diphenylamine. It was readily oxidized with a redox potential high enough to allow it to be used as an internal indicator in the titration of ferrous ions by dichromate. The reaction

0 0

 $\dot{NH} \rightarrow NH^+$ which took place could $\theta / \theta /$

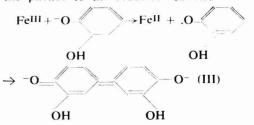
be used as a spot test for chromate. The charged free radical $\emptyset_2 NH^+$ was reasonably stable as the electron deficit was spread over the whole of the conjugated molecule, i.e. throughout the two phenyl rings.

Diphenylamine was in fact used as a spot test for vanadate, another oxidizing anion. Here oxidation of the organic reagent was being used to produce a colour. A similar reagent to diphenylamines was

In the presence of a large number of oxidizing anions and cations it yielded an intense colour which was due either to the semiquinone (I) or the quinone (II), both of which were free radicals:—



Cations which gave a colour with benzidine included Ce^{IV}, Pb^{IV} and Sn^{IV}, all strong oxidizing agents. Another parallel reaction was that of the polyhydroxyphenols such as gallic acid and tannic acid, which gave colourations when added to oxidizing cations such as Fe^{III}, Ce^{IV} or Ti^{IV}. In this case the reactions, which formed the basis of spot tests, had been extensive studies both with regard to their end products (3) and the kinetics of the reactions (4). Both studies led to the conclusion that the initial stage of the reaction was the transfer of an electron from the phenol to the reducible cation:—



The formation of final products such as (III) was considered to be particularly good evidence of radical initiation (3). Either the intermediate radical or the final product was coloured in these reactions.

The examples given so far concerned the oxidizing cations but reducing cations were equally able to give reactions of this. kind (see Table I). For example, cacotheline, an alkaloid related to brucine, reacted with reducing cations such as Sn^{II}, FeI^I (5), Ti^{III}, Sb^{III} and Re^{III} to give intense colours. The reactions were well known as spot tests. It was probable that the colour produced was that of a negatively charged free radical.

The characteristics of these reactions should be noted carefully. The first stage was the formation of a complex of the cation, though the complex might only be This stage was followed by transitory. the transfer of charge and required activation energy. The complex then broke up into a coloured free radical. Now, unfortunately, the radical was usually unstable and the colour either rapidly disappeared or changed to some other form. General quantitative use of such reactions would only be possible if the free radicals could be stabilized-a difficult task. Even if it were possible to solve this problem the colour produced in the reactions, that of the free radicals, would not be specific to one cation but would appear with either a large group of oxidizing agents or reducing agents (see Table I). The specific properties of the reaction scheme :---

$$\begin{array}{ccc} X^- + M^{++} \rightleftharpoons X^- & M^{++} \rightarrow & X \cdot M^+ \rightarrow \text{ radical} \\ \text{(a)} & \text{(b)} & \text{(c)} \end{array}$$

chain decomposition, were the formation constants of the complexes (a) and the activation energies of the second step (b). If we were to obtain specific colour reagents we had to make use of these properties and also prevent the decomposition of the activated complex to free radicals. But so far in this discussion the only coloured compounds had been the free radicals. Here was the analysts' dilemma-the colour produced in many reactions was by its very nature unstable.

A New Possibility

Returning to the first example of a coloured inorganic compound, ferric thiocyanate, a new possibility arose. This complex though unstable was of long life and was coloured. Might it not be possible then to find a series of coloured complexes which were sufficiently stable for analytical purposes?

Known coloured complexes were next examined.

The colour of a molecule represented absorption of radiation (visible light), an

absorption of energy. A molecule absorbed energy by virtue of its electrons and could not absorb all energies but only which corresponded with given those energy differences between discontinuous electronic states of the molecule concerned. We wished to know what type of electronic transition was usually responsible for colour in inorganic complexes and our approach would be an examination of coloured complexes in order to discover empirically the rules controlling their formation. Everyday analytical experience provided a useful starting point.

Coloured Complexes

Silver iodide was readily differentiated from the cloride by its yellow colour. Cupric and ferric iodide, in so far as they were known, were black, their bromides brown, chlorides (anhydrous) yellow and the anhydrous fluorides were the least coloured of all the halides. It was also known that the iodides of gold, lead, bismuth, mercury and many other cations were more strongly coloured than the other halides of these metals. It was seen earlier that the halides which were most readily decomposed, someeven by the absorption of light, were also the most highly coloured. It was clear that the greater the oxidizing power of the cation and the greater the reducing power of the anion in any complex the greater was thelikelihood that the complex would be coloured.

These facts had been known for some time and it was now generally considered that the colours were due to a process of mutual oxidation-reduction on the absorption of light (6), i.e. the same step which was required to activate the decomposition of the complex to give radicals. Thus the oxidation of iodide required less energy than the oxidation of chloride and the internal photochemical oxidation-reduction in a given iodide required less energy than in the corresponding chloride.

The light absorbed by the iodide was always of longer wavelength than that absorbed by the chloride. The problem under consideration, that of obtaining stable coloured complexes, was now seen to be related to the problem of stabililizing the activated state of the decomposition reaction. The analysis of the properties of a series of typical coloured complexes—the ferric phenolates—would show how this could be achieved.

Ferric, said Dr. Williams, was the best known oxidizing cation. It was no coincidence that more colorimetric reagents had been advocated for this cation than for any other. The production of colour when a ferric solution was added to a phenol was so general that the reaction was used as a spot test for phenolic -OH. Now phenols were weak reducing agents and most simple phenols were oxidized by ferric ions. The initial colour produced on mixing the components was due to the absorption of light by the complex which was then excited towards the transition state of the decomposition reaction. The light absorption could be thought of in terms of an (incomplete) change to a phenol radical and a ferrous ion*

Absorption Spectra

Although most ferric phenolates were unstable, Brode and Wesp (8) who wished to examine the absorption spectra of these complexes, found that the complexes were stabilized at low temperatures. Working at -50° C they were able to measure the spectra of a series of ferric phenolates.

			ABLE		
	Ferric P	henolate	s Al	osorption S	pectra
Subsi	tituent in	Phenol		Absorption	Stability
Unsubstit	uted			558 mµ	unstable
o-methyl				590 ,,	
m- ,,	• •			555	stable
p- ,,				575 ,,	unstable
o-chloro				570 ,,	
<i>m</i> - ,,				540 ,,	stable
p- ,,				565 ,,	unstable
o-bromo				570 ,,	
<i>m</i> - ,,				540 ,,	stable
p- ,,		• •		565 ,,	unstable
o-COOH				530 ,,	stable reagent
o-CHO				533 ,,	
o-nitro	•••			UV	stable
<i>m</i> - ,, '				505 mµ	.,
p- ,,		• •		515 ,,	

They also examined qualitatively the stability of the complexes formed. Table II showed that there was a good correlation between the stability and the position of the absorption maxima. A substituent in the aromatic nucleus such as $-NO_2$, SO_3H , -COOH or -CHO, electron acceptors, moved the absorption band to shorter wavelengths while a substituent such as $-NH_2$, $-CH_3$, -OH or a halide, electron donors, moved the absorption maximum to longer wavelengths. The latter ferric phenolates were unstable at room temperatures while the former were stable.

Among the former phenols were all the commonest quantitative reagents for ferric ions-solicylaldehyde, salicyclic acid and many sulphonated phenols such as chromotropic acid. A quick look at these observations would suffice to show how a series of phenols of increasing electron donor power similar to the halide series was $Cl^{-} < Br^{-} < l^{-}$. Of course if the reagent includes too many electron withdrawing groups then there was no longer colour associated with the formation of the complex.

The absorption band was now in the ultraviolet, e.g. 5-nitrosalicylaldehyde gave no colour with ferric ions.

A phenol of a somewhat different type was 8-hydroxyquinoline. In this reagent the electron-withdrawing group was the aromatic nitrogen which formed a chelate ring in the ferric complex. As with the simple phenols, so in the 8-hydroxyquinolines the absorption moved to shorter wavelengths if electrophilic substituents were added to the aromatic rings, e.g. $-NO_2$ (9).

The colour reactions with ferric ion were not unknown with other cations such as titanic and ceric, other oxidizing cations, but the position of the absorption band was different for each of the different metal complexes. The absorption was a property of the complex and not of the reagent which formed the complex. Colorimetric determination of vanadium, molybdenum and ruthenium as well as of ferric were possible with 8-hydroxyquinoline but for each of the methods there was a different wavelength of optimum absorbancy. In certain cases the choice of wavelength permitted the determination of three cations with one reagent in the same solution (10).

