

# The Chemical Age

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## The Chemical Industry in 1934

**T**HE year 1934 has signalled a definite improvement in the industrial world, and the tacit satisfaction with the work of the National Government expressed in our Annual Review Number a year ago has been justified by the events of the past twelve months. Even in December, 1933, trade returns were inspiring confidence. It was felt that we were emerging from the trough of the depression, that brighter days were ahead. While there is no justification for pessimism, the wave of economic nationalism throughout the world, coupled with our experience of tariffs and quotas, still leaves something to be desired. Great Britain's export trade may not be much better than that of other countries, but the overseas chemical trade—total exports for eleven months to November 30, amounting to £18,060,545, as contrasted with £17,087,002 for the corresponding eleven months of 1933, an increase of 5 per cent.—is encouraging. We cannot, however, shut our eyes to the fact that in the same period the total imports of chemicals have gone up from £9,066,451 to £10,400,951, an increase of 14 per cent. Progress along the road to economic self-sufficiency can afford little self-satisfaction when it is realised that the maintenance of world prosperity is essential to this country as an exporting nation, anxious to exploit the maritime routes of the world. In other pages the recognised leaders in a dozen different spheres of chemical science and industry review the trend of events in 1934. There is a marked consensus of opinion that the year has been one of consolidation rather than sensational advance, and that is, perhaps, one of the best omens for the future.

Mr. P. Parrish again reviews the prevailing conditions in the heavy chemical industry, and comments upon the close association of the chemical industry with the study of the use of fuel. Certain it is that no one is better equipped for undertaking the hydrogenation of coal than those who have had experience of high-pressure technique, such as the synthetic ammonia industry affords. Nothing is more suitable for hydrogenation than vertical retort or low-temperature tars and creosote, and Mr. Parrish says it is a pity that in the interests of national security the gas and coking industries did not originally interest themselves in synthetic ammonia production. Fear is expressed by Mr. A. J. Hall in a review of textile bleaching, dyeing and finishing developments that the point has been reached at which price-cutting, carried too far, is retarding progress. Although methods of colouring and finishing textile

materials are constantly being improved, the margins of profit allowed to the dyer and finisher are being decreased, and many firms are now finding it difficult to make a profit. The plant available is capable of dealing with much more textile material than is at present in demand, and there is, therefore, keen competition—with price-cutting—for the work that is to be had.

The amount of work in the nitrogen field has been far less than during the development period, owing to the abnormal position, especially in the synthetic ammonia industry, in which the capacity of existing plants is greatly in excess of the present demand for nitrogenous fertilisers. The German fixed nitrogen plants are reported to be working at only 35 per cent. capacity, and the erection of new plants in the German nitrogen cartel has been prohibited for the next five years. In Great Britain larger sales of nitrogenous fertilisers to home consumers are reported, but export sales have declined. The synthetic fertiliser industry generally has shared with other great industrial ventures the difficulties of recent times and its progress in the past twelve months must therefore be measured by relative standards and not only by tonnage output. The industry has continued to demonstrate its inherent security and strength.

The solvents industry would appear from superficial observation to be in a state of quiescence so far as new developments are concerned; indeed, there has been a tendency to revert to the well-tried solvents and plasticisers and to curtail adventures with new materials whose properties are not well known and the use of which has occasionally led to disaster. There has been a large number of new patents dealing with unusual substances as plasticisers. The value of many of them is problematical, and several appear to be blocking patents of no technical significance. Many of the suggestions put forward are obvious variations of the many possible esters which can be obtained and the inventive capacity exhibited has not been of a high order. Long-range research on methods of production of the more widely used solvents has been the feature of the year's activities, and Dr. T. H. Durrans shows in his review of the industry that the realisation of some of these ambitions may profoundly affect the situation and cause a shift in the area of production which may be detrimental to this country.

No striking novelty has been noted in the realm of fatty oils and soaps, technical movements during 1934

having been directed more to coping with economic pressure of various kinds. Professor T. P. Hilditch tells us that in connection with the manufacture of soap the progress in the production of highly efficient detergents of the so-called sulphonated type must be regarded seriously, even if it is unlikely on the ground of production costs that these will be active competitors with ordinary soaps for some time to come. There is a continued increase in the demand for preparations of the flake or powder types as distinct from the older bar soap. Some of the most conspicuous advances during the year have been concerned with the prevention of spoilage of fats and soaps during storage, while the outstanding achievement in the pure chemistry of fats has been the first complete synthesis of oleic and elaidic acids by Noller and Bannerot. In methods of technical analysis, discussion has continued with reference to the comparative merits of different procedure for the determination of iodine values and of the acetyl values of fats.

"A year of improving trade." That is the epitome of 1934 so far as plant is concerned. Work is going in plenty. Inquiries are multitudinous. More than one order has been lost through the sheer impossibility of making estimates in time. If any considerable proportion of the work for which estimates are being got out really matures, the plant manufacturers are in for a boom. The manufacturers of steam boilers are working with a satisfactory volume of orders; that fact has been suggested as indicative of the better spirit abroad. If boiler users think a trade improvement is likely to be temporary, they push their old Lancashires a little harder. If they think it is to be permanent, they add to their boiler capacity. It is virtually impossible to give any precise account of the activities of the chemical plant manufacturer's work during the past year. That is not a confession of ignorance; it is a challenge to the sales methods of our engineering firms. On previous occasions we have wagged a denunciatory finger at the chemical manufacturer who still maintains the Victorian atmosphere of secrecy concerning his doings. The manufacturer of chemical plant has far less cause for silence about his activities than has the manufacturers of chemicals.

The British chemical plant manufacturer in his working of metals is unsurpassed. Very seldom indeed is it necessary to go abroad for plant. Whenever there is difficulty in finding a British maker competent to undertake manufacture, a line to the British Chemical Plant Manufacturers' Association will in most instances solve the difficulty. When foreign and British plant is compared it is not infrequently found that the British plant is a little more expensive. It is of extreme importance that mere figures in £ s. d. should not be made the criterion of value; even if value and price are apparently coincident there is the further objection to foreign plant that the foreigner is a long way away and that it is much more difficult to get compensation for unsatisfactory goods from a foreign firm than from an English firm. Price must not be the criterion of value; that motto should be prominently displayed on every buyer's desk. Perhaps the unskilled buyer is the greatest bugbear in industry to-day. An example which has recently come under our notice may suffice to explain this point. A firm specialising in the erection of plant,

making nothing, but designing and erecting plant bought outside, has in charge of the buying a man who for many years has been with the concern in a clerical capacity and has never seen an engineering works from the inside. This man's idea of buying is to start a "Dutch auction" for each item, whether worth a few shillings or thousands of pounds. Having reduced prices to the bare minimum, the lowest tenderer is then told that someone is below him, but that a further reduction will get him the order. Too often in these days the manufacturer bites and the result is a contract that leaves no profit for the maker if he is conscientious, or, alternatively, that leaves the ultimate purchaser with an article not so reliable as that for which he originally paid the main contractors. This buyer is described by his employers as "keen"—instead of "stupid." A contract should never be settled on price, unless quality of workmanship, materials and design, and all those imponderabilia that constitute value have been taken into account. That is one of the major lessons of 1934.

Another important feature is the necessity for more specialisation among the manufacturers of chemical plant. Many firms can make a good article when someone has designed it for them and has done all the donkey-work of development. Few have specialised in a particular plant so that they know it inside-out and can talk about it to the chemical manufacturer with the weight that comes along from superior experience. The chemical manufacturer himself is partly responsible for this since he often does not invite the maker to see his plant again once it has entered the chemical works. Even so, however, the major fault lies with the plant maker, who, in most instances, tries to tackle any and every job without a skilled chemical engineer upon his staff. How that specialisation is to be brought about it is not our duty to suggest. It has been achieved in other closely allied industries, usually as the result of patented processes, but much chemical plant is semi-standard and is not covered by patents. A possible way out would be the provision of a central chemical engineering consulting body, which would handle all projects, would design and discuss with clients and would then put out the work of manufacture to members of the Plant Manufacturers' Association.

Socially, the chemical industry has well maintained its activities during the past twelve months. We have been amazed lately, in revising the particulars of chemical and allied organisations for the 1935 edition of *THE CHEMICAL AGE Year Book*, to notice the large number of new societies that have come into being in the last twelve or eighteen months. Although some of the older societies show a slight falling off in membership, and others report that they have had great difficulty in maintaining their strength, there has been a definite consolidation of effort, and our pages have borne testimony to the vast amount of thought and enterprise that has been applied to the presentation of papers on every conceivable aspect of chemical research and practice. So far as the personal welfare of the individual is concerned, we need only mention the good work done by the Institute of Chemistry and by the unemployment department of the British Association of Chemists, to indicate how thoroughly the organisations carry out their functions.



# Conditions in the Heavy Chemical Industry in 1934

**R**ECORD should be made of the death of Professor Fritz Haber, which occurred in Bâle in February. To Haber, in collaboration with Le Rossignol, was due the first direct synthesis of ammonia. After many disappointments, they succeeded in constructing an apparatus which, by the use of high temperature and pressure, and the selection of osmium and uranium as catalysts, achieved the desired end. The Badische Anilin- und Sodafabrik became interested, and following a demonstration of the process (at first the apparatus refused to work), the company took it over, and it was gradually translated from the laboratory scale to works dimensions: the two huge plants at Oppau and Merseberg represent the ultimate outcome.

Haber was a man of vision, depth of thought, and perspective, and his greatness lay in the conception of ideas of a scientific nature. Creation of work pleased him more than the consummated task. The writer learned much about the father from the son, whom he had the privilege of meeting in Czecho-Slovakia some four years ago, and with whom he spent several agreeable days.

## Synthesis of Ammonia

Technique in the synthesis of ammonia has become so firmly established that there is nothing of fundamental importance to report. One hears that chemical engineers of countries such as China are now sufficiently confident of the technique, that they purchase, for moderate sums, designs of complete plant, and proceed to purchase the necessary equipment unaided, and to erect without hesitation or demur. Production costs of synthetic ammonia are still of the order of about £11 10s. per ton (or 2s. 3½d. per unit of ammonia), at medium-sized works, when water gas is used as the source of hydrogen. Where coke oven gas is employed, under favourable circumstances, the price is of the order of £7 per ton. The German nitrogen cartel has decreed that the erection of further nitrogen plants in Germany shall be prohibited. In view of the excess production capacity throughout Europe, it can hardly be imagined that there is any inducement to further extension.

The water gas reaction for the production of hydrogen has been studied by a group of Japanese workers, who have produced a  $\text{Cr}_2\text{O}_3/\text{Fe}_2\text{O}_3$  catalyst of permanent activity. Hydrogen from a variety of sources is now used in the synthesis of ammonia. One process utilises by-product hydrogen (a) from the manufacture of NaOH, (b) from fermentation processes, (c) from the cracking of natural gas, and (d) from water gas, as well as electrolytic hydrogen.

The poisoning of various catalysts by gaseous impurities has been the subject of work by a group of Russian investigators. Patents dealing with the removal of inert impurities by "bleeding" a cyclic system have been published by the Atmospheric Nitrogen Corporation (U.S. Pat. 1,938,598, U.S. Pat. 1,931,678 and Brit. Pat. 413,517). These patents cover the removal of  $\text{NH}_3$  from the "bled" gas by (a) treatment at a higher pressure in the main circuit, (b) passage over a more active catalyst in a secondary system, and (c) treatment at a higher temperature.

## Conversion to Sulphate of Ammonia

Where synthetic ammonia and oleum are the raw materials in the manufacture of ammonium sulphate, it has been found that a considerable quantity of steam is generated in the dilution of the oleum to the desired specific gravity for the bath. Indeed, the quantity is adequate to admit of a suitable suspension of crystals within the mother liquor of the saturator, and the growth of a product of satisfactory crystal size. Such a method has a distinct advantage in that it promotes economy by reducing the consumption of extraneous steam.

The close association of the chemical industry with the study of the use of fuel has been urged in these reviews for

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several years now. Such close connection was strikingly demonstrated at the recent annual meeting of the Institute of Fuel. No one is better equipped for undertaking the hydrogenation of coal than those who have had experience of high-pressure technique, such as the synthetic ammonia industry affords. Yet the best material for hydrogenation is in the hands of the gas and coking industry. Nothing is more suitable for hydrogenation than vertical retort or low-temperature tars and creosote. The pity is that in the interests of national security, the gas and coking industry did not originally interest itself in synthetic ammonia production.

Hydrogenation of low-temperature tars, with the use of the recognised catalysts, leads one to expect a yield of about thirty gallons of petroleum light spirit per ton of tar treated. Where attempts are made to accelerate hydrogenation by rapid turbulence, and without catalysts, yields of the order of 15 to 16 gallons per ton can reasonably be anticipated.

## Action of Solvents on Coal

Much information on the action of solvents on coal has been contributed by the Fuel Research Board, in its Technical Paper No. 37, and Dr. G. E. Foxwell's review of that publication (THE CHEMICAL AGE, January 6, 1934), can be commended as an excellent synopsis of that work. Pyridine is regarded as a more convenient solvent than benzene, although both are primarily of use for research purposes only. Neither solvent would appear to be suitable for extracting the whole of the coke-forming constituent, since a large proportion of the groupings which promote tar formation is not extracted, either by pressure-benzene or pyridine. Evidently, the determining factor as regards the efficiency of pyridine is the presence of moisture. Relatively small percentages of water, either in pyridine or the coal substance, greatly reduce the solvent action of pyridine, particularly the  $\beta$  fraction. Since tar is evolved during the earlier stages of carbonisation, it originates in the external groupings. In point of fact, it is found to diminish in quantity when these external groupings have been removed, as, for example, by partial hydrogenation, which is the initial action of hydrogenation.

This year assuredly marks a new era in the low-temperature carbonisation of coal, since it has been established by Low-Temperature Carbonisation, Ltd., on a profitable basis. In a recent contribution, Colonel Bristow has made it clear that the utilisation of the gaseous product is the least-developed part of the low-temperature carbonisation industry. That the gas possesses potentialities cannot be doubted. Stripped low-temperature gas contains as much as 10 to 12 per cent. of ethane. Chlorinated ethanes and ethylenes constitute a useful range of non-inflammable solvents.

The erection of a plant designed to produce yearly 100,000 tons of oil from coal, and capable of treating, in addition, certain quantities of high- or low-temperature tar, was commenced in the autumn of 1932 by Imperial Chemical Industries, Ltd. The capital expenditure on the plant in question is estimated at more than two and a half million pounds. It has been made clear that the elimination of the patent difficulties by the formation of the International Hydrogenation Patent Co. was a striking example of co-operation of world interests, directed to secure the orderly development of an industry with an attractive future.