Phenanthrolines

Another series of ferric complexes which was remarkably similar to the phenolates was the phenanthrolines. Again the addition of electrophilic substituents shifted the absorption of ferric phenanthrolines to shorter wavelengths (11). It seemed highly probable that the colour was again due to

^{*} The oversimplification here was that the activation step of decomposition was identical with that on absorption of light. In fact, the activation step was the complete transfer of an electron from the ferric ion to the phenolate ion. On absorption of light complete transfer of the electron did not occur in this case and rather was the electron to be thought of as being only increasingly associated with the ferric ion in the excited state. The absorption of light was a step on the way to activating the decomposition of the complex. The modifications which were required in the argument were obvious and for the purpose of illustrating principles it was thought better to oversimplify. The details of the absorption spectra appeared in a recent article (7). In halides the activation energy of decomposition was directly relatable to the energy of the absorption band, in which case an irreversible photochemical oxidation-reduction took place unlike the reversible partial oxidation reductions which were required in a quantitative colour reagent (6) (14).

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partial photo-chemical oxidation-reduction. Ortho-phenanthrolines also formed complexes with ferrous and cuprous ions and these complexes were used in the colorimetric determination of copper and iron (12).

TABLE III Absorption Spectra of Some Ferrous and Cuprous Phenanthrolines

Substituent in 1, 10 phenanthroline			Positi Absor Maxi Fe 11	ption mum Cu I
			Complex	Complex
None			510	435
4, 7 diphenyl			533	
3-carbethoxy 4-hydroxy			561	
Several 4,7 diammonium	salts		530-560	
2-aza 8-carbethoxy	7-hyd	гоху		
4- methyl			605	
3-(2'-pyridyl) - 4 - p - m		enol		
cinnoline			530	470

The effect of substituents in the phenanthroline upon the spectra of the reducing cation complexes was however in the opposite direction from that in those of the oxidizing cations.

We could interpret this as meaning that the reducing cations acted as electron donors while oxidizing cations acted as electron acceptors. Such an assumption would be in keeping with the facts made evident in the discussion of spot tests for there it was made clear that reducing cations gave colours with an entirely different group of reagents from those found useful for the identification of oxidizing cations. Support for the idea came from the general observation that all strongly reducing cations gave intense colours with orthophenanthroline.

To quote but a few examples there were the well known reactions with divalent molybdenum, chromous, ruthenous, osmous and titanous. Many similar ligands also produced colours with these cations, the best known being di- and ter-pyridyl. The effect of substituents, mentioned above, was dealt with in more detail in Table III, which showed how the absorption maximum in the substituted ferrous phenanthrolines was shifted to longer wavelengths by electron The effect of too many such acceptors. substituents was to move the absorption to such long wavelengths that oxidation of the complex became facile (13).

General Principles

It was not the purpose here to give details of individual examples and the stress had been put upon the general principles involved. In attempting to summarize, the following two points stood out. A colorimetric reagent could be found for weak

oxidizing cations if a complexing agent for them could be discovered which was also a weak reducing agent. Similarly a reagent could be found for weak reducing cations if a complexing agent for them could be prepared which was also a weak oxidizing agent.

Quantitative Colour Reagents

Table IV contained a list of examples where a suitable combination had been found. It would be observed that of these reagents there were none that were not capable of forming π bonds-double bonds with the cations. It would appear that a more complete charge transfer was permitted in such bonds than in the more usual σ bonds without so great a possibility of a reaction producing free radicals. Such hypotheses as this had no place in so general a discussion as the present one, however, so the details of this argument would be left to find their proper place elsewhere (14).

Some Quan	titativ	e Colour Reagen	ts
			Class of
Reagents		Cation	Reaction*
		II	
Terpyridyl		Со	0
		ш	
Acetylacetone		Fe	R
		IV	
Oxime (plus bromine)		Ni	R
December 1991 937 178		и?	10 M 27 GW
Dithiol	• •	SN	R?
		vi vi ?	
Thiocyanate		Mo W Re	R
		II	-
Cupferron		UO2	R
		III	
Pentanedione	• •	Mn	R
		пп	0
Dipyridyl	• •	Ru Os	0
<i>a</i>		IV	
Chromotropic acid		Ti	R

TABLE IV

* R is used to signify a partial reduction of the cation on absorption of light and O a partial oxidation of the cation.

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Analysis of Alcohols

Small Scale Preparation of Halides

IDENTIFICATION of an alcohol by determination of the refractive index of its iodide or bromide is sometimes desirable. An apparatus has been designed by Mr. P. Sleightholm of the chemistry department, Technical College, Blackburn, for the preparation of about one ml. of product, which is sufficient for a determination of refractive index and also of boiling point.

A 20 ml. Pyrex glass distillation flask has its side arm attached to the side of a 6 in. by 1 in. filter tube. A 6 in. by $\frac{3}{4}$ in. filter tube serves as a cold finger condenser. The top of the distillation flask is closed by a good quality cork or better by a ground glass stopper.

Two ml. of the alcohol is placed in the distillation flask together with 10 ml. of constant boiling hydriodic acid or 12 ml. of constant boiling hydrobromic acid. The contents of the flask are then distilled after adding a few pieces of porous pot. The distillation is stopped when no more oily drops appear in the receiver. The lower layer of the aliphatic halide is removed with a capillary pipette and transferred to a test tube where it is washed with an equal volume of concentrated hydrochloric acid (to remove unchanged alcohol).

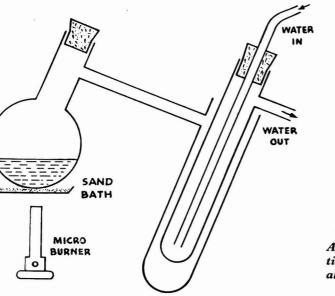
The pipette is again used to remove the acid, and the halide is washed with distilled water, a five per cent solution of sodium bicarbonate and distilled water. The final product is transferred to a small specimen tube and dried with a few pieces of calcium chloride. The residues in the distillation flask are redistilled in an apparatus with a thermometer and the constant boiling fraction recovered for use in later preparations.

Scottish Oils Ltd. Plan Close-Down

Scottish Oils Ltd. have announced plans to restrict output at their Roman Camp works and to close down gradually on production of the three supplying mines. The process is expected to take about two years. The Roman Camp works retort shale to produce crude oil and naphtha and ammonia liquor, and would require extensive modernization to bring it again to an economic production capacity.

BP Bottled Gas

Recent extensions to the gas bottling plant at BP's Grangemouth Refinery, on the Firth of Forth, have included the commissioning of a new filling shed. The refinery now has a bottle-filling capacity of 16.000 tons a year of liquid petroleum gas.



Apparatus for the preparation of small quantities of aliphatic iodides and bromides

Automation in the Laboratory

Baird & Tatlock's New Instrument

THE fully automatic laboratories built by Baird & Tatlock (London) Ltd. and shown at the 1955 British Instrument Industries Exhibition at Earls Court, London, and later at the international conference on the peaceful uses of atomic energy at Geneva, are believed to be the first of their kind in the world.

The Analmatic laboratories are designed to carry out chemical analyses completely automatically and so relieve the scientist of the problems of routine analytical work. On continuous working one automatic laboratory could, it is claimed, replace three skilled men, each operating an eight-hour shift. In certain cases, say the manufacturers, it may be possible to design an Analmatic laboratory which could carry out operations normally requiring a laboratory team.

In an Analmatic laboratory, analysis is done completely automatically—from taking the sample to recording the final result on a chart. It is said to be possible to go even further and connect the laboratory to a full scale production process so that any variations from a pre-determined standard, detected by the instrument, are automatically signalled to the control valves and mechanisms in the main plant and appropriate corrective action taken.

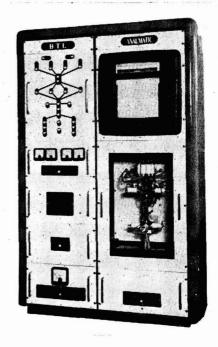
Standard Units

The Analmatic laboratory normally comprises one or more standard units which have been developed to carry out specific laboratory techniques. These units are assembled with their power supplies and services in standardized cabinets and interconnected in such a manner that the desired process is carried out and the result automatically recorded.

The centre of operations is the metering and mixing unit. Electro-magnetically controlled valves admit the various reagents and fluids to the vessel and electrical probes determine the volumes of the various liquids entering the vessel. Mixing is achieved by gas stirring.

The sequence of operations and their timing is controlled by a sequence control unit and a time pulse unit. This is the 'brain' of the instrument. Apart from determining the order and timing of each operation, the unit must also decide before any operation is started that the previous operation has been satisfactorily completed. This is done by means of 'feed-back' signals from the various mechanisms of the laboratory. The sequence control unit is also connected to an illuminated diagram giving a visual indication of the various stages of the operations carried out by the laboratory.

Working in collaboration with the sequence control unit is a fault alarm. When the sequence of operation is interrupted or when any operation has not been satisfactorily completed, no 'feed-back' signal will be received: an alarm signal will be given both visually and aurally and the sequence control unit will stop all further operations until the fault has been cleared. A faultfinding multi-point switch connects each



A BTL Analmatic Automatic Laboratory

main electrical circuit separately to a pilot light. With the aid of this switch the faulty circuit may be readily located.

The characteristics of the sample are determined by analysing units which receive the various substances after their passage through the metering and mixing vessel. The results of the analysis are then presented graphically on the chart of a recorder unit. The type of analysing unit and recorder unit employed will obviously depend upon the nature of the work for which the laboratory is designed.

Analysis of Uranium Liquors

For instance, in the Analmatic laboratory used for the analysis of uranium liquors, developed in co-operation with AERE, Harwell, measured volumes of samples are drawn automatically from four different sources in turn. To each of these samples two reagents are added and the mixture is then forced up the centre of a double walled tube by gas pressure and mixed in a chamber at the top of the tube. The mixture is delivered to an absorptiometer, where the intensity of the colour produced by the reaction is compared against a known standard, so that the concentration of the sample can be readily determined. The recorder unit indicates and records the concentration of the samples drawn from the various sources in four different coloured inks.

In another Analmatic laboratory, demonstrated at the British Instrument Industries Exhibition, a unit for pH measurement is also incorporated.

The Analmatic laboratory can at present handle liquids and gases and within these limits carry out a wide variety of chemical analyses. Additional units are at present being developed to extend the range of laboratory techniques capable of being accomplished by the instrument.

The immediate applications of the equipment are likely to be found in process control laboratories and large industrial laboratories where repeated analyses of a standard type are being carried out. It is likely to be of particular benefit in refineries, sewage works, solvent recovery plants, the recovery of precious metals and expensive chemical compounds from large scale production plants, hospital laboratories engaged in routine analysis, the control of the various stages in the production of pharmaceuticals, testing the characteristics of chemical products, petroleum testing and gas analyses.

The manufacturers stress that it should not necessarily be assumed that an Analmatic laboratory already exists to fit a particular requirement. It may be necessary to adapt the instrument and produce additional units to carry out specific operations. For example the handling of solids, centrifuging, measuring specific gravity and combustion present a number of practical problems which must be overcome.

Analmatic laboratories have been constructed to provide easy servicing and replacement of components. In cases where numbers of similar Analmatic laboratories are being employed, spare units could be provided in case of failure.

The application of the principles of automation to the laboratory itself, although revolutionary, is but the logical development of modern techniques. The Analmatic laboratories so far developed are, no doubt, only the first of a wide range of instruments which will be used for automatic process control and automatic laboratory analyses. In industry the application of scientific methods will increase the demand for scientists for research and development and for the scientific control of fac-Analmatic automatic tory processes. laboratories may help to overcome the shortage of trained technicians and scientists by releasing those engaged on routine work for more fruitful and creative work.

Junior Award Winner

FOR outstanding contributions to the literature of chemical engineering, Professor William E. Ranz, Ph.D., B.Ch.E., A.C.S., A.I.Ch.E., associate professor in engineering research at Pennsylvania State University, was awarded the 1955 Junior Award in Chemical Engineering granted by the American Institute of Chemical Engineers at the Hotel Statler. Detroit, on 29 November.

Professor Ranz was selected for the award in recognition of his paper 'Friction & Transfer Coefficients for Single Particles & Packed Beds' published in *Chemical Engineering Progress* in 1952. The award is given for the best paper published by a junior member of the institute. Professor Ranz is an authority on aerosols, sprays, heat and mass transfer, and fluid mechanics.

Autumn Research Meeting

Gas Engineers Hear Technical Papers

PAPERS covering many aspects of the gas industry were presented at the 21st Autumn Research Meeting of the Institution of Gas Engineers, held at Church House, Westminster, London SW1, on 22 and 23 November.

In a paper 'The Bacterial Oxidation of Gas Liquor' by E. H. M. Badger, London Research Station, the Gas Council (research communication GC26), experimental work was described on the bacterial oxidation of spent gas liquor from vertical retorts, and on some of the separate constituents, without dilution with sewage. The process used resembled the activated sludge treatment of sewage in that the aeration was carried out in glass vessels or metal tanks and settled solids were recirculated.

It was shown that it was possible to oxidize phenol and ammonium thiocyanate separately and also a mixture containing 1.000 ppm of each constituent in continuous flow. The efficiency of destruction was 99 per cent or better. Further experiments were then carried out with spent liquor from vertical retorts, which contained many different kinds of phenolic substances. It was found that with a continuous flow of undiluted liquor the destruction on an oxygen absorption (O/A) basis was 96 to 97 per cent starting from liquor with an O/A of 10,000 ppm. The residue was resistant to prolonged aeration but the O/A could be further reduced by treatments with solid absorbents and azone.

Thiocyanate

It was found that the thiocyanate in spent gas liquor was more difficult to destroy than when pure phenol only was present. In some experiments the treated liquor still contained a high proportion of thiocyanate, but in others there was almost complete destruction after passing the liquor through two aeration vessels in series; and on some occasions after only one treatment.

It was shown by gradually increasing the proportion of gas liquor to water that the bacteria which oxidized phenols could survive when the concentration of all phenols was as high as 3,000 ppm. With pure phenol in water 1,500 ppm could be tolerated.

The thiocyanate bacteria reacted in equeous solutions containing 2,000 ppm of CNS⁻, and in an experiment on which sewage sludge was added to the solution the bacteria were active in a concentration of 3,590 ppm. In some cases the addition of sewage sludge seemed to have a tonic effect on the bacteria.

Pilot Plant Work

Work on a larger scale in a 1,000 gal. surface aeration unit had largely confirmed the laboratory work. Destruction of thiocyanates, however, was poor. The rate of uptake of oxygen, although adequate for normal sewage, was found to be insufficient for gas liquor. The rate of reaction was controlled by the rate of solution of oxygen and a retention time of about 14 days was necessary.

Results so far obtained with a new pilot plant indicated that the retention time could be substantially reduced by providing more intensive aeration.

W. M. Catchpole, chief research chemist, British Petroleum Co. Ltd., discussed 'Some Aspects of Research in the Petroleum Industry.'

The manufactured products of the petroleum industry fell into three main groups, motor gasoline, middle distillates and residual fuel oils, he said. Although other products might be of importance they represented a minor proportion of the yield from crude oil.

Competition within the industry was a large factor in stimulating research, particularly in improving the quality of motor gasoline. Changes in engine design had necessitated research on lubricants as well as on fuels.

The oil industry supported fundamental research in the expectation that the knowledge gained would occasionally lead to discoveries of significant commercial value, but mainly as an essential background to the applied or objective research that formed the major part of the research in the industry. The importance of adequate forecasting and forward planning in the direction of this applied research could not be over-stressed, said Mr. Catchpole.

The information gained in research had resulted in the industry making more and more use of catalytic processes to achieve molecular rebuilding of hydrocarbons in the desired direction and it was evident that so far as motor fuels were concerned the fuel of the future would tend to become a blend of synthetic hydrocarbons produced from erude oil by catalytic processes.

Town Gas

Where the gas industry was turning to oil as a raw material it could assist the petroleum industry to achieve the desired balance of products by having available processes that could produce town gas economically from fractions of crude oil other than gas oil.

The final results of experiments carried out by the Water Pollution Research Laboratory in collaboration with the gas research laboratory of the University of Leeds were contained in a paper entitled 'The Treatment of Gas Works Liquor in Admixture with Sewage in Percolating Filters' by W. H. Blackburn and T. G. Tomlinson. This paper was a continuation of a Gas Research Board publication presented at the 1951 Autumn Research Meeting.

For earlier work a small scale sewage works at Stivichall and a battery of small scale filters were placed at the disposal of the laboratory by Coventry Corporation. The spent liquor was obtained from Coventry and Learnington gas works. It was recognized, however, that full-scale trials were necessary before final conclusions could be reached, as an important factor in the operation of percolating filters was their condition during the winter months.

The present paper gave the results of experiments with spent liquor from Hinckley gas works where electro-detarring of the hot gas was practised and the retort house liquor and purifier condensate were disposed of separately.

Large scale experiments previously carried out at Stivichall showed that spent liquor from Coventry or Leamington had an appreciable effect on the quality of the effluent when added to the sewage in a concentration of 0.5 per cent. These experiments were continued with spent liquor from Hinckley which, because of departure from normal gas works practice, had already been reported as having less effect on sewage purification than normal spent liquor.

Confirmation of this was obtained by experiments at Stivichall sewage works which showed that spent liquor from Hinckley, from which retort house and purifier liquors had been excluded, had a much smaller effect than the spent liquor from Coventry and Leamington tested in 1950.

Concurrently with the large scale experiments using Hinckley spent liquor, a series of small scale experiments was made with Hinckley retort house liquor, which, it was realized, contained large quantities of those constituents known to have a deleterious effect on sewage purification. In these experiments a retort house liquor before and after various treatments was added to sewage, and similar experiments were made with synthetic solutions made up to correspond to the treated retort house liquor. The addition of the crude liquor to the sewage treated increased the permanganate value of the effluent considerably and this effect was not materially altered by the removal of ammonia. Extraction of the phenols reduced the effect, and subsequent removal of ammonia caused a further re-The effect on the biochemica! duction. oxygen demand of the effluent was much less pronounced.

In discussing their paper the authors said that the effect of gas liquor on the treatment of sewage was usually assessed on the increase in permanganate value or biochemical oxygen demand. The biochemical oxygen demand test would probably be considered to be the most important.