Interest was aroused, towards the end of last year, concerning the production of sulphur by the gas and coking industry. Several methods are available: indeed, they increase each year. Practice necessarily varies in different countries. Hydrogen sulphide is available in crude coal and

coke-oven gas, in the waste gas from ammonium sulphate plants where gas liquor is distilled, and there are growing quantities of  $\text{SO}_2$  available from smelter and other allied works.

Growing nationalism is beginning to exert an influence in connection with this question. Belgium has been regarded as the country with cheap sulphuric acid, arising from the roasting of zinc blende. She now finds that Germany is indisposed to take delivery, to the same extent as hitherto, of the cheap sulphuric acid which she manufactures. The question arises at once: how will it be possible for Belgium to maintain her spelter industry, in the absence of important Continental markets previously available? Obviously, she is being forced to consider methods for the production of sulphur from the smelter gases, or to seek fresh outlets for sulphuric acid. Neither alternative admits of ready solution, for obvious reasons.

Several methods are available for the conversion of the  $\text{SO}_2$  of smelter gases to sulphur. These involve reduction processes—usually the use of water-gas, in the presence of a suitable catalyst, such as iron, nickel or cobalt. But not infrequently, low concentration of  $\text{SO}_2$ , and the presence of oxygen in the gases to be treated, constitute factors that affect either (a) the efficiency of the technique, or (b) the economics of the recovery. Provision has been made by certain patented processes, not only for the removal of oxygen, and for the concentration of  $\text{SO}_2$ , but for the preparation of the last-named gas, in a suitable condition for rapid and economic reduction to sulphur. More will be heard concerning this process during 1935, but the pyrites companies have already available at least two processes for the recovery of sulphur from pyrites. The Lenander process has been established in Norway, and at the Rio Tinto Mines in Spain. It is capable of immediate production on a relatively large scale, immediately world conditions for the sale of sulphur are more favourable.

### The Gas and Coke Industries

If, as was urged in last year's review, spent oxide is becoming increasingly impure, and the growing cheapness of sulphur (American and Italian) renders spent oxide a less attractive sulphur material for the manufacture of sulphuric acid, is it likely that the gas and coking industry will remain apathetic in the face of a threatened loss of market? Will the gas and coking industry seek to introduce improvements in its oxide purification, by the adoption of the Lenze (tower purification) system, or will the coking industry resort to the Phyllox process, of which details were furnished in the contribution "Recovery of Sulphur from Coal Gas" (THE CHEMICAL AGE, December 23, 1933)? Or will the gas industry cheapen its methods of producing spent oxide, and proceed a step further, and subject the spent oxide to heat treatment, at known temperatures, at which the tarry matter will be sintered to a coke, and thus admit of extraction of the sulphur by carbon bisulphide, in a relatively pure form?

Certainly, when the closing of Continental markets is a *fait accompli*, some such consideration as the foregoing will become imperative. The depreciation of the pound sterling has aided the sale of sulphur-containing spent oxide abroad, but it would be inexpedient to rely on the permanency of this outlet, having regard to rapidly changing fiscal and economic circumstances.

### Sulphuric Acid

Recent developments in the tower chamber process, where oxides of nitrogen are used as the catalytic medium, bid fair to stimulate acute interest. Those who have asserted, in the past, that the days of the tower chamber process were numbered, that the contact process would usurp it as a more practical and economic system, may yet have to retract their words. The intensive systems which have just claims to recognition are the Gaillard-Parrish, the Mills-Packard and the Petersen (essentially a continental system). More than a million tons of sulphuric acid is being produced annually by the Gaillard-Parrish and Mills-Packard plants.

In America, a trial has been made of the Gaillard-Parrish system, in a tower 50 ft. square by 72 ft. high, with four large-sized turbo-dispersers arranged in compact formation. Without dispersers operating, the amount of acid condensed in

the tower chamber in question was equivalent to 43.1 tons of 60° Bé. acid per day. With dispersers working, the production increased to 89.6 tons of 60° Bé. acid daily, representing a make of 17.6 kg. of 60° Bé. acid per  $\text{m}^3$  per day, but results equally striking have been obtained by the interposition of two tower chambers in a rectangular set, one after the Glover and the other before the Gay Lussac towers. The production in two separate cases has been increased by more than fifty per cent., with a remarkably low nitric acid consumption. It is clearly preferable to erect tower chambers, equipped with turbo dispersers, rather than apply turbo dispersers to the old type of rectangular chamber plant, where the connections and draughting may be such that full advantage cannot be taken of the liquid phase system.

The production, with the Gaillard-Parrish system, is known to increase materially when heated towers are used, and strongly nitrous acid is dispersed. The invention of a double toggle block of obsidianite now admits of the satisfactory lining of large tower chambers, without any fear of collapse of the lining, or any corrosion of the lead. This marks a distinct advance in technique, and production should increase up to 40 kg. or more of 60° Bé. acid per  $\text{m}^3$  per day, according to the strength of the nitroso dispersed.

Apart from the above (which only briefly recounts certain directions in which experimental work by the writer has proceeded), intensification of the chamber or tower chamber processes has been the subject of experiment by several chemical engineers in other countries. Two Russians, Samarski and Ziberlich, have declared that in a small tower, of special construction, lined with fused basalt or acid-resisting concrete, and possessing a capacity of 100 cubic metres, they can produce as much sulphuric acid as a Petrusen plant of 2,000 cubic metres capacity, and consisting of two Glovers and two to three Gay Lussac towers. Again, E. K. Lapatto and A. M. Savinaev have published results to the effect that sulphuric acid is obtained in yields of 45 tons per diem per  $\text{m}^3$  of reaction space by injecting sulphur dioxide under pressure into  $\text{NO}_2\text{OH}$  at 20° to 50° C., the process of absorption and oxidation taking place simultaneously.

### The Lead Chamber Process

Following the foregoing, Professor Ernst Berl has worked in America on the reaction mechanism of the lead chamber process, and the constitution of violet acid [ $(\text{H}_2\text{SO}_4)\text{NO}$ ]. He has confirmed what was known, that nitric acid cannot act as a catalyser, has determined the vapour tension of nitroses, within the range of concentration of 50° to 62° Bé., at temperatures of 30° to 150° C., and has conducted pressure synthesis experiments, with such success that phenomenal quantities of sulphuric acid can be produced per unit volume of reaction space in unit time. Indeed, by pressure synthesis the following data have been arrived at:—

	Production, Kg. of 60° Bé. $\text{H}_2\text{SO}_4$ per $\text{m}^3$ per 24 hours.	Loss, Kg. of 36° Bé. $\text{HNO}_3$ per 100 kg. $\text{H}_2\text{SO}_4$ .
At 13 atmospheres	3,800	0.9
At 25.3 atmospheres (extrapolated)	12,600	—
At 53 atmospheres (extrapolated)	59,000	—

In conducting his experiments, Professor Berl has synthesised nitrosylsulphuric acid, and has found that it is a real nitrous compound.

The production of sulphuric acid by pressure synthesis is now regarded as an essentially engineering problem, within the ambit of practical realisation. A wet catalytic process has been developed in Germany. Vanadium is used as the catalyst. Its application is as follows:  $\text{H}_2\text{S}$  is burned to  $\text{SO}_2$ , the conversion of the latter to  $\text{SO}_3$  is effected, and steam is produced. The two products (steam and  $\text{SO}_3$ ) are condensed as sulphuric acid of 96 to 98 per cent.  $\text{H}_2\text{SO}_4$ . The plant is peculiarly compact, and easily controlled.

In the construction of lead tower chambers, it has been found that it is a great advantage to suspend the side sheets, rather than to attach these rigidly to the framework, whether the latter is constructed of wood, steel or ferro-concrete. The life of the lead with this method of suspension is practically double that of the rigid form of attachment.

Sodium nitrate has been used as a flotation agent in the

pyrites plant of Cia. Minera de Tocopilla for eighteen months now, with apparently good results.

The Norsk Hydro, Oslo, have covered a process (Brit. Pat. 407,312) for the production of sodium nitrate from nitrous gases and sodium chloride. The nitrate solution obtained by absorbing the residual gases of a nitric acid plant in a soda ash solution is converted with ammonium carbonate to ammonium nitrate, and a soda ash solution for re-use by the employment of base-exchanging substances. Sea water or other suitable naturally occurring solution can be used to provide sodium chloride for the last step of the process. If this technique develops satisfactorily on a large scale, Norway, instead of being a large importer of soda ash, may have a surplus for disposal.

Trials are being carried out in Scandinavia on a process (the "Nodon"), for the production of nitric acid from peat bogs, by electrolysis.

### By-Product Ammonia

Valuable work relating to the disposal of effluent liquor has been undertaken during the last twelve months, and results of peculiar interest have been published. There is now available a wealth of information on a variety of aspects of the liquor effluents problem, and gas undertakings should seek the advice of the Liquor Effluents and Ammonia Sub-Committee. The results afforded by the Fourth Report in this connection should be applied with discrimination. Increasing quantities of concentrated gas liquor are being manufactured, as represented by approximately 1,600,000 tons of coal. This product is being utilised in the manufacture of ammoniated superphosphate and other mixed fertiliser products.

### Ammonium Bicarbonate

It is interesting to observe that Russian chemists have been occupied in determining certain data relating to this product. I. S. Teletov, G. I. Groschtein and Z. P. Tkatschenko have found that the volatility of ammonium bicarbonate increases rapidly with rise in temperature, and with fineness of grinding. It is unaffected by adding ammonium nitrate, ammo-

ing the Nordengren process. The principal difficulty in the manufacture of phosphoric acid lies in the filtration of calcium sulphate. By the formation of the desired crystal of calcium sulphate, the development of which the Nordengren process ensures, by control of the acid concentration and temperature during the reaction, filtration can be effected in automatic suction filters, and phosphoric acid obtained at a concentration of 65 per cent. Where phosphoric acid of a concentration not exceeding 35 per cent. is a desired end product, the temperature required is sufficiently low to admit of working in open vessels. Filter construction on the principle of the Dwight-Lloyd band affords a very exact subdivision of the fractions to be washed, and advantage can be taken of the displacement principle, and mother liquor of constant concentration maintained.

### Superphosphate

Last year reference was made to the Broadfield and Oberphos processes—comparatively modern ones—for the production of calcium superphosphate. The Anglo-Continental Guano Works, Ltd., have apparently fostered the development of the Broadfield process, and have been responsible for its erection at Victoria Dock, E., Goole and Plymouth. On the other hand, Fison, Packard and Prentice, Ltd., have erected the Oberphos process at Ipswich, and are now concerned, through Natonal Fertilisers, Ltd., with the erection of a plant at Avonmouth.

What characterises the Broadfield process is essentially this: It is a continuous process, admitting of better attack of the rock phosphate by sulphuric acid. It produces a material of lower moisture content and better physical condition. The Oberphos process, on the contrary, is an intermittent process, conducted in apparatus as represented in Fig. 1. Two features characterise this process: (1) The material neither sets nor adheres to the walls of the autoclave; (2) the lead lining of the vessel is likely to remain intact for a long time. As regards the chemical and physical aspects of the process, it can be remarked: (a) the temperature of 150° C., and the pressure of about 14 lb. per sq. in., obviate conversion of any water-soluble P<sub>2</sub>O<sub>5</sub> to the insoluble form. (b) There is optimum concentration of sulphuric acid. (c) The rapidity of the reaction is remarkable, by reason of the temperature. (d) The mass is maintained in a liquid state. When the reaction is virtually completed, the pressure is reduced, and the autoclave is emptied. The sintered product is ground, and the ultimate material is in a granulated and suitably graded condition of attractive appearance and handle.

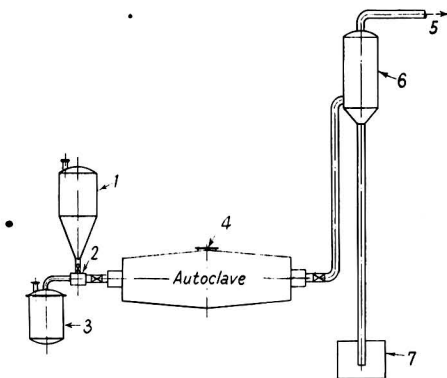


Fig. 1. The Oberphos Process. (1) Ground phosphate rock silo; (2) Mixing valve; (3) Acid cistern; (4) Manhole; (5) Outlet to vacuum pump; (6) Barometric condenser; (7) Condenser water suction tank.

nium sulphate, ammonium chloride or low-boiling-point hydrocarbons. It is augmented by peat, wood charcoal and phosphorite, in the order given, and the volatility is considerably diminished by high-boiling-point mineral oils.

A process for the manufacture of anhydrous sodium sulphide with a purity of 95 per cent., by the reduction of sodium sulphate with hot hydrogen gas, has been described by Dr. Paul Ley, in the "Chemiker Zeitung," October 24, 1934. The hydrogen can be heated either by gas, in a Cowper stove, or by other known means. The production cost depends primarily on the cost of hydrogen.

Further interesting information has been furnished concern-

## Leather Trades' Chemists

### Work of the British Section

DURING the year, the international character of the International Society of Leather Trades' Chemists has been widened by the formation of a Portuguese section, under the presidency of Professor Ney Matta, supported by an active committee. Apart from scientific work, this section is going closely into the question of instruction in leather technology in Portugal. The parent Society is now making plans for its biennial conference in 1935.

With regard to the British section of the Society during the year, this has actively co-operated with the Federation of Curriers and Light Leather Tanners in several joint conferences at which many technical subjects were discussed, to the advantage of both manufacturer and chemist. In addition, two meetings of the section were held, one at Leeds and the other in London during the Shoe and Leather Fair. The greatest achievement of the section during the year undoubtedly is the organising of a symposium on "Technical Aspects of Emulsions." This took place on December 7, under the chairmanship of Professor F. G. Donnan, at University College, where some 200 chemists from all parts of the country (several also coming from abroad for the occasion) participated in some valuable discussions. These papers will be made available shortly in book form. The officers for the year 1935 are: President, Mr. M. C. Lamb, vice-president, Dr. R. H. Marriott, treasurer, Dr. D. Burton, and secretary, R. Faraday Innes.

# Progress in Chemical Engineering

SEVERAL papers have been published during 1934 which mark definite steps along the road of chemical engineering, although no spectacular by-paths have been opened. Most of the development has been in the field of heat technology, chiefly by American investigators.