Large Scale Experiments

Large scale experiments with hot liquor from Hinckley gas works showed that it was more easily treated than that from either Coventry or Leamington, but it had to be emphasized that the difference could not be entirely ascribed to hot gas detarring. At Hinckley the retort house circulating liquor was kept separate from the rest of the liquor and was disposed of separately. This practice was not thought to be feasible at large gas works situated in urban areas.

Experiments described in Section IV of the present paper showed that solvent extraction followed by distillation in the presence of lime effected a considerable improvement in the quality of the liquor. Such treatment, however, would be expensive.

Superphosphate Manufacture

AFRICAN Explosives & Chemical Industries Ltd., through their associated Rhodesian company, A.E. & C.I. (Rhodesia) Ltd., are to establish superphosphate manufacture at Rodia Factory, near Salisbury. Plans have been now completed and construction of the plant will start in the immediate future. A.E. & C.I. (Rhodesia) Ltd. will continue the manufacture of compound fertilizers, and a granulation plant is also being installed at Rodia Factory so that fertilizer products will be produced there in both powder and granular form. These plant extensions, which it is hoped will be completed by about 1958, will represent a capital investment of over £3,000,000.

Superphosphate has hitherto been imported from the Union of South Africa and from overseas, but the new plant in Rhodesia will have an initial capacity sufficient to meet the full requirements of superphosphate (both single- and triple-super) for fertilizer purposes, in the territories comprising the Central African Federation, for some years ahead.

The superphosphate project includes both phosphoric acid and sulphuric acid plants. The sulphuric acid plant will use pyrites mined in Southern Rhodesia as its raw material. It is hoped that these developments in the chemical field will help to promote other industrial development in the Central African Federation.

The two parent companies behind A.E. & C.I. in this venture are De Beers Consolidated Mines Ltd. and Imperial Chemical Industries Ltd.

Shortage of Scientists

THE SHORTAGE of trained scientists. engineers, and technicians which applied in the US as well as in Britain was referred to by Mr. Winthrop W. Aldrich, US Ambassador in London, speaking at the anniversary dinner of the Royal Society in London on 30 November. Both countries, he said, had a compelling obligation to adopt vigorous measures to meet this problem.

Sir Cyril Hinshelwood, the newly elected president of the society, responding to the toast of 'The Royal Society', proposed by Mr. Aldrich, said that it was perhaps surprising that one of the major problems today was the shortage of scientists. That was, perhaps, due to the tendency to treat young scientists merely as backroom boys. The remedy lay with the community at large, which should send more of its sons into that field. The situation was in fact rapidly improving.

Oil Tank Fires New Method of Control

THE CONTROL and extinguishment of oil tank fires by a new method called 'air agitation' developed by Socony Mobil Oil Co. Inc. of New York, was described in a film, 'Beneath the Flames', shown to the Press in London last week.

The method is based on the principle that an inflammable liquid can burn only when the flames are fed by vapours rising from it mixed with oxygen in a burnable ratio. When this burnable ratio is destroyed, the flames are no longer fed and the fire must die, or when the rate of mixing vapour and air is reduced, the feeding of the fire is slowed down and the flames and heat intensity are reduced accordingly.

At a routine fire control demonstration of this principle in the US it was discovered accidentally that the burning surface of a pail of kerosine could be cooled, vapours reduced and the fire extinguished not merely by a conventional cooling agent, such as ice, but equally well by unheated kerosine itself. Experiments were carried a stage further by blowing air into the bottom of the pail through a tube, thus pushing cooler kerosine from the bottom to the top and extinguishing the fire, though this time the kerosine already in the pail had put out the fire. Research on the idea continued on an increasing scale. A special group conducted tests on a variety of oil products in larger and larger tanks. Finally, in probably the biggest test fire ever made, a tank containing 2,000,000 gallons of home heating oil was set on fire. When the flames were leaping 50 ft. into the air, compressed air was passed into the bottom of the tank and in less than two minutes the fire was out.

The film shows the development of the air agitation method of oil fire control and its application to various inflammable liquids. Copies of the film, which is in colour and runs for 20 minutes, may be borrowed from the Information Section, Mobil Oil Co. Ltd., Caxton House, Tothill Street, London SW1.

IN THE EDITOR'S POST

' Enforced Order' Crusher

SIR,—Fundamental analysis of the effects producing crushing and of the practical requirements of continuous crushers have led to constructions on the principle of the 'Enforced Order' Mixer, and along the lines of Fig. 1 and of Fig. 7 (without reciprocating provision) of my article in THE CHEMICAL AGE, 1955, 73, 947). These constructions, with continuous rotation of the inner and outer screw-components in opposite directions, most effectively produce all necessary crushing effects, without any reciprocating motions.

Such constructions provide, as can be shown, 20 main distinguishing features in crushing performance over conventional types, and furthermore provide minimum wear, easy adjustability (axial displacement of inner screw component) to compensate for wear, avoidance of excessive and fluctuating loads on bearings etc, which make for minimum maintenance and running costs.

The required dynamic features for crushing fulfilled by these constructions are:

(A) That the material should continuously be gripped between components the operative surfaces of which exert on the lumps forces in three mutually normal directions with impact, while having correspondingly relative velocities in three mutually normal directions, to split, shear and break up the lumps and to separate the fragments from one another, and that the same process be continued on the fragments up to any required degree of fineness of grain. These impacts should not take place merely at one spot, but all over the surface of the lump in order to ensure hitting the weak spots for splitting.

(B) Every part of the operative surface enclosing the material should simultaneously exert the above crushing effects, and every section of the volume enclosed should be utilized for the crushing action, with no part of the volume wasted.

(C) The crushing effects should be in some order ensuring the same series of operations for all sections of the material passing through the apparatus, to provide uniformity of output.

(D) The actual crushing forces should, at least for the greater part, cancel within the system, to relieve load on bearings.

The proofs for this are not given here owing to lack of space.

Yours faithfully,

M. S. FRENKEL.

London N16.

Silicon Carbide Heaters

HEATING elements in the form of silicon carbide tubes have been developed by Siemens-Schuckert (Great Britain) Ltd.. Faraday Works, Great West Road, Brentford, Middlesex. Laboratory furnaces containing these tubes are claimed to be suitable for tests and heat treatment at very high temperatures, up to 1,500°C.

Advantages are said to include long life and simple operation. Owing to the form of the element, uniform temperature distribution is obtained.

In form these Silit tubes, as they are called, have a large diameter at the end and a thinner central heating portion. The material to be heated is placed in a refractory heating tube which is fixed in the centre of the Silit tube. The latter is surrounded by another ceramic tube and the whole fitted into a steel cylindrical body with suitable insulating material.

All the electrical equipment is housed in the bench mounting stand and includes a tapped transformer to assist accurate temperature control, tapping switch, main contactor, pilot lamp, manual power regulator and ammeter.

Boron Development

AFTER two years of research, Borax Consolidated Ltd., of London, is offering amorphous elemental boron from pilot-scale production. Two grades are available—90 to 92 per cent purity, and 95 to 97 per cent purity.

From the start, the company says, there was no difficulty in obtaining boron of 89 to 90 per cent purity, and the aim was to develop a process which would give a product of higher quality at a price which would enable industry to investigate its commercial applications.

Prices envisaged by Borax for amorphous elemental boron are: $\pounds 5$ to $\pounds 6$ per lb. for the lower grade in ton lots, and from $\pounds 6$ to $\pounds 7$ per lb. for the higher grade in ton lots.



ANNUAL REVIEW OF PHYSICAL CHEMISTRY. Vol. 6. Annual Reviews Inc., Stanford, California. Distributed by H. K. Lewis, London, 1955. Pp. ix+515, \$7.50.

This volume retains essentially the basic framework of topics that has been used in the five previous volumes, with certain exceptions. Thus the editors have decided, in view of the establishment of the Annual Review of Nuclear Science, to omit the subject of radioactivity in the present instance. Furthermore, molecular structure and crystallography have been combined in a single review, while spectroscopy has been treated under the separate headings of electronic spectroscopy and vibration-rotation spectroscopy. A topic which has not previously appeared is that of chelate compounds.

The following chapter headings indicate the scope of the present volume:-Thermochemistry and the Thermodynamic Properties of Substances (D. W. Scott); Cryogenics (F. G. Brickwedde, R. P. Hudson, and E. Ambler); Heterogeneous Equilibria and Phase Diagrams (J. F. Schairer); Solutions of Electrolytes (O. Redlich and A. C. Jones); Solutions of Nonelectrolytes (M. L. Huggins); The Solid State: Diffusion in Metals and Alloys (O. J. Kleppa); Radiation Chemistry and Hot Atom Chemistry (J. E. Willard); The Quantum Theory of Valance (R. Parr G. and F. Ellison); О. Electronic Spectroscopy (H. Sponer): Vibration-Rotation Spectroscopy (R. M. Badger); Metal Chelate Compounds (A. E. Martell); Experimental Molecular Structure and Crystallography (E. W. Hughes); Kinetics of Reactions in Solution (R. Livingstone); Kinetics of Reactions in Gases (N. Davidson and R. G. Snowden); Surface Chemistry and Contact Catalysis (G. D. Halsey); Electrode Processes and the Electrical Double Layer (D. C. Grahame); Polymerisation Kinetics and Polymer Properties (W. H. Stockmayer, F. W. Billmeyer and F. K. Beasley); Colloid Chemistry, Exclusive of High Polymers (R. D. and M. J. Vold); Isotopes (H. Craig and G. Boato); Nuclear and Paramagnetic Resonance (J. N. Shoolery and H. E. Weaver); Statistical Mechanics (I. Prigogine).