The mechanism of the flow of single-phase substances has been fairly well developed from the chemical engineering viewpoint, but much yet remains to be done on the flow of multi-phase substances, such as slurries and powders in gaseous medium. With regard to flow of single-phase substances, the chief paper published during the year is by Cooper, Drew and McAdams ("Ind. Eng. Chem.," 1934, 26, 428) on the isothermal flow of liquid layers. With regard to the latter aspect, Meldau and Stach (Report C56, German State Coal Council. See "J. Inst. Fuel," 1934, 7, 336) investigated the fine structure of powders in bulk, studying the packing of powdered coal and bakelite dust by a new technique, consisting in fixing the powders in their original configurations with molten wax prior to examination. A feature of loose powders was found to be the marked amount of bridging which occurs. This publication was illustrated by some remarkable photographs of the flow structure of powders obtained by using layers of various coloured bakelite in their experiments. The work is of significance with reference to powders in bulk, as in silos and bunkers, and for the design of plant for the handling of finely-powdered materials, such as filling and conveying machinery.

## Heat Transfer

Data useful for the design of heat transfer plant, using organic liquids as the heating media, and operating at temperatures of about 200-400° C., is being gradually accumulated, and Heindel ("Chem. Met. Eng.," 1934, 41, 308) gives account of some properties of Dowtherm A (73.5 per cent. diphenyl oxide and 26.5 per cent. diphenyl), as well as describing typical plant. The use of mercury vapour does not seem to be developing very rapidly, although it possesses many advantages for this class of process heating (Hulsart, "Chem. Met. Eng.," 1934, 41, 313), but the poisonous nature of the vapour and the high cost of mercury are serious drawbacks. Several power stations using the mercury vapour binary cycle have been erected in the United States during the last few years, and valuable experience has been gained in the design of industrial plant for mercury vapour processes.

An interesting cycle in which the application of chemical principles is probably more extensive than in other power cycles, is due to Koenemann (World Power Conference, Berlin, 1930), in which the addition compounds of zinc chloride and ammonia are employed. The addition compounds are decomposed in the boiler, and the highly superheated ammonia passes to a turbo-generator and exhausts to a specially designed jet-condenser "mixer," containing weak ammoniacate solution. It is probable that the mercury vapour cycle has a slight efficiency advantage, but the substances used in the Koenemann cycle are cheap, being by-products of the chemical industry.

## Heat Transfer in Finned Tubes

Findlay ("Power Engineer," 1934, 20, 434) has developed a combined diphenyl-steam cycle with an ideal efficiency between 80° and 100° F. of 57.3 per cent., which is about 5 per cent. higher than a corresponding mercury-steam cycle, and compares with a value of about 40 per cent. ideal efficiency for a modern steam cycle operating between the same temperature limits. The efficiency of the Findlay cycle is actually about 2 per cent. above the corresponding Carnot cycle efficiency, partly due to the property which diphenyl possesses of superheating on expansion. Since the theoretical efficiencies, and in some cases those obtained in practice, of such power cycles are greater than that of the steam cycle, a new field is opening up in which the chemical engineer will undoubtedly play his part.

With regard to heat transmission, Kay and Furnas ("Ind. Eng. Chem.," 1934, 26, 783) have derived an equation correlating the heat transfer coefficients obtained when heating

By H. Lewis, B.Sc., A.I.C., A.M.I.Chem.E.

and when cooling a turbulent liquid in a pipe, which differ, since during heating the relatively stationary film is comparatively thicker. The correlating factor is the inverse ratio of the viscosities of the fluid at the respective film temperatures to the  $n$ th power, the value of  $n$  being found experimentally.

A paper on heat transmission using finned tubes was published by Rauber ("Chaleur and ind.," 1934, 15, 73). Dieterlen, Margoulis and Meyer ("Chaleur and ind.," 1934, 15, 87) have experimentally studied the influence and arrangement of fins on the heat transmission to gases and liquids, developing graphical methods for the determination of shape factors and heat transfer coefficients for such tubes. Such papers are extremely valuable from the practical standpoint, and it is to be regretted that the literature of heat transmission is sadly lacking in certain aspects pertaining to actual practice, a notable example being the transfer of heat by coils. It is to be hoped that researches on this latter aspect will be undertaken soon, and full use should be made of the fundamental investigations of Hawes ("Trans. Inst. Chem. Eng.," 1933, 10, 161), who determined the temperature-velocity distribution curves for water flowing through a coiled pipe.

## Film and Drop-Wise Condensates

Since the first extensive discussion by Schmidt, Schurig and Sellschopp ("Tech. Mech. Thermodynamik.," 1930, 1, 5) on the character of the condensate layer obtained from condensing steam, interest has been focussed on the conditions governing the formation of film and drop-wise condensates. Several investigators have obtained values for the heat transfer with drop-wise condensation four times, and even greater, that obtained with film condensation. The chief factors governing the character of the condensate layer is the physical condition of the surface of condensation, *i.e.*, the number of the nuclei present upon which the vapour can condense, and the velocity of the vapour. Some interesting experiments relating to this question were reported by Jeffrey and Moyihan ("Mech. Eng.," 1933, 55, 751). The question is being studied at one of the American schools of chemical engineering and the results of their experiments are to be reported at an early date. Kirschbaum and Kranz ("Chem. Fabr.," 1934, 176) found that both film and drop-wise condensation may occur in practice, the latter being favoured by the presence of oil (see also Schmidt loc. cit.; Spoelstra, "Arch. Suiderind. Nederl.-Indie," 1931, 3, No. 23). Thus, using quiescent steam and a vertical copper tube, they found that generally mixed condensation was obtained, the heat transfer coefficient being about twice that calculated, according to Nusselt's theory ("Z. Ver. deut. Ing.," 1916, 57, 199), and that spraying in oil increased the heat transfer to six times the theoretical. A low wall temperature also favoured film condensation.

## Values of Coefficients

Hebbard and Badger ("Ind. Eng. Chem.," 1934, 26, 420) have measured the heat transfer film coefficients for practically air-free steam condensing on the outside of a 1 in. outside diameter vertical tube, 12 ft. long. Several investigators have studied heat transfer with shorter tube lengths, and the present work was undertaken from the viewpoint of application to forced circulation evaporators. The values of the coefficients obtained averaged about 33 per cent. from that called for by the Nusselt equation, but no explanation was given of this deviation, since it was concluded that there was no turbulence in the condensate layer, and that the constancy of the tube temperature indicated the absence of drop-wise condensation.

Kirkbride ("Ind. Eng. Chem.," 1934, 26, 425), in a paper on heat transfer by condensing vapour on vertical tubes, inclines to the opinion that the higher results obtained in practice than those predicted theoretically are due to turbulence in the condensate film. Kirkbride attacks the

problem of film condensation from the basis of dimensional analysis, but Colburn ("Ind. Eng. Chem.," 1934, 26, 432) attempts a more exact solution for the case of the film condensation of steam on a vertical pipe, when the condensed film is so thick that the flow is turbulent at the lower end. The derived equations do not, of course, hold under conditions causing drop-wise condensation, but the figures presented make possible a conservative estimate for the heat transfer surface. The question of the relative importance of drop-wise condensation or turbulence in the condensate layer in giving high heat transfer coefficients will play a big part in chemical engineering research in the immediate future, and may soon become of practical importance in the design of condensing plant (e.g., Wiltsch, Brit. Pat. 379,943; See "Ann. Rep. Soc. Chem. Ind. Prog. Appl. Chem.," 1933, 18, 14).

### Convection Heat Losses in Air

Lander ("Trans. Inst. Chem. Eng.," 1934, 12, 86) has given an account of some modern methods of attacking heat transmission problems, and of particular interest is the optical method of investigating the effect of shape and size of hot bodies on the convection heat losses in air. A ray of light is sent parallel and very close to the hot surface, and becomes bent away from the surface as it passes over it, due to the temperature gradient; the deviation, which is measured on a suitable screen, allows the heat loss to be calculated. The method has been shown to be applicable to convection in liquids and future investigations may allow of it being extended to forced convection. Incidentally, Lorenz ("Z. tech. Physik.," 1934, 15, 362) has studied the heat loss from a smooth vertical plate in oil with natural convection.

Several papers have been published on the various aspects of thermal insulation ("Krebe Elektrowarme," 1934, 4, 129; Woodward, "Trans. Ceram. Soc.," 1934, 33, 73; Stratton, "Paper Trade J.," 1934, 98, T.A.P.P.I. Sect. 115; and Boxser, "Amer. Dyestuff Rep.," 1934, 23, 442), but probably the most important is that by Kistler and Caldwell ("Ind. Eng. Chem.," 1934, 26, 658) on the thermal conductivity of silica aerogel. Silica aerogel powders possess the lowest heat conductivity at atmospheric pressure of any insulator so far reported. The average value for the aerogel powder is 10 per cent. less than that for still air. Kistler and Caldwell suggest an interesting use for such powders in the vacuum insulation of large containers.

### Crushing

Turning to the question of crushing, Carey and Bosanquet ("J. Soc. Glass Tech.," 1933, 17, 384) have shown, under free crushing conditions, the load being applied by simple crushing, shear or impact, that homogeneous brittle solids break down with a constant fracture pattern independent of the original specimen size, this relationship, however, being of a statistical nature. The minimum work required to crush a given powder is the product of the weight of the material crushed by the logarithm of the mean size reduction by a constant specific to the material. Their calculations show that the power requirements for industrial crushing are often a hundred times greater than the theoretical requirements. Carey ("Trans. Inst. Chem. Eng.," 1934) shows that when coal is crushed to less than one-third of its original size, the particles aggregate to a flake which requires a great expenditure of power for further size reduction, and states that ball mills and ring rolls tend to form aggregates; impact machines suffer excessive windage losses, hence future developments in crushing machinery should be in the direction of the multi-stage, high-speed roll crusher.

A review of recent theoretical and experimental work on the crushing of brittle minerals was given by Rothelieu ("Tek. Tid. Upl. C., Bergsvetenskap.," 1934, 64, 41, 40); and of practical value is a publication by Gow, Guggenheim, Campbell and Coghill ("Amer. Inst. Min. Met. Eng.," 1934, Tech. Pub. No. 517) describing the effects of the various factors influencing ball mill operation, and giving a formula by which the power requirements may be calculated from the mill dimensions. Of related interest is a paper on screening by Hudson ("Crushing, Grinding, Mining, Quarrying J.," 1934, 2, 161); a paper by Traxler and Baum ("Rock Prod.," 1934, 37, No. 6, 44) on the determination of particle size distribution in mineral powders by air elutriation; and a paper

by Alekseevskii ("Khimstroï," 1934, 6, 270) on a simplified calculation for an air-separation column.

An extensive investigation on the mechanism of filtration has been carried out by Carman ("J. Soc. Chem. Ind.," 1933, 52, 280T; *ibid.*, 1934, 53, 159T, 301T). The mathematical development was based on the theory that the solid structure of the filter cake transmits the whole of the pressure to the filter cloth, whereas the pressure of the liquid throughout the cake gradually decreases as it approaches the cloth. The pressure difference thus created causes deformation of the solid particles, restricting the pore space, and giving increase in cake resistance on approaching the cloth. The derived equation was tested experimentally for constant pressure filtration of ferric oxide and calcium carbonate, which form compressible cakes, and for kieselguhr, which gives a practically incompressible cake. The observed values of the specific resistance of the cakes were found to agree very well with those calculated. For compressible sludges, the specific resistance is proportional to a power function of the pressure employed in the filtration, and the actual value of the power was found to vary according to the method of preparation of the slurry. In general, it can be stated that the theory developed by Carman accounts for experimental results with a greater degree of accuracy than those propounded by previous experimenters in this unit process.

An explanation of the deviation obtained from theoretical results when using a large press, due to a particle settling effect obtained in the chambers of a large press, may lead to further developments in press design. Of immediate significance are the observations on the effect of the method of slurry preparation on the filtration rate. A slurry prepared as a floc will behave as a compressible cake, whereas a precipitate formed as separate small particles will form a cake of non-compressible characteristics. A big field for future investigations lies in the direction of determining the conditions for slurry formation to produce optimum filtration operation. Köppen ("Arch. Pharm.," 1934, 272, 698) has published a paper on the dependence of the velocity of filtration on the chemical and colloid structure of the particles.

### Evaporation

With regard to evaporation, Cessna and Badger ("Ind. Eng. Chem.," 1934, 26, 485) carried out experiments on entrainment in a laboratory forced-circulation evaporator and attempted to analyse the problem mathematically, and, although they developed an approximate fundamental formula, the practical results were almost as well correlated by considering the entrainment as proportional to the mass velocity. Claussen ("Z. Ver. deut. Zucker-Ind.," 1934, 84, 193), considering the influence of the air content of steam on the efficiency of evaporation, emphasised the fact that, as condensation occurs at the heat exchange surface, a local increase in air content is obtained, producing an almost stationary layer, which considerably reduces the efficiency, and he then discusses design factors to obviate this difficulty.

### Drying

A new drying process is reported from Germany. Kiesskalt ("Chem. Fabr.," 1934, 7, 281) has developed a process for accelerating the rate of drying by varying the pressure of the air in contact with the substance being dried, the pressure variation being of a pulsating nature of about 20-100 cycles per minute, using a pressure difference of the order of 10 in. of water. A decrease in the drying time up to 30 per cent. is claimed, and economy is particularly effected in the drying of viscose. Some further information on this process will be awaited with interest, since such drying acceleration would be expected to occur only with porous substances when the rate of drying was controlled by the diffusion of the vapour through the stationary gas film, and not to influence the rate when internal diffusion of vapour or liquid was the controlling factor, which is so often the case in industrial applications.

The literature of spray drying is exceptionally meagre, and one welcomes a critical article on the factors influencing the design and operation of spray dryers. Lewis ("Ind. Chem.," 1934, 10, 439), in a paper on this subject, also develops Sherwood's theory of the mechanism of drying to explain the characteristics of spray-dried products and suggests its application in the control of spray dryer operation.

In a continuation of work on tower absorption coefficients,



Simmons and Osborn ("Ind. Eng. Chem.," 1934, 26, 529) state that the actual free volume of an absorption tower is an inverse function of the rate of liquid flow and that its use simplifies the equations for absorption coefficients. Hollings and Silver ("Trans. Inst. Chem. Eng.," 1934, 12, 49) discuss the physical laws of equilibrium between gas and solution on the basis of the two film theory, and, although they do not contribute anything fresh to the development of this aspect of chemical engineering theory, they present useful confirmatory evidence with particular reference to the washing of illuminating gas. Keyes ("J.S.C.I.," 1934, 53, 692) published a study of the absorption of sulphur dioxide from flue gases.