In every instance the review articles are thoroughly documented with original literature references.—H. MACKLE.

METHODEN DER ORGANISCHEN CHEMIE (Houben-Weyl). Vol. III. PHYSIKALISCHE FORSCHUNGSMETHODEN. Part 2. 4th edition. Georg Thieme Verlag, Stuttgart, 1955. Pp. xxviii + 1078. Moleskin, DM.186.

During the last two decades the armoury of the organic chemist has been extended by the inclusion of a considerable number of physical methods, without which many important advances would have been quite unthinkable. Thus the recent announcement of the elucidation of the inordinately complex structure of vitamin B12 showed the miracles that can be accomplished by collaboration between organic chemists and Xray crystallographers; the current literature contains countless examples of the ever increasing importance of spectroscopy in the characterization, quantitative analysis and structural investigation of organic compounds. It is therefore not surprising that the new edition of 'Houben' devotes more than two large tomes to the discussion of physical methods.

The second part of volume III (part I will be published shortly) deals with the application in organic chemistry of electrical, optical, magnetic and acoustic methods. It opens with a brief account of conductivity measurement, followed by substantial chapters on *p*H determination, indicator papers, potentiometric and conductometric titration, electrophoresis, redox potentials, polarography and dielectric properties.

The survey of optical methods includes chapters on refractometry, polarimetry,

light scattering, phosphorescence and fluorescence, and there are excellent brief accounts of electron diffraction, electron microscopy and X-ray methods.

In view of the importance of spectroscopy to the organic chemist, it is appropriate that the longest chapters in the book should be devoted to this topic. The section on specscopy in the uv and visible regions (172 pages) is largely devoted to a valuable, detailed survey of the relation between spectrum and constitution, while the 106 pages on infra-red methods are about equally divided between techniques of measurement and their application to chemical problems. Raman spectroscopy is discussed briefly and there is a welcome section on the little known microwave method. Spectroscopy in the microwave region, which lies between the infra-red and radio frequencies, gives useful information about the fine structure of molecules and is likely to become of increasing importance. The volume closes with short chapters on magnetochemistry and ultrasonics.

It is, of course, impossible even in a volume of this size to provide exhaustive accounts of the very wide range of topics detailed above. The aim of most of the contributors has therefore been to provide introductory surveys which expound the theoretical basis of the methods, give details of apparatus and techniques, and indicate the limitations of each method as well as the range of problems to which it can be applied. Nearly all the chapters give excellent bibliographies of standard works, beside numerous footnote references, and the book therefore provides a valuable key to the literature.

In the reviewer's opinion the present volume is easily the most useful part of the new 'Houben' which has appeared so far. It is strongly recommended to those libraries and chemists who have been deterred by financial considerations from acquiring the whole set of sixteen volumes.—J.C.P.S.

GAS KINETICS. By A. F. Trotman-Dickenson. Butterworths Scientific Publications, London; Academic Press Inc., New York. 1955. Pp. 322. 40s.

The vital problems which chemical kinetics sets out to solve are why and how chemical reactions occur and why they proceed at the speed they do. That our progress towards solution of these problems has been slow is due in part to the fact that many of the early systems chosen for investigation were far too complex to give fruitful results. The truth of the matter is that in our present state of knowledge it is only with the very simplest types of reactions that we can hope to disentangle completely the various factors involved. It is with homogeneous gas reactions of simple type that this book is solely concerned. The emphasis throughout is on fundamentals and the reactions chosen for discussion are those which appear to throw most light on the really important problems of chemical kinetics noted above.

The theoretical introduction is well done and should prove very useful to undergraduates at the Honours level. A brief discussion of the collision theory and its more obvious weaknesses is followed by a description of the transition state theory and of the theory of unimolecular reactions. The treatment is not an elementary one but the derivation of the equation is clearly explained and should be easily followed by the reader who has a fair grounding in statistical mechanics. The author then takes up the important question of energy transfer and discusses it in detail. This is probably the best part of the book. Thereafter he deals in turn with unimolecular decompositions and bimolecular combinations, transfer reactions including metathetical reactions and the disproportionations of free radicals and finally radical decompositions and additions.

Although the book is apparently intended mainly for the Honours student, it will prove a mine of information for the young research student commencing work in any branch of chemical kinetics. Very full literature references are given at the end of each chapter, the total number of references running to the large figure for a book of this size of over 800. While it should be made available to all undergraduates studying kinetics, their teachers will find it no less useful and any chemist who wishes to find out what the present position is in the field of chemical kinetics is assured of finding some interesting reading.-R. C. PINK.

Israel's Oil Well

Israel's first oil well at Heletz, Huleikat, began production on 24 November. The first gusher was struck there on 23 September at a depth of 4,905 ft.



Sunbury Expansion

Latest addition to BP's research station at Sunbury, Middlesex, is a laboratory for testing motor fuels. The station is being considerably expanded to meet the oil industry's increasing research and development demands.

Natural Gas Drilling

Sir Harold Smith, K.B.E., D.L., M.I.C.E., M.Inst.Gas E., chairman of the Gas Council, announced recently in London that drilling for natural gas would begin in the next few weeks in Yorkshire.

Food Group Dinner-Dance

The fifth dinner-dance of the Food Group of the Society of Chemical Industry will be held at the May Fair Hotel, London, on 27 January. Tickets, price £2 each, are obtainable from Mr. R. Harold Morgan, M.Sc., Croft House Laboratory, 45 Dollis Avenue, Finchley N2.

Nuclear Enterprises Ltd.

An application by a Canadian firm, Nuclear Enterprises Ltd., of Winnipeg, to lease a site at Sighthill Industrial Estate, Edinburgh, for the construction of a manufacturing plant, has been approved by the local town council. The firm's main products are the scintillometer, an instrument used extensively in geophysical surveys, and plastic and liquid phosphors used in nuclear research and instrument construction.

Chemical Plant Inquiry

Plans for the erection of a new chemical plant on a 50 acre site at Dan's Road, Widny, may have to go before a public inquiry following objections which have been lodged with Lancashire County Council.

Laboratory Apparatus on Show

An exhibition of laboratory apparatus and techniques with particular reference to instrumentation will be held in the Manchester College of Technology (Rooms E1, E17 and E19) on Wednesday, 4 January (2 to 8 p.m.) and Thursday, 5 January (10 a.m. to 8 p.m.) The exhibition, organized by the Manchester & District Section of the Royal Institute of Chemistry, will be given by some 35 exhibitors and will include various demonstrations, such as glass-blowing and vapour phase chromatography.

THE CHEMICAL AGE Subscriptions

With effect from 7 January 1956, the annual subscription rate (home and overseas) for THE CHEMICAL AGE will be increased to 52s 6d. The price of single copies will be increased to 1s 3d, by post 1s 6d.

Cooling Tower Service

Head Wrightson Processes Ltd., who have manufactured mechanical cooling towers for many years, announce that they now offer a complete inspection, maintenance, and repair service for all water cooling towers regardless of the make or model.

Board of Trade Announcement

In 1952, 1953 and 1954 the Board of Trade announced that it had received applications for increased protective duties on lead capsules and polyvinyl chloride sheeting of certain thicknesses. In a current bulletin the Board of Trade announces that the Government has decided that there is no case for increased protection for these goods.

Research Expenditure

The British Government and home industry between them were probably spending about £300,000,000 a year on research, Dr. B. K. Blount, M.A., B.Sc., D.Phil., F.R.I.C., deputy secretary of the DSIR, said when he gave the E. Frankland Armstrong memorial lecture on 'Research in Industry' to the Royal Society of Arts on 23 November. The US was spending five times as much, he added.

Original Papers

The Chemical Society will hold a meeting for the reading of original papers at Burlington House, Piccadilly, London W1, on 15 December at 7.30 p.m. The papers which will be presented are: 'De-localised Metalcarbon Bonds' by Dr. L. E. Orgel, M.A., D.Phil.; 'Complexes of Uncharged Aromatic Systems with Transition Metals & the Bonding Problem of Ferrocene & Similar Compounds' by Dr. E. O. Fischer (Techn. Hochschule, München); and 'Ferrocene Derivatives, Part Three, Ferrocene Dicarbonyl & Related Compounds' by B. F. Hallam & P. L. Pauson, B.Sc. Abstracts of the papers are obtainable from the General Secretary of The Chemical Society.



Basic Chemicals Factory

Reichhold Chemicals of Canada has acquired a 112-acre site at Millhaven, Ontario, for the construction of a factory to produce basic chemicals. Reichhold officials consider that Millhaven is certain to become an important chemical centre. Nearby is the Canadian Industries' recently completed \$20,000,000 Terylene plant.