### Distillation

Practice precedes theory in many aspects of distillation, but Underwood ("Trans. Inst. Chem. Eng.," 1934), in a paper continuing his previous mathematical studies of this subject, has derived general equations for the determination of plate efficiency in fractionating columns for complex mixtures, which does much to lessen this breach. Meyer ("Trans. Inst. Chem. Eng.," 1934, 12, 96) has aided him in the good work, by developing a method for the calculation of the products obtained from the distillation of infinite component mixtures by batch distillation and equilibrium flash vaporisation, which requires neither molecular weight nor specific gravity data, the conclusions finding full experimental support.

Sonders and Brow ("Ind. Eng. Chem.," 1934, 26, 98) have investigated the question of the effect of entrainment on the capacity of a column. A theoretical equation is derived to determine the vapour velocity necessary for entrainment. From this they calculate a factor for maximum capacity, and by means of curves show the relationship between the maximum capacity and the plate distance, and also the effect of various operating factors, such as introduction of the reflux at various levels and the withdrawal of side streams. Another paper of practical significance for the design of fractionating columns is by Rogers and Thiele ("Ind. Eng. Chem.," 1934, 26, 524), who have studied the effect of different operating conditions on the pressure drop in bubble cap columns using a single triangular or rectangular slot.

The literature of chemical engineering contains scarce mention of agitator efficiencies, and data useful for the design of mixers or agitators is practically non-existent, hence studies, such as those by Hixson and Wilkens ("Ind. Eng. Chem.," 1933, 25, 1198) and those by White and Sumerford ("Ind. Eng. Chem.," 1934, 26, 82), on the performance of agitators in liquid-solid systems, tend to fulfil a long-felt want for design purposes. The latter investigators carried out sieve analyses of sand in water drawn from various points in a tank in which an agitator was in operation. Their results afforded definite evidence that classification occurred under the conditions of their experiments. Further work on similar lines to determine the optimum design and operating conditions is still necessary.

### Foaming in Boilers

Foulk and Whirl ("Ind. Eng. Chem.," 1934, 26, 263) have contributed a further paper to the former's already extensive studies on the problem of foaming of water in boilers, the present paper dealing with the loss of foam stabilising properties of suspended solid matter at high boiler pressures. It was found that finely powdered substances, which are not readily wetted by water, tend to stabilise the foam on introduction into a boiler, but, owing to a change in the wettability as the boiling proceeds, the solids soon lose their stabilising properties by being removed from the sphere of action in the surface films. Lewis ("Power Engineer," December, 1934) has reviewed the present state of knowledge on the foaming in boilers, and advances the suggestion that this change in wettability is due to the gradual removal of a surface film of adsorbed gases on the solid particles. He also suggests a mechanism of the action of anti-foams, which often contain polar hydroxyl groupings, in being involved in the orientation of the molecules of the anti-foaming agent, e.g., castor oil, at the surface of the films in the foam. These suggestions need experimental verification.

Hunter and Nash ("J.S.C.I.," 1934, 53, 95T; see also "J.S.C.I.," 1932, 51, 285T) have applied phase rule graphical methods to the design of liquid-liquid contact equipment, triangular co-ordinates being employed for the representation

of equilibrium in a three-component system, a method also being given to deal with the practical case when the ideal equilibrium is not fully established.

Although the development of alloys for the construction of chemical plant is all-important to the furtherance of chemical engineering practice, it is such a vast subject and publications so numerous that it must be considered separately and to fall outside the scope of the present article. Mention must be made, however, of the special materials of construction number of one of the technical publications ("Chem. Met. Eng.," October, 1934) in which current practice is systematically summarised. Mention can also be made of a paper by Perry ("Paint and Varnish Prod. Mgr.," 1934, 10, No. 7, 5) on a study of the action of various chemicals upon different woods used for chemical tanks, from which it was concluded that wooden tanks can be used for practically all commercial solutions in reasonable concentrations, cypress, pine and fir being the most suitable woods; and nitric acid and caustic soda have the most pronounced action on wood. Attention must also be called to a small treatise by Stevens and Donald on rubber in chemical engineering, published by the Rubber Growers' Association. In connection with rubber and its derivatives, Twiss ("J.S.C.I.," 1934, 53, 723) contributes an article on the character of rubber as a chemically-resistant material; Coolahan ("J.S.C.I.," 1934, 53, 630) publishes an article on Torneis; and chlorinated rubber also receives further mention ("J.S.C.I.," 1934, 53, 761).

With regard to general chemical plant construction, the list of cements and their properties recently published ("Ind. Chem.," 1933, 9, 381) receives further additions ("Ind. Eng. Chem.," News Ed., October 10, 1934; see also Olive, "Chem. Met. Eng.," 1934, 41, 10). Gaskets receive treatment in two articles, one by Sandstrom ("Chem. Met. Eng.," 1934, 41, 130), and one by Perry ("Chem. Met. Eng.," 1934, 41, 194), the former giving an account of stress-strain behaviour, with especial reference to pressure vessels. Audibert and Raineau ("Ann. combustibles liquides," 1934, 9, 203) discuss the construction of cylindrical pressure vessels for chemical processes, making special reference to the design of vessels for operation in contact with hydrogen at high temperatures, such as in hydrogenation processes. Mann and Parkes ("J.S.C.I.," 1934, 53, 847, 907) give a practical account of the problem of tar still corrosion.

## Colour Users' Association

### Some Valuable Achievements

FOR the Colour Users' Association the year has been a period of important work and valuable achievements, and the council is confident that the whole-hearted support of colour users will be forthcoming for the work that lies ahead in the safeguarding of their interests. The outstanding event has been the passing of the Dyestuffs (Import Regulation) Act, 1934. The present position is that the 1920 Act becomes permanent and the importation of dyestuffs is prohibited except under licences granted upon the recommendations of the Dyestuffs Advisory Licensing Committee. Dyestuffs are not to be subject to import duties but intermediates are still subject to 10 per cent. tariff. The Import Duties Advisory Committee is constituted an appeal board to which representations may be made by interested parties who consider they are unduly prejudiced on price grounds. A joint standing committee has been set up.

The situation arising from claims made in respect of dermatitis alleged to be caused by dyed materials has been considered by the council, and steps have been taken along with other interested bodies to investigate the whole matter. A small committee has been set up representing dye-users and dye-makers, with a view to collating all available information and seeking the best legal and technical advice.

The work of the joint technical committee, consisting of four dye-makers and four dye-users, has contributed largely to the smooth working of the Dyestuffs Act and to the speedy dealing with applications for licences. The year has been an active one and the council has been called together frequently, principally to deal with the Import Duties Advisory Committee's report, the Dyestuffs Act, representation on the Dyestuffs Advisory Licensing Committee, the intermediates question, and dermatitis.

# Textile Bleaching, Dyeing and Finishing

By A. J. Hall, B.Sc., F.I.C., F.T.I.

IT is somewhat ironical that although methods of colouring and finishing are constantly being improved, the margins of profit allowed to the dyer and finisher are continually being decreased. In both the woven and knitted trades, prices for bleaching, dyeing and finishing have steadily come down during the past year until most firms find it difficult to secure a profit. The reason for this is not far to seek. The dyeing and finishing plant available is capable of dealing with much more textile material than is at present in demand. Consequently, there is exceptionally keen competition for the work that is to be had. Attention is drawn to this economic situation in the dyeing and finishing trades because it has an important bearing on technical progress. It is mainly in dyeing and finishing works that research is carried out for the discovery of improved methods. This research involves expenditure and if the profit margins are small then there is likelihood of a curtailment of research costs. Price cutting when carried too far is thus able to retard progress, and it is feared that this point is now being reached.

## Wool Processing

During the past year quite a considerable amount of attention has been given to wool processing. Early in the year S. R. Trotman and H. Horner published a paper dealing with the influence of sulphuric acid in the dyeing of wool with acid dyes ("J. Soc. Dyers Col.," 1934, 50, 65), and quite recently the absorption of acid dyes by wool has formed the subject of researches by J. B. Speakman and his collaborators (*ibid.*, 342 and 348). S. R. Trotman, H. S. Bell and H. Saunderson ("J.S.C.I.," 1934, 53, 267T) have also investigated the effect of chlorination upon the affinity of wool for acid dyes.

When wool is dyed with an acid dye in the presence of sulphuric acid there is absorption of both acid and dye. The amount of dye absorbed increases directly with that of the acid up to a maximum of about 0 per cent.; the absorption of sulphuric acid takes place rapidly and reaches a maximum before the dye does since this is absorbed slightly more slowly. These facts were elucidated by dyeing wool at the boil for 3 hours with 10 per cent. of Indigocarmine, 6 per cent. of sulphuric acid, and 20 per cent. of Glauber's salt. When the amount of sulphuric acid present in the dyebath is 6 per cent. practically all the absorption of acid is complete within the first hour, but by increasing the acid concentration to 10 per cent. the period for maximum acid absorption is reduced to one half-hour. Absorption of acid and dye is similar when hydrochloric acid is used instead of sulphuric acid, but with acetic acid considerably less dye and acid are absorbed by the wool. All these facts are of importance in the dyeing of wool goods where levelling difficulties are met with.

Hitherto, an objection to the chemical theory of dyeing wool with acid dyes has been that de-aminated wool has been found by various workers to have an affinity for dyes not substantially different from that of ordinary wool. Wool is known to contain amino groups and it is reasonable to assume that in dyeing these combine with the colour acid and also that the amount of dye so fixed within the wool would be proportional to the amino content. These amino groups can be removed by treating the wool with nitrous acid (de-amination) so that the affinity of the wool for acid dyes should then be considerably less. Benz and Farrell ("J.S.C.I.," 1897, 106, 406), Paddon ("J. Phys. Chem.," 1922, 26, 384) and Trotman ("J.S.C.I.," 1924, 40, 77) have not found this to be the case. Quite recently, however, J. B. Speakman and E. Stott ("J. Soc. Dyers Col.," 1934, 50, 341) have re-investigated this point and have been able to reconcile the facts with the chemical theory of dyeing.

## Affinity of Wool for Dyes

Speakman points out that the affinity of wool for acid dyes must not be attributed entirely to the presence of amino groups but due regard must be paid to the basic character of the amino groups which are present. Thus, if wool could be completely de-aminated then it is unlikely that wool would have absolutely no affinity for acid dyes. At the most, there-

fore, de-amination could only produce a lowering of its dye-stuff affinity. But, in addition to this, Speakman points out that it is most difficult, if not impossible, to effect a complete removal of the amino groups with nitrous acid, since the guanidine group of arginine (this is the main basic amino-acid of wool) reacts extremely slowly with nitrous acid and drastic treatment is necessary to ensure complete reaction. When such drastic treatment is applied to wool it is found that the resulting de-aminated wool has much less affinity for dyes than before. Thus, the main objection to the chemical theory of dyeing is removed.

General practical experience shows that wool which has been chlorinated (as in the non-shrink process) is liable to dye unevenly during the application of acid and basic dyes. The chlorinated wool usually dyes deeper than before chlorination and the unevenness of dyeing is probably due to uneven penetration by the chlorine. But, according to Trotman, Bell and Saunderson (*supra*) a further notable property of chlorinated wool is that it absorbs dyes very rapidly. When chlorinated and non-chlorinated wools are dyed together with a moderate amount of acid dye it is found that practically all the dye goes on to the chlorinated wool, and this latter property has now been utilised as the basis of a control test in the non-shrink processing of wool materials.

In the non-shrink process, wool is treated with chlorine liquors until 25 to 35 per cent. of the wool fibres appear under the microscope to have their epithelial scales removed or damaged. The more drastic is the chlorination, the larger is the number of damaged fibres, but chlorination should not be carried further than the above-mentioned limits, otherwise the wool loses most of its durability. It is now proposed to determine the degree of chlorination by dyeing the wool under standard conditions and ascertaining the amount of dye absorbed. Trotman and his collaborators find that the dye-stuff absorption of satisfactorily chlorinated wool should not exceed 0.35 millimols of Kiton Red G per 100 grams of wool.

## Non-Shrinking Wool

Progress during the past year in the treatment of wool materials has also been definitely marked by the discovery of a new non-shrink process for wool, details of which are to be found in Brit. Pat. 417,719. This discovery was made in the laboratories of the Wool Industries Research Association at Torrion. Briefly stated, the process consists of treating wool with chlorine gas under defined conditions and then removing this by passing through the wool a current of air followed by washing with water which may contain an anti-chlor. The success of the process is due to the manner in which the wool is treated with the chlorine, evenness of penetration being secured by first exhausting the wool of occluded air within a closed chamber connected to vacuum. It is claimed that wool in all its forms (loose fibres, yarns and manufactured articles) can be made unshrinkable by this process and that instead of losing strength the treated wool is actually 30 to 50 per cent. stronger. After chlorination by this process loose wool is more adaptable to spinning.

Factors which affect the level dyeing of acid dyes on wool have been investigated by J. B. Speakman and H. Clegg ("J. Soc. Dyers Col.," 1934, 50, 348), and their conclusions are particularly interesting. It seems that the levelling properties of acid dyes are intimately related to their colloid character in aqueous solution, this fact having been determined by comparing the minimum concentrations of sodium chloride and sulphuric acid needed to precipitate such dyes from solution and the amounts of dye unexhausted (during dyeing) in the presence of high concentrations of sodium sulphate. As a dyestuff solution becomes more colloidal so it is the less likely to yield well-levelled dyeings. The tendency for a dye to become colloidal in an acid dyebath increases with the molecular weight of the dye and decreases with increasing degree of sulphonation. The best levelling properties are to be found in dyes having their sulphonic acid

groups far removed from the azo group and from each other.

Turning now to rayon, it must be noticed that quite a large amount of research has been devoted to improving methods for producing rayon and rayon goods having a matt appearance. During the past year the vogue for delustrated rayon materials has persisted and it would seem that the coming year is likely to see no change in this whim of fashion. Fabrics of viscose or cellulose acetate rayon can be made from delustrated rayon yarns or they can be made with bright rayon yarns and afterwards delustrated. In general, it is only by use of delustrated rayon yarn that a permanent matt appearance can be obtained, for in this case the yarn is produced by adding opaque pigments to the rayon spinning solution. When delustrating forms part of the finishing process the reduced lustre is secured by coating the surface of the rayon fibres with a superficial layer of pigment (usually china clay or bentonite) and this is easily removed by washing.