Kuwait Gil Production

By the end of October, the 1955 production of crude oil at Kuwait (where BP has a 50 per cent interest) had reached a total of 45,162,457 tons. This compares with 38,543,619 tons for the January-October period last year.

Libyan Concession

On 29 November the Texas Gulf Oil Co. announced that it had received oil concessions covering about 5,000,000 acres in Libya, North Africa. Under terms of the agreement the company will pay the Libyan Government a $12\frac{1}{2}$ per cent royalty on all oil and gas produced up to 15,000 barrels a day, and 50 per cent of the net profits resulting from production exceeding 15,000 barrels daily.

Belgian Nylon Plant

The first Belgian nylon factory has been opened at Zwijnaerde, near Ghent, by the Belgian rayon manufacturing group Fabelta. The factory will make nylon yarn under American patents, the Belgian rights of which have been purchased from the French holder of the European concession. The Belgian nylon yarn is to compete inside the Benelux frontiers with the Dutch product Enkalon, made under German licence by the use of the Perlon patents.

Mexican Ammonia Plant

An ammonia factory is to be built in Mexico at a cost of \$12,000,000. The Mexican Government has authorized construction by a Franco-Mexican finance group, comprising the Nacional Financiera and the Banque Nationale pour le Commerce et l'Industrie and Compagnie Saint-Gobin. Technical equipment will be provided by France and the plant will utilize the gas and coke produced by the Altos Hernos Co. and the Demenclova Blast Furnaces in N. Mexico.

Chrome in Zululand

Professor Jeppe of Witwatersrand University says that the chrome deposit recently found near Eshowe, Zululand, may become the richest mine in the world. The ore so far tested averaged 44 per cent chrome. An American company is reported to have signed a contract for £600,000 worth of the chrome ore yearly.

Spanish Sulphuric Acid

Union Española de Explosives is to extend its San Jerónimo factory at Seville for the manufacture of sulphuric acid and superphosphate. Planned capacity is 105 tons of sulphuric acid a day and 120,000 tons of superphosphate a year. Two furnaces in the sulphuric acid plant will have a daily capacity of 50 tons of pyrites each. The superphosphate plant will consist of two groups for the grinding of phosphates. with a joint capacity of 16 tons an hour.

Pyrethrum Research Laboratory

A. new £25,000 laboratory, recently opened at Nakuru by the Governor of Kenya, Sir Evelyn Baring, is about to start work on the pyrethrum plant. The laboratory is the only one in the world devoted solely to pyrethrum research.

Safflower Seed Programme

A programme to determine the commercial value of safflower has been arranged by the plant industry division of the Australian Commonwealth Scientific and Industrial Research Organisation in conjunction with the Queensland Department As a result of previous of Agriculture. Queensland trials, safflower promises to develop into a more important crop than Yields have been twice as great linseed. as those from linseed under similar conditions. Safflower is said to have certain advantages over linseed oil. It is easily extracted, requires no complicated process in refinement, remains stable in storage and does not sludge. The oil has a high iodine count which makes it a good drying oil. and it is particularly suitable for the new plastics paints and in the manufacture of certain synthetic resins for industry.



PROFESSOR SIR CYRIL HINSHELWOOD, M.A., D.Sc., F.R.S., Dr. Lee's Professor of Chemistry in the University of Oxford, was elected president of the Royal Society in succession to LORD ADRIAN, O.M., at the anniversary meeting of the society at Burlington House, London, on 30 November. Sir Hinshelwood Cvril has been foreign secretary of the Royal Society since 1950 and was, until recently, chairman of the Fuel Research Council of DSIR. DR. H. G. THORNTON, head of the department of soil microbiology at Rothamsted Experimental Station, was elected foreign secretary in succession to Sir Cyril Hinshelwood. Other members of the council elected included SIR CLAUDE GIBB, C.B.E., chairman and managing director, C. A. Parsons & Co. Ltd.; SIR RUDOLPH PETERS, M.C., professor emeritus of biochemistry, University of Glasgow; DR. B. F. J. SCHONLAND, C.B.E., deputy director, Atomic Energy Research Establishment; and PROFESSOR M. STACEY, professor of chemistry in charge of organic and biological chemistry, University of Birmingham.

DR. W. C. DAVEY, M.Sc., Ph.D., F.I.R.I., manager of the chemical research division at the Dunlop Research Centre, Birmingham, has been appointed manager of the compounding research division in addition to his present duties.

MR. G. R. BLAKELY, B.Sc. (Eng.), M.I.C.E., M.I.Mech.E., M.I.Prod.E., has been appointed to the board of directors of C^A. Harvey & Co. (London) Ltd., as works firector. Mr. Blakely joined the company in 1952 as general works manager of the Greenwich, London, Metal Works.

MR. M. D. EDINGTON, general manager of the carbonization department of Durham Coal Board, has been appointed carbonization director for the board. Mr. Edington is a vice-president of the Coke-Oven Managers' Association.

After 35 years with the Burmah Oil Co., MR. W. E. ABRAHAM has retired, but will continue to serve the company in a consultative capacity. Mr. Abraham joined the Burmah Oil Co. as a geologist in 1920, and

in 1936 was appointed senior geologist in Burma. During the last war he served with the rank of Major-General, and soon after his demobilization was appointed to the board of the Burmah Oil Co., becoming joint-managing director in 1948.

MR. J. O. HITCHCOCK, B.Sc., F.R.Ae.S., F.I.M., sales director of The Mond Nickel Co. Ltd., was elected a member of the Institution of Mining and Metallurgy on 10 November.

DR. ROBERT T. ARMSTRONG, Ph.D., has been elected vice-president-technical director of Celanese Corporation of America. Dr. Armstrong has been associated with Celanese in various technical capacities since 1946. He was named director of technical control in 1951 and, in 1952, associate director of the central research laboratories at Summit, New Jersey. He became technical director of the textile division in 1953 and, in that capacity, co-ordinated the research and development activities which led to the production of the company's new triacetate fibre. Arnel. He holds several patents in the fields of rubber, chemicals, synthetic polymers and man-made fibres.

At a banquet following the annual meeting of the American Institute of Chemical Engineers in the Hotel Statler, Detroit, on 29 November, PROFESSOR E. PIRET, Canadian-born professor of chemical engineering at the University of Minnesota was presented with the William H. Walker Award in recognition of his outstanding papers on chemical engineering progress published by the AICE. Professor Piret, a specialist in the fields of staged reactors and the crushing of solid substances, holds the Palmes Academiques of France for pioneer work in initiating courses in chemical engineering at Nancy. In 1954 he was awarded the Bronze Medal of the Swedish Association of Engineers. Born in Winnipeg, Professor Piret took his B.Sc. degree in chemical engineering at the University of Minnesota in 1932, and in 1936 obtained a doctorate in biological chemistry and bacteriology at the University of Lyon, France. He returned Minnesota where he took his Ph.D. to

degree in 1937. A consultant to the US Naval Research Laboratory, Professor Piret also acts as consultant to the Minnesota Mining & Manufacturing Co. and other firms.

Obituary

The death has occurred of MR. JOHN CROMBIE, Woodmarch, Larchfield Avenue, Newton Mearns, Renfrewshire, who was a former chairman of the British Watercolours Association and a past president of the Oil and Colour Chemists' Association. He was managing director of James A. Morrice (James Anderson & Co. Ltd.), and a director of James Anderson (Colours) Ltd. He was 66.

The death has taken place at the age of 80 of MR. JOHN RITCHIE, of Redholme, Blairbeth Road, Burnside, Lanarkshire, former managing director of J. & J. White, now British Chrome & Chemicals Ltd, Shawfield Chemical Works, Rutherglen.

After a short illness, MR. FRANCIS HERON ROGERS, F.C.I.P.A., M.I.Mech.E., M.I.Chem.E., a director of Stream-Line Filters Ltd., of London, died on 27 November in the Mayday Hospital, West Croydon, Surrey. A past president of the Institution of Chemical Engineers, Mr. Heron Rogers became a director of Stream-Line Filters Ltd. in 1936 when the company was converted from a private to a public company. Mr. Heron Rogers was 79.

DR. FRANCIS HEREWARD BURSTALL, M.Sc., D.Sc., F.R.I.C., senior principal scientific officer, Chemical Research Laboratory, DSIR. Teddington, Middlesex, died on 2 December at the age of 52.

MR. R. W. E. STICKINGS, O.B.E., B.Sc., F.R.I.C., deputy managing director of May & Baker Ltd., died suddenly at Brentwood District Hospital, Essex, on 3 December. Mr. Stickings was also chairman of M & B Plastics Ltd., a director of Pharmaceutical Specialities (May & Baker) Ltd., and was on the boards of the M & B associated houses in Canada and India. He joined May & Baker Ltd. in 1919 as a research chemist when the M & B laboratories were in Wandsworth, eventually becoming technical manager of the factories at Battersea and Wandsworth. On the company's move to Dagenham in 1934, he continued as works manager and was appointed a director in 1939.