#### Delustrated Rayon Yarns

Delustrated rayon yarns are now almost all produced with titanium dioxide. This substance, when produced in a suitable form, is the most satisfactory substance so far found for adding to viscose and cellulose acetate solutions before spinning, because it is of a good white colour and is very inert towards acids and alkalis and, indeed, all reagents with which rayon is likely to come in contact during bleaching, dyeing and finishing processes. The delustrating pigment is first obtained from titaniferous ores in the form of titanium hydroxide, and although this can be used for delustrating rayon by external application, it is better for the purposes of spinning delustrated rayon directly to calcine it until its refractive index is increased from 1.8 to about 2.7. The higher is the refractive index the greater is its delustrating power when added to a rayon spinning solution. The titanium hydroxide must, however, not be excessively calcined, otherwise it cannot be satisfactorily ground into particles sufficiently fine not to block the spinnerets. A disadvantage of titanium dioxide is that it causes a high rate of wear and tear on the plant.

Recently it has been found that chlorinated diphenyl (Brit. Pat. 384,224) is useful for adding to rayon spinning solutions as a delustrant; its refractive index is about 1.6 to 1.7, this being somewhat less than that of titanium dioxide. On account of this lower refractive index it has therefore been recommended (Brit. Pat. 409,625) to use mixtures of titanium dioxide and chlorinated diphenyl. Improved methods of producing titanium dioxide suitable for delustrating are disclosed in Brit. Pat. 405,669.

#### Prevention of Fading

Early in the use of delustrated rayon containing titanium dioxide it was noticed that the opaque pigment could accelerate the fading in sunlight of dyes present in the rayon. Thus, delustrated viscose stockings, washed and hung out to dry in sunlight, were found to fade extremely rapidly. Further experience showed that the fading took place particularly when the stockings were wet during exposure to light. Ultimately it was ascertained that the true cause of the trouble (Sandoz Chemical Company; "J.S.C.I.," 1934, 53, 34) was that in the presence of glycerine (this could be present in the rayon as a manufacturing residue) the titanium dioxide became photo-sensitive so that it could be reversibly reduced to titanous oxide and in these changes it catalytically affects organic dyes. Certain dyes have been found to be comparatively immune to this peculiar action of titanium dioxide and these have been selected by various dyemakers and recommended for use with delustrated rayon. L. Keiner ("Textilber.," 1934, 75, 118) has discussed this matter fully in reference to both viscose and cellulose acetate rayons.

Although it is possible to secure good delustrating of viscose rayon by treating it successively with solutions of barium chloride and sodium sulphate so that insoluble barium sulphate is deposited within the fibres, there are several disadvantages of this delustrating process. Lately, alternative methods have been disclosed. Thus, according to Brit. Pat. 408,240 barium stannate (formed from barium chloride and an alkali stannate) and barium tungstate (Brit. Pat. 415,822) are more effective delustrants than barium sulphate. Aluminium sulpho-acetate or aluminium formate can also be used (Brit. Pat. 401,477) for delustrating rayons.

Practical dyers now find that they have considerable quantities of rayon materials which require to be dulled after

dyeing. It is possible to use for this purpose the various insoluble barium compounds indicated above, but the simplest method is that of impregnating the rayon fabric with a suspension of a colloidal clay, such as china clay or bentonite, followed by drying. In this manner the rayon fibres become covered with a thin opaque film which has the disadvantage of being fairly easily removed by washing. In spite of this defect, huge quantities of delustrated rayon goods are produced by this simple method and numerous products are now being marketed which consist of pastes of opaque insoluble substances. Latex has been suggested as a means for securing more permanent adherence of the opaque pigment particles to the rayon.

(To be continued.)

## Society of Public Analysts

### The Year's Papers

THE Society of Public Analysts, whose president is Mr. John Evans, now has 740 members. During the year papers concerned with every branch of analytical chemistry have been read and discussed at the meetings and subsequently published in the "Analyst." Several of these papers have been the outcome of work done under the Society's analytical investigation scheme, although chemists who undertake such work are not necessarily members of the Society. "Six problems are still being investigated.

The following publications have also been issued by the Society: "Fifty Years of the Society of Public Analysts," comprising Dr. Dyer's reminiscences of the Society since its inception in 1874 and a complete survey of the activities of the Society, which is practically an epitome of the progress of analysis during the period (price 12s. 6d.); and "A Bibliography of certain Heavy Metals occurring in Food and Biological Material," in which the references are classified (with a précis of their contents) under the respective metals (price 3s.).

The North of England section has held regular meetings for papers and discussions, and at the summer meeting of the section a lecture was given by Professor Laurie, D.Sc., on "Old Masters and Modern Forgeries." Particulars regarding the Society can be obtained from the secretary, 85 Eccleston Square, S.W.1.

## Newcastle Chemical Industry Club

### Losses by Death and Removal

THE Newcastle Chemical Industry Club has continued to serve a useful purpose for scientists in the Newcastle-upon-Tyne and North-East Coast area during the past year under the presidency of Dr. J. T. Dunn. Mr. H. Dunford Smith has again proved a popular chairman of committee, and it is pleasing to record another successful year and an increase in membership. The expansion of the library has been somewhat curtailed by the falling-off in subscriptions from local firms owing to the continued industrial depression. It is gratifying, however, that some firms and individuals have continued their support even under such trying circumstances and they deserve great credit.

The club has been unfortunate in losing some prominent members, one being Mr. Francis de Gisbert, the Arctic explorer and scientist, who died in July last. It is hoped that the results of some of his work will be published. Another death was that of Mr. Amos Shaw, who was killed when on duty by the tragic explosion at Washington, Co. Durham. The club has lost, by resignation, a former hon. secretary, Mr. G. B. Howarth, who has left England to take up the post of fuel adviser to the Government of New South Wales. Lectures and talks have covered a wide range of subjects, including "Heraldry," by Mr. Appleby Miller, "Science and the Drama," by Mr. Herbert J. Scott, of the People's Theatre, Newcastle, and "The Measurement of Colour in the Soaps and Detergents Industry," by Mr. H. S. Priestley. There is an item of particular interest in February next, when Mr. Russell Goddard, F.L.S., Curator of the Hancock Museum, will open a discussion on "The Preservation of Museum Specimens."

# Recent Trends in the Nitrogen Industry

By E. B. Maxted, D.Sc., Ph.D., F.I.C.

**O**WING to the abnormal position, especially in the synthetic ammonia industry, in which the capacity of existing plants is greatly in excess of demand for nitrogenous fertilisers, the amount of work in this field has been far less than during the development period; and this trend towards a smaller number of papers and patents has been favoured by the gradual stabilisation of technique referred to in last year's review. Thus, the German fixed nitrogen plants are stated to be working at only 35 per cent. of their capacity, and the erection of new plants in the German nitrogen cartel has been prohibited until 1940<sup>1</sup>. In Great Britain, larger sales of nitrogenous fertilisers to home consumers, compared with those during the previous year, are reported<sup>2</sup>, but export sales have declined. It will be remembered that at one time the world capacity of synthetic ammonia plants was being increased at a rate of the order of 200,000 tons of nitrogen yearly. The agricultural nitrogen market is very sensitive to factors associated with confidence and with the general degree of prosperity (indeed, this may well account for the increase in home sales, referred to above); and, with the gradual return of the world to normal conditions, the erection of plants having capacities commensurable with the estimated potential demands, rather than with the actual sales during difficult times, may still be more than justified. Moreover, apart from this rational agricultural aspect, the possession of adequate, and even of a reserve, manufacturing capacity for fixed nitrogen is, of course, an asset of fundamental importance in each country from the standpoint of national defence.

## Synthesis of Ammonia

General procedure in this industry, with particular reference to the practice of the Nitrogen Engineering Corporation, has been dealt with by Brown<sup>3</sup>. In the course of an interesting discussion of the various available methods of nitrogen manufacture, electrolysis in the ordinary way, followed by the compression of the gas, is stated to be more economical in practice than electrolysis under pressure. Normally, however, hydrogen for the synthesis is obtained from some form of fuel gas or as an already existing by-product. The paper also includes a number of chemical engineering data.

Several papers and patent specifications deal with the elimination of poisons from the gases used, and with the effect of the commoner poisons on the output of ammonia. Thus, pre-catalysts for the elimination of carbon monoxide have been discussed by Kamzolkin and Avdeeva<sup>4</sup>. In addition to nickel, which is widely used, natural titanomagnetite was found to be effective at a temperature of 250° to 300° C. The same authors have also studied the influence of temperature on the toxic effect of a given concentration of a poison in the synthesis itself, this effect being, as would be expected, less as the working temperature is increased. With an iron-potash-alumina catalyst at 450°, the activity was depressed to 30 per cent. of its original value by 0.0001 per cent. of hydrogen sulphide in the reaction gases, whereas, at 500°, ten times this concentration of the poison only reduced the activity to 75 per cent. of its normal value. Similarly, phosphine is stated to be non-toxic above 510°, while, at a lower temperature, the depression in activity is reported to vary directly with the poison concentration and inversely with the temperature. The poisoning of ammonia catalysts by small concentrations of carbon monoxide or oxygen has been dealt with again by Epschstein and Upolovnikov<sup>5</sup>, using a titanomagnetite catalyst. As has been previously observed, subsequent treatment with pure gases restores the activity.

Loud<sup>6</sup> discusses measures for avoiding the excessive accumulation of diluents such as argon and methane in the circuit

gases. In place of blowing off the gas directly from time to time, the mixture is further compressed and circulated through auxiliary catalyst chambers at the higher pressure, whereby more ammonia is obtained, the gas being finally blown to waste. In a modification of the method<sup>7</sup>, a part of the gases from a primary circuit is allowed to pass, at the same pressure, to a secondary circuit containing a more active catalyst. In view of the argon content of atmospheric nitrogen and of the passage of the whole of the carbon monoxide in the impure hydrogen into methane, the adoption of the most effective procedure for dealing with the gradual accumulation of these is a factor of considerable importance in the economics of the synthesis.

## Nitric Acid and Nitrates

In previous reviews the use of multiple platinum or platinum-alloy nets for the oxidation of ammonia to nitric acid has been discussed, the general view being that, as normally operated, there is little advantage in employing more than one thickness of gauze. The subject has been brought up again by Handforth and Kirst<sup>8</sup>, who claim the use of 5 to 50 nets in series. It would be interesting to have comparative figures for the conversion under these conditions, since the number claimed is far greater than that examined in previous publications. A variation which certainly would seem to lead to mechanical stability has been described by E. A. Taylor<sup>9</sup>. The warp and the wool wires of the gauze are constructed, respectively, of a metal having a high mechanical strength, in the one case, and of a metal having a high catalytic activity, in the other. Suitable combinations are platinum and platinum-rhodium.

Base metal catalysts have continued to receive attention. Thus, Fogley<sup>10</sup> describes the preparation of cobalt-alumina in granular form. To this end, cobalt nitrate and aluminium nitrate are heated together. The mass is granulated, and the dust which remains is mixed with a subsequent batch. The preparation of cobalt oxide in a pure form for the oxidation of ammonia to nitric acid is treated by Bray<sup>11</sup>, in that the impurities in commercial cobalt are removed by dissolving this in nitric acid and adding sufficient alkali to precipitate about 5 per cent. of the cobalt, together with the impurities. The remaining cobalt is precipitated separately and is used as the oxide. Finally, an interesting discussion of the use of chromium oxide for the oxidation of ammonia has been contributed by Adadurov and Astroschtschenko<sup>12</sup>. With chromium oxide supported on silica, yields up to 90 per cent. were obtained at a temperature of 750° C. The catalyst is preferably stabilised by the addition of cobalt nitrate.

## Concentration by Enrichment

Of the specifications dealing with the conversion to nitric acid of the nitrous gases resulting from the above oxidation, those depending on concentration by enrichment are of special interest. This method of preparing concentrated nitric acid is facilitated by the use of oxygen in place of air for the ammonia oxidation reaction, by reason of the high concentration of the nitrogen oxides in the product. According to procedure suggested by Caro and Frank<sup>13</sup>, the dilute nitric acid resulting from primary condensation is mixed with the nitrogen peroxide obtained by cooling the reaction gas and sprayed at a temperature of 70° or over, and at a pressure of 20 atmospheres, into a tubular vessel in the presence of oxygen, when concentration by enrichment takes place normally.

<sup>1</sup> Atmospheric Nitrogen Corporation, Brit. Pat. 413,517.

<sup>2</sup> U.S. Pat. 1,919,216.

<sup>3</sup> U.S. Pat. 1,927,993.

<sup>4</sup> U.S. Pat. 1,936,936.

<sup>5</sup> U.S. Pat. 1,918,957.

<sup>6</sup> J. Appl. Chem. Russ., 1933, 6, 1029; ex Brit. Chem. Abs. B, 1934, 236.

<sup>7</sup> Brit. Pat. 405,450.

<sup>1</sup> THE CHEMICAL AGE, 1934, 30, 296.

<sup>2</sup> *Ibid.*, 1934, 30, 303.

<sup>3</sup> "Chim. et Ind.," 1934, 32, 759.

<sup>4</sup> "J. Chem. Ind. Russ.," 1933, 10, 32, 37; ex Brit. Chem. Abs., B, 1934, 45, 959.

<sup>5</sup> *Ibid.*, 1934, 10, 37; ex Brit. Chem. Abs., B, 1934, 623.

<sup>6</sup> U.S. Pat. 1,938,598.

The absorption of nitrous gases by treatment with sulphuric acid of varying strengths is dealt with in Brit. Pat. No. 413,828. The gases are dried by passage through sulphuric acid having a concentration exceeding 82 per cent., and are then absorbed in an acid of 72-85 per cent. strength. The preparation, from nitrous gases, of sodium nitrite containing as little nitrate as possible has been discussed by Handforth<sup>14</sup>. The products obtained during the absorption of these gases by alkalis depend not only on the ratio of nitrogen peroxide to nitric oxide in the gas but, to a greater degree, on the pH value of the alkali. A high value of this leads to a greater proportion of nitrite; and it has been found more convenient and economical to use sodium carbonate in place of the hydroxide. Fresh sodium carbonate is added from time to time during the adsorption process.