MAJOR KENNETH GORDON, C.B.E., M.C., M.A., F.Inst.P., the Director-General of Ordnance Factories under the Ministry of Supply since 1952, who died in Charing Cross Hospital, London, on 29 November. aged 58, was at one time a director and joint-managing director of ICI's Billingham Major Gordon was appointed division. Director-General of Ordnance Factories in June 1952, an appointment he was enabled to take up through the generosity of Head Wrightson Processes Ltd. who released him from his position of deputy managing director of the company, one year after his leaving Trinidad Leaseholds to take up the appointment. After service in the 1914-18 War, Major Gordon studied at Oxford and, following a brief period as a university lecturer, joined Synthetic Ammonia & Nitrates in 1923. This company later became the Billingham division of ICI. At Billingham in 1933 Major Gordon was responsible for the coal hydrogenation plant which was to produce high octane spirit for the RAF in the last war. In 1946-47 he was responsible for the petroleum chemical plant at Wilton and the extensions to Billingham.

Celebration Dinner

ONE of the most representative chemical trade gatherings to be held in the North of England will take place on Monday, 12 December at the Midland Hotel in Manchester where a dinner will be given in honour of Mr. William Collison to celebrate 70 years' service to the chemical industry.

Mr. Collison, who is the managing director of William Blythe & Co. Ltd., and Jorg Riley & Sons Ltd., joined William Blythe as a junior in 1885. In December 1918 he was appointed a director, and in December the following year he was made managing director. Aged 83, Mr. Collison is still active and is the chairman of several associations.

At the dinner in his honour, Sir Alexander Fleck, D.Sc., LL.D., F.R.I.C., the chairman of ICI, will be in the chair supported by Mr. L. P. O'Brien, F.C.I.S., of Laporte Chemicals Ltd., and the organizing committee—Mr. T. C. Fawcett, Mr. G. K. Hampshire, M.A., F.R.I.C., and Mr. K. G. Holden—and more than 100 guests.

New Wool Scouring Method

A new method of de-greasing wool by dry cleaning with white spirit instead of scouring with soap solutions was demonstrated to delegates to the International Wool Textile Research Conference in Melbourne recently. The inventor, Mr. J. F. Sinclair, pointed out that the process was quicker, more efficient, and no more costly than normal scouring methods. The main advantage of the new process was that the wool fibres were not entangled to anything like the same degree as with normal scouring. This fibre entanglement made wool sorting and reclassing difficult and costly. The new process also gave a higher proportion of tops, and the yarn was of better colour. Other advantages were the smallness of the plant and elimination of the effluent problem. Valuable fleece byproducts could also be easily recovered. 'The cost of a complete plant,' said Mr. Sinclair, ' is about the same as the usual scouring set.'





FLOTATION CHEMICALS

Write Dept. B/21 for further details



Commercial Intelligence

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

Mortgages & Charges

(Note.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described herein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every company shall, in making its Annual Summary, specify the total amount of debt due from the company in respect of all Mortgages or Charges. The following Mortgages or Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an *—followed by the date of the Summary but such total may have been reduced.)

CHURCH PAINT & VARNISH CO. LTD. (Lancs).—8 November, charge, to Midland Bank Ltd. securing all moneys due or to become due to the bank; general charge. *Nil. 16 May, 1955.

CONSTRUCTEX LTD., London EC., dealers in cement, lime etc.—2 November, £5,000 further charge, to Credit for Industry Ltd.; charged on land and factory at Oyster Lane, Byfleet, comprised in mortgage dated 4 December, 1950 etc. *£9,575. 16 March, 1954.

Satisfactions

BRITISH DIESEL OIL & PETROL CO. LTD. (formerly L.T.C. Distillates Ltd.), Bolsover.— Satisfaction, 11 November, of debenture registered 4 April, 1938, to the extent of £6,000.

GERARD BROTHERS LTD., Nottingham, soap manufacturers.—Satisfactions, 12 November, of debentures registered 14 December, 1920 and 24 September, 1925.

LONGWORTH, ENTWHISTLE LTD., Manchester, germicide manufacturers.—Satisfaction, 11 November, of debenture registered 13 May, 1950.

Company News Celanese Corp. of America

Net income of Celanese Corp. of America for the first nine months of 1955, after all charges and taxes, amounted to \$8,695,105; equivalent to 88 cents a common share after preferred stock dividends. This compared with a net income of \$3,940,456 in the first nine months of 1954 which, after provision for preferred stock dividends, resulted in earnings of seven cents per share of common stock. Net income for the three months' period ended 30 September, 1955, after charges and taxes, amounted to \$1,473,218: equal to five cents a share on the common stock after preferred stock For the first nine months of provisions. 1955, net sales amounted to \$132,358,391. compared with \$105,361,648 in the same period of 1954. Net sales for the September quarter of this year totalled \$39,110,129. In his report to stockholders, Mr. Harold Blancke, president of the Corporation, said that facilities for the company's new industrial fibre, Fortisan-36, have been brought into production and the fibre will soon be marketed. Mr. Blancke also announced the commercial production of Forticel, a new cellulose propionate plastic material.

Esperanza Copper & Sulphur Co. Ltd.

At the recent annual general meeting of the company in London it was announced that subject to Treasury consent arrangements had been made for the issue of £200,000 51 per cent convertible debenture stock 1960/65 to be subscribed at par by Lazard Brothers & Co. Ltd., the British Metal Corp, Ltd., Robert Fleming & Co. Ltd., and the London Tin Corp. Ltd. This stock will be convertible at any time at the end of 1960 into five ordinary shares of 2s 6d each per £1 per nominal of the stock. making a conversion price of 4s per share, and except by consent of the trustees, the company will make no issue of share capital. The stock will be secured by a specific charge on the shares of Cyprus Sulphur & Copper Co. Ltd., a wholly-owned subsidiary. In his statement circulated to stockholders, Mr. A. Hedley Williams. A.M.I.M.M., chairman M.Inst.Pet. and managing director, said :- As foreshadowed, the trading results of the subsidiary company for the year ended 31 March 1955. were poor, but the Limni mill had operated with increasing efficiency. The estimated net mine operating profit for the six months from 31 March 1955, to 30 September, 1955, exceeded £110,000, and had increased recoveries of sulphur, gold, silver and copper, all valuable constituents of Limni be anticipated. ore, could Since 31 March 1955, 17,000 tons of pyrites concentrates had been produced, and by 31 December 1955, it was expected that shipments of Kinousa cupreous pyrites to Germany would total 45,000 tons. In the current financial year to 30 September 1955, shipments of Kinousa cupreous pyrites to Germany totalled 40,430 tons, compared with 18,670 tons in the same period last year.



The addition of small amounts of Sequestrol (ethylene diamine tetraacetic acid Geigy) to both acid and alkaline cleaners for metals, whether simple immersion or electrolytic in type, often much improves their action by solubilising the more intractable surface films of oxide, carbonate etc. Cleaners formulated from concentrated Sequestrol solutions are valuable for specialised applications, for example, alkaline electrolytic de-rusting, cleaning of brazed assemblies, removal of welding flux, etc. Particulars on request.

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Next Week's Events

MONDAY 12 DECEMBER

Incorporated Plant Engineers

'Tar Dundee: Mathers Hotel, 7.30 p.m. Distillation Plant' by R. M. Crighton (William Briggs Ltd.).

TUESDAY 13 DECEMBER

Institute of Physics

London: 47 Belgrave Square SW1, 5.30 p.m. 'The Effects of Radiation Damage on the Properties of Solids' by Dr. E. W. J. Mitchell, A.Inst.P.

Incorporated Plant Engineers

Manchester: Engineers' Club, Albert Square, 7.15 p.m. 'Corrosion Technology'. The Mond Nickel Co. Ltd.

WEDNESDAY 14 DECEMBER

Society of Instrument Technology Chester: 5 King's Buildings, King Street, 7 p.m. 'Control Valves, Design & Operating Characteristics' by B. W. Walls, B.Sc., A.M.I.Chem.E.

Newcastle: King's College, 7 p.m. ' Pneutronics in Industry' by J. E. Fielden (Fielden Electronics Ltd.).

Institution of Mechanical Engineers

London: Institution of Civil Engineers. Great George Street SW1, 5.30 p.m. British 'The Pres-Nuclear Energy Conference. surized Water Reactor as a Source of Heat for Steam Power Plants' by J. M. Kay, M.A., Ph.D., A.M.I.Mech.E., & F. J. Hutchinson, M.(Eng.), M.I.Mech.E.

Incorporated Plant Engineers

Nottingham: County Hotel, Theatre Square, 7 p.m. 'Grit & Dust Collection' J. E. O'Breen, F.Inst.F., M.I.Mech.E.

SCI (Food Group)

London: Rooms of the Linnean Society, Burlington House, Piccadilly W1, 6.30 p.m. 'Nutrition Requirements & Food Fortification' by H. M. Sinclair, M.A., D.M., B.Ch., B.Sc.; Miss A. M. Copping, M.Sc.; & J. B. Coppock, B.Sc., Ph.D., F.R.I.C.