Improved procedure for the concentration of nitric acid is described by Handforth, Kirst and Zeisberg<sup>15</sup>. A vertical concentration tower is employed in conjunction with a series of heated horizontal tubes, in which the nitric acid vapour evolved flows in countercurrent to the liquid concentrated. For details of other proposed variants in the operation of nitric acid concentration towers involving the use of sulphuric acid, reference may be made to U.S. Patents 1,895,012 and 1,896,287. It may also be noted that Kirst<sup>16</sup> has proposed, for the concentration of nitric acid in metallic vessels, that the wall, which may be of chromium steel, should be maintained at a temperature above the boiling point of the acid, in such a way that it does not come into effective contact with the liquid. Finally, the simultaneous production of sulphuric and nitric acid has been considered. Ionas and Merkin<sup>17</sup> find that the presence of sulphur dioxide in ammonia does not impede the catalytic oxidation of either component by a platinum catalyst, although the temperature and time of contact required are very different, and this result has little bearing on the possible preparation of sulphuric acid. For the simultaneous production of the two acids, however, a specification of Kachkaroff and Matignon<sup>18</sup> may be consulted. The method involved is a variation of the ordinary sulphuric acid chamber process.

### Manufacture of Nitrates

Various patents covering the manufacture of nitrates have been published during the year. Thus, methods involving the use of zeolites in the preparation of sodium or potassium nitrate have been claimed by the Norsk Hydro-Elektrisk Kvaestofabrik<sup>19</sup>. For instance, on passing a solution of calcium nitrate through a zeolite mass containing replaceable sodium, sodium nitrate is produced. The calcium zeolite is subsequently revived in the usual way by the action of sodium chloride. Conditions for the economic preparation of potassium nitrate from nitric acid and potassium sulphate in solution have been worked out by the Kali-Forschungs-Anstalt<sup>20</sup>. The concentrations of the salts are adjusted in such a way that, after the separation of potassium nitrate crystals, the mother liquor is also saturated with respect to the double salt of potassium nitrate and potassium hydrogen sulphate. The manufacture of mixed nitrates in a form suitable for scattering in the course as their use as fertilisers is described by Lüscher<sup>21</sup>. Thus, the double calcium-ammonium or calcium-potassium nitrate is crystallised out at or near the boiling point of the solution, and the crystals obtained are mixed with a little calcium carbonate. A further procedure for preventing the caking of nitrates has been suggested by Wyler<sup>22</sup>, who proposes the addition of a small percentage of water glass to ammonium nitrate.

According to the proposals of Rosenstein<sup>23</sup>, alkali nitrates are prepared by extracting with liquid ammonia the reaction product obtained by treating sodium or potassium chloride with a liquid oxide of nitrogen. It has also been proposed to utilise the heat of crystallisation of ammonium nitrate in

the production of the dry salt<sup>24</sup>. The hot solution of the salt is concentrated to incipient crystallisation by evaporation at a reduced pressure. When crystallisation sets in, the heat evolved evaporates water and tends to produce a dry salt. Finally, the conditions which cause hygroscopicity in ammonium nitrate mixed with calcium carbonate have been discussed by Guyer and Schütze<sup>25</sup>.

### Other Ammonium Salts

As in previous years, a number of patents have been published dealing with minor variations in the production of ammonium sulphate by the ordinary neutralisation process. For instance, Atwater<sup>26</sup> describes procedure in which ammonia gas is passed first through a saturation vessel containing ammonium sulphate together with a little sulphuric acid, and subsequently through a second saturator containing aqueous sulphuric acid alone. The small crystals formed in the second saturator are returned to the first tank, where they grow into large crystals. Among the waste products which have been suggested as raw material for the manufacture of ammonium sulphate, the sulphuric acid sludge resulting from the refining of petroleum has been proposed by the Bataafsche Petroleum Maatsch.<sup>27</sup> Other specifications dealing with the production of ammonium sulphate are those of van Ackeren<sup>28</sup> which refers to the drying of this salt, and that of Cashmore, Clifford and Imperial Chemical Industries<sup>29</sup>, which relates to the production of potassium ammonium sulphate by a cyclic solution process.

An interesting method of separating ammonium chloride from alkali nitrates is described by the Interessen Gemeinschaft<sup>30</sup>. If a solution, which is saturated both with respect to ammonium chloride and to potassium nitrate, is agitated with a small percentage of oleic acid, the froth is found to contain a considerable proportion of the ammonium chloride. This froth is separated and broken down, and the oleic acid is re-used. Cole<sup>31</sup> has protected a variation for the recovery of ammonium chloride from Solvay liquors, which contain sodium chloride, ammonium carbonate and ammonium chloride. Calcium chloride is added to precipitate the carbonate, then carbon dioxide is passed to precipitate the calcium. Free ammonia is removed by steam and, finally, the solution is evaporated to a stage just above that at which sodium chloride separates, when it is cooled for the crystallisation of the ammonium chloride.

### Cyanides and Cyanamides

The use of sulphur dioxide as a stabiliser in the preparation of hydrocyanic acid has been protected by the Roessler and Hasslacher Chemical Co.<sup>32</sup>. The hydrocyanic acid is produced by allowing an alkali cyanide to react with an acid in the presence of a little sodium sulphite, which is previously mixed with the cyanide. For the formation of sodium cyanide by the action of sodium on calcium cyanamide, reference may be made to a specification of Pranke<sup>33</sup>. The crude cyanamide is heated with sodium to a temperature above 900° C. Mention may be made of a method of rapidly drying sodium cyanide with minimum decomposition, also due to Pranke<sup>34</sup>. The cyanide is allowed to fall on to a hot metallic surface, from which the solid is immediately removed. Finally, the production of calcium cyanamide in stable granular form is treated in a patent of the American Cyanamid Co. and Cox<sup>35</sup>. A little water is added to the crude products in order to react with the calcium carbide, followed by a small quantity of nitric acid in order to form calcium nitrate, which is stated to act as a binder. The mass is dried at a temperature above 100° C. This stabilising action of calcium nitrate should be compared with its effect in ammonium nitrate mixtures, described earlier in the present review, in which this component was stated to induce hygroscopicity.

<sup>14</sup> U.S. Pat. 1,903,815.

<sup>15</sup> U.S. Pat. 1,922,278, 1,922,289 and 1,928,749.

<sup>16</sup> U.S. Pat. 1,901,875.

<sup>17</sup> "Trans. Sci. Inst. Fertilisers Moscow," 1032, 68; ex. Brit. Chem. Abs. B, 1934, 272.

<sup>18</sup> Brit. Pat. 202,229.

<sup>19</sup> Brit. Pat. 407,311 and 407,313.

<sup>20</sup> Brit. Pat. 410,774.

<sup>21</sup> U.S. Pat. 1,926,492.

<sup>22</sup> U.S. Pat. 1,932,434.

<sup>23</sup> U.S. Pat. 1,932,939.

<sup>24</sup> Kostner, Brit. Pat. 403,240.

<sup>25</sup> "Z. angew. Chem.," 1933, 46, 763.

<sup>26</sup> U.S. Pat. 1,917,915.

<sup>27</sup> Brit. Pat. 495,556.

<sup>28</sup> U.S. Pat. 1,936,866.

<sup>29</sup> Brit. Pat. 411,820.

<sup>30</sup> Brit. Pat. 411,587.

<sup>31</sup> U.S. Pat. 1,913,372.

<sup>32</sup> Brit. Pat. 401,351.

<sup>33</sup> U.S. Pat. 1,995,304.

<sup>34</sup> Brit. Pat. 411,177.

<sup>35</sup> Brit. Pat. 401,788.



# Analytical Chemistry in 1934

By C. Ainsworth Mitchell, D.Sc., F.I.C.

**A**LTHOUGH analysis is primarily a means to an end, the investigation required to establish a new procedure frequently adds much to our knowledge of chemistry. In a short survey of the progress of analytical chemistry, however, it is not possible to refer to more than a few of the numerous contributions that have been made during the year.

## Physical Methods

A symposium on the analytical applications of quantitative spectroscopy was the subject of a meeting of the Society of Public Analysts on November 7. Dr. J. J. Fox gave an outline of the development of spectroscopy; Mr. F. Twyman discussed the construction of the different types of spectroscopic instruments; Dr. S. Judd Lewis described his ratio-quantitative system of determining elements by spectrographic comparison with standard ratio powders; and Mr. D. M. Smith gave an account of the application of spectroscopic methods to the examination of metals. Petry ("Ind. Eng. Chem.," Anal. Ed., 1934, 6, 434) has shown that comparison with standard spectrographs affords an accurate means of determining traces of fluorides in water.

Among new applications of the quartz mercury-vapour lamp are its use for differentiating English from Italian ryegrass seed (Francois, "Ann. Falsificat.," 1934, 26, 34); for distinguishing between virgin and refined olive oil (Cocking and Crews, "Pharm. J.," 1934, 133, 86); and for detecting up to 10 per cent. of rhabonic rhuibarb in Chinese rhuibarb (Wallis and Withell, "Pharm. J.," 1934, 133, 90). Grant ("Proc. Techn. Sec. Papermakers' Assoc.," 1934, 4, 335) has critically reviewed the methods of applying fading tests by means of the ultra-violet lamp. He has also ("Analyst," 1934, 59, 749), devised ultra-violet tests for the identification of imitation water-marks in paper, and for measuring the degree of water-resistance of paper ("J.S.C.I.," 1934, 53, 349). An ingenious fluorescence method of developing finger-prints on a multi-coloured background was published by Brose ("Analyst," 1934, 59, 25).

A photometric process of determining traces of lead sulphide in suspension has been worked out by Samuel and Sholkey ("J.A.O.C.," 1934, 17, 141). It is sensitive to 0.013 mg. of lead, but bismuth, mercury or tin must not be present. Hirano ("J. Soc. Chem. Ind. Japan," 1934, 37, 177B) uses a photometric method for determining small amounts of iodide.

There has been much discussion during the year as to the significance of the freezing point as a criterion of the purity of milk; the generally accepted view is that the freezing-point method affords valuable information, but should not be regarded as the sole test to be applied to milk. Tabulated observations extending to some 3,000 samples of milk have been published by Elsdon and Stubbs ("Analyst," 1934, 59, 146, 585); the same chemists have also studied the technique for obtaining comparable results by the Hortvet method (*loc. cit.*).

A simple form of micro-apparatus for the determination of molecular weights by the vapour-density method has been devised by Colson ("Analyst," 1934, 59, 529). The use of dioxan is recommended as a solvent in the determination of molecular weights by the cryoscopic method (Oxford, "Biochem. J.," 1934, 28, 1,325).

An apparatus for determining the viscosity of tar has been designed (Lee, "J.S.C.I.," 1934, 53, 60T), in which the results obtained under a driving pressure are calculated into absolute values.

## Gas Analysis

A rapid colorimetric method for the detection and determination of small quantities of oxygen in gases has been based by Ambler ("Analyst," 1934, 59, 14) on Pfeiffer's method of comparing the colour of the alkaline pyrogallol, after absorption of oxygen, with standard iodine solutions. Ambler (*ibid.*, 593) has also devised a precision method for the absorption of oxygen by phosphorus, and for the determination of impurities in commercial carbon monoxide (*ibid.*, 809). Refer-

ence may also be made to a method of determining butadiene in gases (Tropsch and Mattos, "Ind. Eng. Chem.," Anal. Ed., 1934, 6, 104).

## Inorganic Analysis

Several new indicators have been suggested, including nitroazine yellow, which changes to deep blue between pH 6.4 and 6.8 (Wenker, "Ind. Eng. Chem.," 1934, 26, 350), and colloidal selenium as an indicator for the bromate titration of arsenic (Szebelledy and Schick, "Z. anal. Chem.," 1934, 97, 186). The use of complex ions as indicators in the analysis of metals was discussed by Ubbelohde ("Analyst," 1934, 59, 339). Among new colour reactions which may be used as qualitative tests are one for nitrites and nitrates depending on the formation of resorufin (Eichler, "Z. anal. Chem.," 1934, 96, 17), and another embodying the use of Magdala red for the detection of traces of nitrites in water (Eichler, *ibid.*, 99). A colour test for ammonia, which is much less sensitive than the Nessler reaction, has been based on the blue colour obtained with hypobromite and thymol (Lapin and Heim, "Z. anal. Chem.," 1934, 98, 236).

Increasing use is being made of organic reagents for the precipitation of inorganic ions. A method of separating iron, aluminium and chromium from bivalent metals by precipitation with ammonium benzoate has been devised by Kolthoff and his co-workers ("J. Amer. Chem. Soc.," 1934, 56, 812). Anthranilic acid precipitates manganese quantitatively (Funk and Demmel, "Z. anal. Chem.," 1934, 96, 385); 2,3-diaminophenazine is used as a reagent for cupric and mercuric ions (Favolini, "Ann. Chim. anal.," 1934, 16, 23);  $\alpha$ -nitro- $\beta$ -naphthol for the determination of cobalt and palladium (Mayr, "Z. anal. Chem.," 1934, 98, 402); nitron as a precipitant for nitrates (Heck *et al.*, "Analyst," 1934, 59, 16); and resorufin for precipitating a number of elements (Eichler, "Z. anal. Chem.," 1934, 96, 22). Barr and Thorogood ("Analyst," 1934, 59, 378) have shown that a zirconium alizarine reagent may be used for the accurate colorimetric determination of traces of fluorides in water. The clinical significance of such traces of fluorine has been discussed by Ainsworth ("Analyst," 1934, 59, 380).

## Quantitative Analysis of Copper

A scheme for the quantitative analysis of copper has been based by McIlroy ("Analyst," 1934, 59, 103) on its precipitation with  $\beta$ -phenylenediamine, and a method for determining iron by precipitation with butylphenylarsonic acid has been devised by Craig and Chandler ("J. Amer. Chem. Soc.," 1934, 56, 1,278). Haddock ("Analyst," 1934, 59, 163) has worked out a method for determining traces of bismuth in the absence of lead and thallium. It consists in a colorimetric estimation of the bismuth after separation by means of diphenylthiocarbazone ("Analyst," 1934, 59, 163). The same organic reagent (diphenylthiocarbazone) has been used by Roche Lynch, Slater and Osler for the separation of lead from bone after wet oxidation. The lead is subsequently determined colorimetrically as sulphide. Special precautions must be taken to exclude bismuth ("Analyst," 1934, 59, 787). For the determination of traces of lead in the presence of traces of bismuth, Hamence ("Analyst," 1934, 59, 274) precipitates both metals as sulphides, and then separates them by the pyridine thiocyanate process. Berry ("Analyst," 1934, 59, 736) has found that chloramine-T may be used in conjunction with potassium iodide as an oxidising agent in the direct titration of various oxidisable substances, notably thalious salts.

Schoeller and his collaborators have continued their revolutionising work on tantalum, niobium and their associates. They have shown that manganese may be quantitatively precipitated from tartrate solution by means of tannin and ammonia; they have also devised a new method for the analysis of tantalite ("Analyst," 1934, 59, 463, 667) based on their previous work.

A rapid test of the thickness of tin coatings on steel has been published by Clarke ("Analyst," 1934, 59, 525); it consists in dissolving the tin in a solution of antimony trichloride in hydrochloric acid, and determining the loss in weight of the sample. For the analysis of magnesium alloys Nickolls ("Analyst," 1934, 59, 16) has devised a method in which aluminium is precipitated by alkali sulphide as hydroxide, whilst the magnesium is dissolved as hydrosulphide. For the detection and identification of metal particles in manufactured products Lockwood ("Analyst," 1934, 59, 812) has devised systematic tests based upon the action of silver nitrate solution as described by Ward (*ibid.*, 1933, 53, 28). In a critical investigation of the various methods of determining free silica in coal-measure rocks, Shaw ("Analyst," 1934, 59, 446) has shown that "rational analysis" (removal from the sample of all minerals except quartz) gives the best results, but his contention has not been generally accepted by mineralogists.

Several papers have been published on electrolytic methods, including one on simplifications in the methods of separating metals by means of graded potentials (Lindsey and Sand, "Analyst," 1934, 59, 328), and a description of an electrolytic method of separating germanium in the presence of arsenic (Coase, "Analyst," 1934, 59, 748).

### Organic Analysis

Among the methods applicable to groups of organic compounds is one for the identification of aldehydes and ketones, based on their condensation with thiohydrazides to form substituted thiohydrazines identifiable by their colour reactions and m.p.s. (Lacourt, "Bull. Soc. Chim. Belg.," 1934, 43, 206). A quantitative reagent for carbonyl compounds has been found in 2,4-dinitrophenyl-hydrazine (Houghton, "Amer. J. Pharm.," 1934, 106, 62), and methoxyacetic anhydride has been found suitable for the determination of hydroxyl groups (Hill, "J. Amer. Chem. Soc.," 1934, 56, 993). A micro

method for methoxyl and ethoxyl groups devised by Nanji ("Analyst," 1934, 59, 96) consists in the formation of alkyl iodide and oxidation of this to iodate, which is determined iodometrically. Fourey ("J. Pharm. Chem.," 1934, 126, 116) has shown that quinone may be used as a reagent for amines. Griffiths and Hilditch ("J.S.C.I.," 1934, 53, 75) have studied the transformation of oleic into elaidic acid as an aid to the analysis of mixtures of oleic, linolic and linolenic acids, and have shown ("Analyst," 1934, 59, 312) that the reaction may be utilised as a semi-quantitative method.

During the year a further Report (No. 11) of the Essential Oils Sub-Committee of the Society of Public Analysts Committee on Uniformity of Methods of Analysis has been published. It deals with the determination of aldehydes other than citronellal. An important contribution to the analysis of starch products has been made by Chinoy, Edwards and Nanji ("Analyst," 1934, 59, 673) who found that starch iodide can be precipitated as a definite entity, dried without decomposition at 100° C., and weighed.

### Foods and Drugs

Space does not permit of detailed reference to the numerous methods of analysing various foods and drugs, but reference must be made to the important paper published by Hughes and Maunsell ("Analyst," 1934, 59, 231) on the analysis of fruit and fruit products, in which use is made of statistical methods of calculation; also to the cognate paper by Hinton (*ibid.*, 248) describing a method of examining fruits and jams by means of lead precipitation. Many of the papers in the biochemical journals contain analytical information of interest to chemists in general, and in this connection the discussion on the chemical evaluation of vitamins, to which Bacharach, Lester Smith, Norman Evers, and Bennett contributed ("Analyst," 1934, 59, 70), gives a valuable survey of the chemical as contrasted with the biological methods of examining these accessory food factors.

## The Work of the Public Analyst

By ERIC VOELCKER, A.R.C.S., F.I.C. (Consulting Chemist and Public Analyst)

THE ever vexed question of added water in milk is one that has been prominent in the minds of the public analyst during 1934, and the admirable work of Elsdon and Stubbs on the freezing point of milk has been continued and extended. In a paper recently published by them ("Analyst," 1934, 59, 146) on the examination of 1,000 samples of milk, including "appeal to cow" samples, they have shown that the average freezing point of genuine milk is  $-0.544^{\circ}$  with extremes of  $-0.529^{\circ}$  and  $-0.563^{\circ}$ , when using the Hortvet method without any corrections. Some workers dispute these figures and put forward wider limits, but I think it is true to say that in those cases the Hortvet technique has not been strictly followed. Elsdon has further shown that the figure for cows' colostrum varies from  $-0.541^{\circ}$  to  $-0.596^{\circ}$ . Elsdon and Stubbs ("Journ. Soc. Chem. Ind.," 1931, 50, 135) explain that the osmotic pressure of cows' milk is a constant quantity that varies very little from animal to animal, and it is reasonable to assume that the osmotic pressure of the milk will bear some constant relationship to the blood of the animal. It is known that the sera of blood and milk have similar freezing points, and that these are constant for different animals of the same species. From this can be deduced that the osmotic pressure of milk, and therefore the freezing point, is constant.

### Analysis of Milk

The amount of solids-not-fat in a milk may, under some circumstances, be affected without alteration in the depression of the freezing point. This is brought about by the fact that an alteration in the amount of one of the ingredients secreted in the milk will alter the osmotic pressure, so that, in order to keep the latter constant, some change is necessary in one or more of the ingredients. But the various constituents present in the serum do not, weight for weight, affect the osmotic pressure to the same extent, hence there will be an alteration in the amount of solids-not-fat. If this be so,

it gives a complete explanation as to why, in a normal milk, the Veith ratio (lactose : protein : ash = 13 : 9 : 2) is a constant, whilst in an abnormal milk, where the solids-not-fat are greatly reduced, the ratio will be very considerably reduced.

Bearing these facts in mind, it is apparent that the great value of the freezing point test for milk, as compared with other tests, is that the figure obtained appears to be quite independent of that of the non-fatty solids. In other words, a normal milk is not one having a non-fatty solid content of 8.5-9.0 per cent., but one having a freezing point depression of about  $0.54^{\circ}$ . The main disadvantage of this test is that it is only applicable to fresh or reasonably fresh milk. Work is, however, being carried on in an endeavour to overcome this difficulty after souring has taken place. The value of the freezing point test as a confirmation in doubtful cases of the watering of milk has received further verification at the hands of several workers and is one that may be considered of real value to the public analyst in framing his report.

It may be convenient to point out here that nitrates do not occur naturally in cows' milk, even when food containing nitrates has been given. The finding of traces of nitrates, either by the diphenylamine or diphenylbenzidine tests, is an indication that water has been added—as most natural waters contain nitrates in varying amount. This point is useful in testing a sample of supposed reconstituted milk which relies on the emulsification of dried milk powder with water, and, in the case of reconstituted cream, with the addition of butter.

### Herbs and Spices

Another subject of interest is the work on herbs and spices by Sage and Fleck ("Analyst," 1934, 59, 614) from the point of view that the volatile oil which these herbs contain may be the only constituent of any value—each containing its own peculiar volatile oil on which it depends for suitability for

flavouring purposes. Tables, which are of value to the public analyst, of the volatile oil content are given for a number of the more common types of herbs and spices. The common herb, mint, has been in several cases found to be adulterated with leaves of ailanthus, or "Tree of Heaven" (*Ailanthus glandulosa*), a Chinese tree which has been acclimatised in Europe. The leaf does not possess the properties of mint, sage, marjoram, or thyme (which belong to the same order of herbs as mint), and is, in fact, quite useless. Fresh mint should contain 0.8 to 1.4 per cent. of volatile oil which is absent from ailanthus.

### Estimation of Vitamins

The subject of vitamins has provoked much discussion and Bacharach and Smith ("Analyst," 1934, 59, 70) summarise the present knowledge of the chemical composition of the various vitamins and also make observations on the analytical procedure that must be adopted for their determination. They consider that vitamin A is probably a partial oxidation product of half the carotene molecule and has the formula  $C_{26}H_{44}O$ . Vitamin B appears to have the formula of either  $C_{12}H_{20}O_2$ ,  $N_2S$  or  $C_{12}H_{18}O_2$ ,  $N_2S$ —it is a basic substance, though not a primary amine.  $B_2$  has the probable formula  $C_{17}H_{22}O_4N_4$  and is identical with a crystalline orange brown fluorescent pigment, lactoflavine. Vitamin C is identified with ascorbic acid (3-keto-1-gulonolactone) having the formula  $C_6H_8O_6$ . Vitamin D is an isomer of ergosterol whose formula is  $C_{28}H_{44}O$ . Of vitamin E little can be said of its composition except that it appears to be closely related with the sterols as is common with many substances of physiological activity.

The chemical reactions have been investigated as bases for the determination and estimation of the vitamins. For vitamin E there is no chemical test available, also for vitamin D, excepting in the case of calciferol (which is not, strictly speaking, a natural product). The blue test for vitamin A has received a further modification well known as the Carr-Price test, in the replacement of arsenic trichloride by antimony trichloride. This brings about a slight reduction in the sensitiveness of the test, but a great increase in its usefulness. Vitamin C is a strongly reducing substance and is therefore capable of being titrated with a number of oxidising reagents such as iodine and certain dyestuffs, notably by the method of Tillman, using 2,6-dichlorophenol-indophenol. This latter method is shown by Bennett ("Analyst," 1934, 59, 91) to be the basis of routine control of the vitamin C concentration of citrus fruit juices. It has also been found that the vitamin C may disappear altogether from citrus juices when preserved or sterilised in so little a time as 12 days, if access to air be allowed.

### Uses of Ultra-Violet Light

- In the field of filtered ultra-violet light and its application work has been done by Radley and Grant. In a paper by Baetslé and De Wever ("Annales de la Brasserie," 1934, 138) on "Woods light and its application in the Brewery" many particulars are given. For instance, they say (1) that from any source of light containing ultra-violet light rays the ordinary visible light (4,000-8,000 angstroms) must be filtered off; (2) that various screens have been tried as filters, *i.e.*, solutions of nitrosodimethylaniline and methyl violet 4R, but these were not efficient enough; (3) wood was successful with quartz on which nickel oxide was deposited; and (4) light from a quartz mercury vapour lamp filtered through a wood screen, ultra-violet light of about the useful figure of 3,650 Å was obtained. On examining sugars they found pure glucose to give a bluish grey fluorescence; 2 per cent. dextrin started it going yellow, and 3 per cent. definitely so. Invert sugar gave a very fine blue fluorescence, anhydrous cane sugar a violet, becoming blue as moisture comes in. Beet molasses gave grey, sometimes very pale, whilst cane molasses gave a chestnut or maroon fluorescence. Continuing their work to barley, they found that the corns varied in given samples, some violet and some yellowish to brown. On separating the corns according to colour, they found the yellowish ones always to be definitely higher in nitrogen. This occurred with four distinct varieties of barley.

Use of ultra-violet light is now employed in practically all branches of chemistry, be it for forensic work or for a

mere routine sorting-out test for impurities in some types of foodstuffs. The general use of the spectroscope has been extended and quantitative determinations are now practicable, and can be made on substances of almost any type with great accuracy. Actually, spectroscopic determinations are not of wide interest for major components, since closer results are normally obtainable by chemical methods; but for small quantities, and more especially very small quantities, spectroscopic methods are often much more accurate and reliable than chemical methods. It also appears to be a general tendency at the moment to inflict on the analytical chemist methods of analysis that entail the use of most expensive physical apparatus. All are exceedingly useful in their way, but some can hardly be considered as standard equipment of the laboratory of the public analyst. Those who are fortunate or wealthy enough to possess all will find themselves in a somewhat unique position.

## The Ceramic Society

### Valuable Contributions During 1934

THE activities of the Ceramic Society have resulted in the publication of many valuable data obtained through planned researches bearing on problems encountered by members of the Society's three sections—pottery, refractory materials and building materials. The pottery section meetings were, as usual, held at the Stoke-on-Trent headquarters of the Society, and the other two sections arranged joint meetings at Llandudno and in London.

One important paper by Clews and Green on "The Significance of Permeability to Gases in Relation to the Texture and Industrial Usage of Refractory Materials," which first appeared in B.R.R.A. Bulletin No. 27 in 1932, is reprinted in the Society's Transactions by permission of the council of the British Refractories Research Association, together with an interesting discussion to which it gave rise among members of the Society. In like manner, S. R. Hind's paper on "The Carder Tunnel Oven," B.R.R.A. Bulletin No. 21, originally issued in September, 1929, was, by permission, reprinted in the current year's Transactions. Another useful paper, by C. S. Woodward, describing "Modern Methods of Heat Insulation," was presented at Stoke, and evoked a number of comments and questions. In a paper on "Rapid Drying of General Earthenware," T. Simpson deals with a subject of great practical importance to all manufacturers, and indicates how considerable economy in fuel and water may be effected. On the same general subject, H. H. Macey discussed "The Principles Underlying the Drying of Clay."

W. J. Rees and his collaborators at Sheffield University contribute several important papers dealing with various furnace linings. In a paper on "Alkaline Casting Slip," Dr. Webb discusses the results obtained by himself and co-operating students in the pottery laboratory at Stoke. Dr. Felix Singer's contribution on "Fuel Economy in the Ceramic Industry" (parts I and II), embodies a comprehensive survey of the types of kilns, etc., actually employed in the different branches of the ceramic industries. Cyril Edwards contributes "Notes on the Testing of Refractory Materials," based on his experiences with the Woodall-Duckham companies.

Other important contributions include Mr. Threlfall's thoughtful paper on "Porosity" (a valuable discussion), Mr. Sarjant's account of "Some Factors affecting Furnace Heating Practice—II," recording further data of practical value, and a paper by Mr. Box, describing "Electro-Magnetic Separators and Extractors for Treating all Classes of Pottery, Refractory Materials and Builders' Materials." The popular question box, involving the discussion of specific practical problems, encountered by members was not overlooked.

At the annual general meeting at Stoke, a presentation was made to Dr. J. W. and Mrs. Mellor, of a morocco-bound copy of "Uncle Joe's Nonsense—A Medley of Fun and Philosophy," produced (inclusive of numerous illustrations) by Dr. Mellor himself in lighter vein—published by the Society to show in some measure the members' appreciation of his services to the Society for many years as hon. general secretary and otherwise.