Society for Analytical Chemistry

Edinburgh : George Hotel, George Street, 7.15 p.m. 'Statistics for Chemists' by B. Woolf, M.A., Ph.D., F.R.S.E.

SCI (Corrosion Group)

London: The Chemical Society, Burlington House, Piccadilly W1, 6.30 p.m. 'Chromate Pigments for Metal Protection ' by H. G. Cole, B.Sc., A.R.I.C.

THURSDAY 15 DECEMBER

SCI (Road & Buildings Group)

London: Lecture Hall, Junior Institution of Engineers, Pepys's House, 14 Rochester Row SW1, 2.30 & 6 p.m. Meeting for the reading of papers on 'New & Revised Test Methods.

The Chemical Society

London: Rooms of The Chemical Society, Burlington House, Piccadilly W1, 7.30 p.m. Reading of original papers: 'De-Localised Metal-Carbon Bonds' by Dr. L. E. Orgel. M.A., D.Phil.; 'Complexes of Uncharged Aromatic Systems with Transition Metals & the Bonding Problem of Ferrocene & Similar Compounds' by E. O. Fischer (Tech. Hochschule, München); & 'Ferrocene Derivatives, Part Three. Ferrocene Dicarbonyl & Related Compounds' by B. F. Hallam & P. L. Pauson, B.Sc.

Society for Analytical Chemistry

Nottingham: Technical College, 7 p.m. Recent Advances in Inorganic Analysis' by Dr. R. Belcher, Ph.D., F.R.I.C., F.Inst.F.

Oil & Colour Chemists' Association

London: Royal Society of Tropical Medicine & Hygiene, Manson House, 26 Portland Place W1, 7 p.m. 'Penetration of Ink into Paper & Its Relevance to Print Quality' by R. R. Cupe, B.Sc., & A. H. Smith, B.Sc.

FRIDAY 16 DECEMBER

Institute of Metal Finishing

Sheffield: Grand Hotel, 6.30 p.m. 'Selection of Protective Paints for Use in the Electroplating Industry' by H. A. Slac'-B.Sc., A.R.I.C.

Institute of Physics

London: 47 Belgrave Square SW1, 6.30 p.m. The Non-Destructive Testing of Non-Metals' by Dr. H. Kolsky, B.Sc., Ph.D., F.Inst.P. (ICI Akers Research Laboratories).

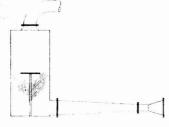
SATURDAY 17 DECEMBER

Institution of Chemical Engineers

Manchester: Reynolds Hall, College of Technology, 3 p.m. 'Some Aspects of Heat Transfer in a Climbing Film Evaporator ' by Professor J. M. Coulson, M.A., Ph.D., M.I.Chem.E.

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More Hypalon for England

LARGER quantities of the new synthetic rubber compound Hypalon (already launched in England by Durham Raw Materials Ltd., of 1-4 Great Tower Street, London EC3) will shortly become available for the English market. This is the result of plans announced by its American producers, Du Pont de Nemours & Co. Inc., who are to build a new factory at Beaumont, Texas, for the full scale production of Hypalon.

Hypalon is a synthetic rubber compound which can be brushed or sprayed on to any material that is likely to suffer from atmospheric or chemical corrosion. It forms a protective coating which, it is claimed, remains intact and flexible at high and low temperatures and provides complete protection against ozone, sunlight, bad weather and many chemicals. It is said to adhere to natural rubber, synthetic fibres, wood, stone and metal and shows unusually good resistance to scuffing and abrasion.

Hypalon coating is primarily intended for industrial equipment such as conveyor belts, cables and hoses, but the wide range of colours available enables it to be used also on floor tiles, footwear, upholstery fabrics and many other household goods.

US Potash Industry

PRODUCTION of marketable potassium salts in the US last year increased by two per cent over that of 1953, reports the Bureau of Mines in a mineral market report just released. Not only did production increase, but the sales and consumption of potassium salts also rose, being 10 and eight per cent, respectively, higher than in 1953.

Exports of US potash materials in 1954 increased by 33 per cent, while imports fell by 10 per cent. Chief suppliers of potash to the US last year were West and East Germany, France, Spain, and Chile, in that order. By the end of 1954 stocks of potassium salts in hand totalled 524,328 short tons with a K_2O content of 309,732 tons.

The report combines both grades of potash and manure salts to avoid disclosing company operations, and lists only one company as producing 48-50 per cent muriate. In 1954 world production of potassium salts increased by nine per cent compared with 1953, with Israel, West Germany, France and Spain accounting for the major part of the increase.

Market Reports

LONDON.—A good demand for industrial chemicals has been reported from pretty well all sections of the market, and there has been a steady flow of orders on export account. The price trend remains firm and with few exceptions rates are unchanged. The non-ferrous metal compounds have fluctuated during the past two weeks, the latest basis prices (2 December) being white lead £144 per ton, red lead £138 10s per ton and litharge £140 10s per ton. The call for fertilizers is not strong at present and no improvement can be expected until the new year. Pitch, the tar acids and creosote oil are active items in a firm coal tar products market

MANCHESTER.-Although the demand for heavy chemicals on the Manchester market during the past week has continued on fairly steady lines, reports have been current of signs of a slackening of buying interest in some quarters due to the approaching yearend stocktaking operations. On the whole, however, considering the time of the year trading conditions both on home and export accounts may be regarded as satisfactory. Prices are maintained pretty well throughout the range. In the fertilizer section there is a brisk demand for basic slag and mixers are taking steady deliveries of sulphate of ammonia, superphosphates and potash, but otherwise business is moderate. A steady movement of most of the tar products continues.

GLASGOW.—The Scottish heavy chemical market has been fairly steady during the past week. Demands have been nominal and some forward bookings have been placed, although the bulk of business is mostly for prompt delivery against immediate require of ments. Little or no change in prices has^{D'} taken place, and a good volume of inquiries is still being received for export showing favourable prospects.

New Plastic Paint

A new plastic paint, Vimacol, which is to be produced in Israel, was recently demonstrated in the plastics and textile factory of the Mayer Investment Co, at Petah Tikvah. Commercial production of the paint had been delayed while the company carried out research and experiments to find a locally produced substitute for polyvinyl acetate, the main ingredient. BDH

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A SMALL Engineering Firm in the Merseyside area has established a Centrifugal Pump Department, dealing mainly with the Chemical Industry. An ENGINEER is required to take complete charge and develop this section, a knowledge of the Chemical Industry, pump design and operation is essential. Applicants should give full details of experience and qualifications, etc., to BOX No. C.A. 3443, THE CHEMICAL AGE, 154, FLEET STREET, LONDON, E.C.4.

ATOMIC POWER

CHEMISTS wishing to work in this new field are invited to apply to 'ENGLISH ELECTRIC,' who have senior and junior vacancies at their Whetstone establishment. Write for further Whetstone establishment. Write for further information or submit applications to Dept. C.P.S., 336-37, Strand, W.C.2, quoting Ref. 1367E.

CHEMIST or **PHYSICIST** required by **MINISTRY** OF SUPPLY Experimental Establishment, Christchurch, Hants, to plan and carry out tests on properties of soils Hants, to plan and carry out tests on properties of soils in connection with their use for roads and airfields and for investigation of soil stabilisation techniques, etc. Appointment as **EXPERIMENTAL OFFICER** or **ASSISTANT EXPERIMENTAL OFFICER** Qualifica-tions: H.S.C. (Science) or equivalent, but degree or H.N.C. in Chemistry or Physics may be an advantage. Salaries within range : E.O. (Min. age 26) - £745-£920 ; A.E.O. - £306 10s. (age 18) to £670. Equal pay scheme. Application forms from M.L.N.S., Technical & Scientific Register (K), 26, King Street, London, S.W.1, quoting A442/5A/EE.

PHILIPS RESEARCH LABORATORIES have a vacancy for an ASSISTANT

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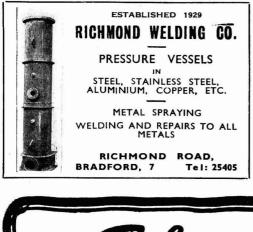
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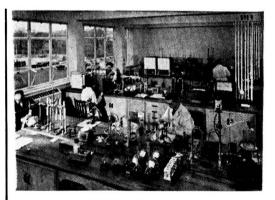
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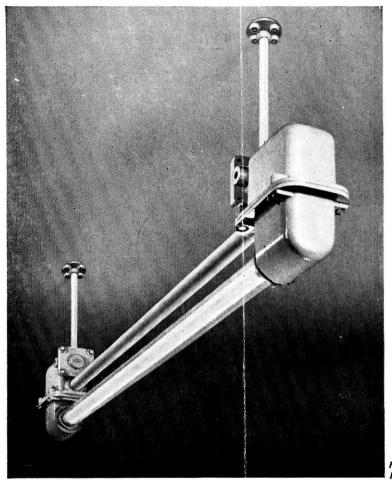
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