# New Fields in Organic Chemistry

By K. F. Armstrong

**W**HILE important discoveries in all branches of organic chemistry have been made in the period under review, the most important activity has been shown in the investigation of natural products. The organic chemist is successfully attacking the problems arising from the work of the biologists, and the elucidation of the chemical fine structure of biologically important substances provides a firm material basis as a necessary prelude to the interpretation of their physiological rôle.

It is desirable to stress that owing to the fact that many of these active substances are only present in minimal concentration in difficultly obtainable biological material, many of these investigations have been necessarily, but successfully, carried out using very small quantities of materials, only a few hundred milligrams being available in some instances. Micro-analysis and the ability to work with small quantities are essential to the modern organic chemist, and a plea may be advanced that more attention should be given to accustoming students to working with small quantities, not only because the problems with which they will later be confronted demand this ability, but also on the grounds that a saving in running costs of a laboratory could be effected.

## New Knowledge of the Hormones

Much progress has been made in the sex hormones related to the sterol group, which are now clearly to be regarded as based on the same fused-ring system as the sterols, but have the side chain broken down. The structure of oestrin, the oestrus-producing hormone of the female is now well established: closely connected with oestrin in its action is the hormone secreted by the corpus luteum, and this hormone has now been obtained artificially by Butenandt by degradation of the sterol stigmasterol. Ruzicka has similarly obtained artificially the male testicular hormone, androsterone, by degradation of dihydrocholesterol. It will probably, therefore, soon be possible for these important hormones to be available in chemically pure crystalline form for clinical use. Valuable light will be thrown on sterol metabolism by the establishment of the fine structure of these compounds, and judging by the activity in this field the day cannot be distant when the hormones will be synthesised completely as well as being obtainable by degradation.

## Plant Products

Another series of compounds with important physiological properties are the so-called cardiac aglucones which have a specific heart action, of which the best known are digitalin and strophanthin. The discovery by Tschesche and later by Jacobs that they also yield the same fused-ring hydrocarbon on selenium dehydrogenation as is obtained from the sterols, has given fresh impetus to their study, and now that it is found that one of them, uzarigenin, can be degraded to a known bile acid derivative, earlier work can be reinterpreted to give a satisfactory formulation of this group.

Kögl, in a work which is a classic of microchemistry, has elucidated with some degree of certainty the formula of auxine, the hormone which is found in the tips of the growing shoots of plants and causes an elongation of the cell at the growing point. An astonishing result is that an identical growth promoting power to that of auxine itself is caused by  $\beta$ -indolyl acetic acid, a product of tryptophane metabolism in animals, bacteria and fungi. This shared specificity by such differently constituted substances is remarkable.

In sugar chemistry there is an academic satisfaction in the synthesis by Austin and Humoller of the two last missing members of the sixteen aldohexoses predicted by Emil Fischer, and marks the close of a chapter. Problems in the future are in the polysaccharides, whose structures are now beginning to yield to the attack. An urgent problem is the structure of the specific polysaccharides of bacteria, important in immunology, but in them the difficulties of obtaining material is supplemented by the difficulties of working with small quantities in sugar chemistry.

The demonstration that vitamin C, ascorbic acid, is a sugar derivative has given new interests to the problem of the interconversion of sugar derivatives in the plant, and such pro-

blems and studies of the metabolism of the sugars will continue to receive attention. Apart from this the preparation of new examples of countless derivatives of the simple sugars hardly merits the amount of attention which it is receiving. Of the vitamins, vitamin B<sub>2</sub> at the hands of Kuhn has had its structure very probably ascertained, and the brilliant synthesis of the degradation product obtained by the action of light, known as lumi-lactoflavin, is an important step towards the synthesis of the vitamin itself. Sufficient vitamin B<sub>1</sub> has been accumulated for a serious attempt to determine its structure to be begun, while preliminary experiments on vitamin E suggest that it may be one more example of the type of fused-ring compound related to the sterols.

## A New Class of Coloured Compound

A discovery of theoretical importance is the new class of coloured compounds discovered by Linstead called the phthalocyanines, formed on heating of phthalonitrile or other suitable compounds with metals. The structure responsible for their colour and special properties is similar to that found in the porphyrins, which, and whose derivatives chlorophyll and the blood pigment, are so widely represented in nature. They are very stable, their metallic derivatives, for example, subliming unchanged at 600°, which suggests valuable technical applications, and they are already being developed as light and reagent fast pigments which may prove valuable for printing inks, etc. The phthalocyanines as synthetic analogues of the porphyrins may lead to a better understanding of the properties of these compounds themselves. The fine structure of chlorophyll is approaching a solution which will satisfy critics in the field, and it is to be hoped that this knowledge will be useful in a renewed attack on the problem of photosynthesis. In this connection the separation of the heavy isotope of oxygen, O<sub>18</sub>, is urgently required.

Synthetic researches by Robinson in the new epindoline group of colouring matters which are isomeric with indigo, are of theoretical interest as finally disposing of an alternative indigo formula.

A good example of the application of simple physical principles to the solution of an organic chemical problem emerges from the work of Ziegler on the formation of large rings. Ordinary conditions for forming cyclic ketones containing many carbons in the ring give but small yields as Ruzicka showed, owing to the majority of the reacting molecules polymerising to long chain compounds. Very good yields can, however, be obtained by arranging the reaction to take place in extremely dilute solution so as to obtain intramolecular, as opposed to intermolecular condensation. By this principle among other syntheses one may easily obtain racemic muscone, an optically active muscone being the principle of musk, and the cyclic lactone ambrettolide of musk seed oil.

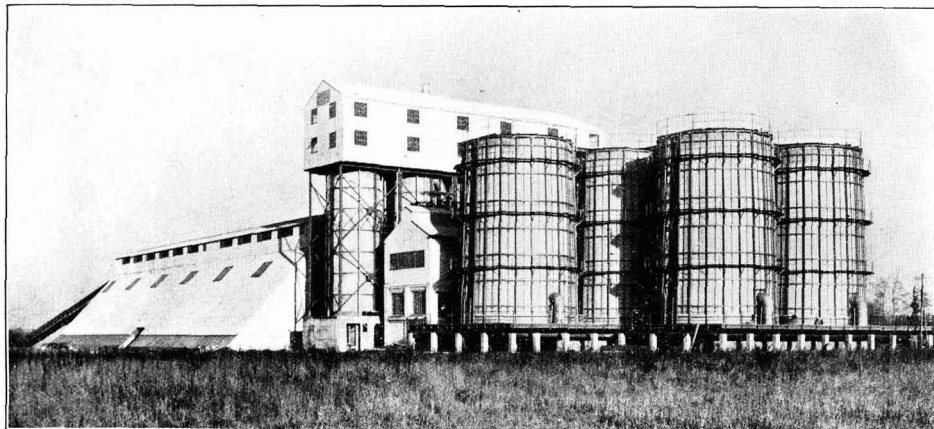
## Chromatographic Adsorption

Finally, attention may be directed to chromatographic adsorption, a new method of adsorption introduced mainly by Winterstein, which appears to be a very useful accessory method for the purification of solids. It differs from ordinary adsorption methods in that material adsorbed from solution by filtering through a vertical column, packed with a suitable medium, is developed by being driven down the column by washing with fresh solvent and is stratified into layers according to the different ease of adsorption of the components of the mixture, which can be separated mechanically and eluted. Solids which form mixed-crystals and cannot be distilled, are thus separable, and without this method the remarkable development of carotenoid chemistry would not have been possible. It can be applied to colourless as well as coloured compounds, especially when some other property, such as fluorescence in ultra-violet light, or a colour reaction or physiological activity can be used as a guide to the success of the separation.

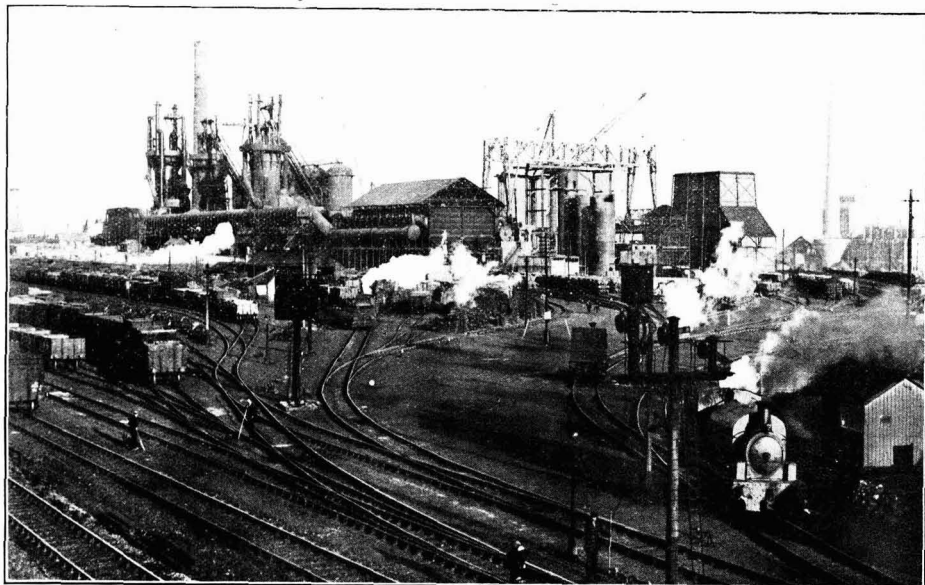
## Some Events of the Year in Pictures

In these pages we reproduce illustrations of some events which have made 1934 memorable. Below are pictures of two notable industrial developments in this country; overleaf are portraits of leaders in their respective spheres of industrial

chemistry and metallurgical science who have passed away during the year. Academic progress is typified by the three groups in pages 592 and 593 of recipients of high honours at Leeds, Birmingham and Madrid.



General view of the sulphuric acid plant at the fertiliser factory of Fison, Packard and Prentice, Ltd., Ipswich, which was opened during the year, and is claimed to be the most modern and best equipped fertiliser plant in Europe. The acid plant was designed and erected by the Mills Packard Construction Co., Ltd., a company associated with Fison, Packard and Prentice, Ltd. The unit comprises a store for pyrites capable of holding 100 tons; a burner house where sulphur gases are obtained by roasting the pyrites in four Herreshoff furnaces, each with a capacity of 12 tons of pyrites per day; and chambers and towers for converting the gases to sulphuric acid. The capacity of the plant is 26,000 tons of acid per annum.



Rapid progress has been made in the development of the scheme of Stewarts and Lloyds, Ltd., for the treatment of iron ore deposits in Northamptonshire and the construction, at Corby, of works for the production of Bessemer steel. The photograph shows the blast furnaces on the left, the new ore bridge and blast furnace gas cleaning plant and the by-product coking plant in course of erection.





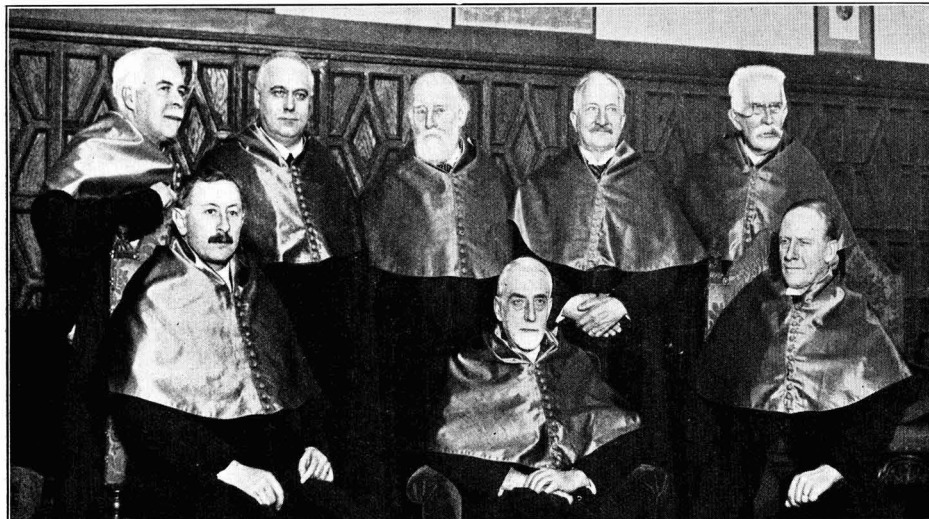
Sir Max Muspratt, a director of Imperial Chemical Industries, Ltd., who died in April in his sixty-third year. He was one of the best known men in the chemical industry, and a business man of extraordinary ability. His grandfather established the first alkali works in this country, 111 years ago, and Sir Max followed his own father in the control of the undertaking up to the time it was absorbed by Imperial Chemical Industries Ltd



Dr. Walter Rosenhain, F.R.S., Fellow and Past-President of the Institute of Metals, by whose death in March the world lost one of the most brilliant metallurgists of all time. He was educated in Melbourne, Australia, and graduated in 1897 from the University of Melbourne in physics and engineering. He was for 23 years superintendent of the Department of Metallurgy and Metallurgical Chemistry at the National Physical Laboratory.



Group at the opening of the Chemistry Department of Leeds University early this year. *Left to right*: Colonel C. H. Tetley, Professor Cohen, Sir F. Gowland Hopkins, Professor Dawson, Mr. A. E. Wilkinson (Lord Mayor), Professor Whytlaw Gray, Sir J. B. Baillie, Professor Challenger and Professor Perkin.



Professor H. E. Armstrong, the doyen of British chemistry, was one of the recipients of the degree of Doctor Honoris Causa in the Faculty of Science at the University of Madrid on the occasion of the ninth International Congress of Pure and Applied Chemistry. Others in the group are (left to right) G. N. Lewis (Berkeley), N. Parravano (Rome), P. Walden (Rostock), H. Le Chatelier (Paris), P. Karrer (Zurich), E. Fourneau (Paris) and R. Robinson (Oxford).



The Honorary Degree of Doctor of Laws was conferred on Sir Harry McGowan at Birmingham University in June. Among those who attended were Professor G. T. Morgan, Sir John Cadman and Mr. Walter Barrow, Pro-Chancellor of the University.

# Progress in Solvents and Plasticisers

By Thos. H. Durrans, D.Sc., F.I.C.

**O**BERVED casually the solvents industry might seem to be quiescent as regards new developments; there has indeed been a tendency to revert to the well-tried solvents and plasticisers and to curtail adventures with new materials whose properties are not well known and the use of which has occasionally led to disaster. Present-day activities are mainly along the lines of "long range" research on methods of production of the more widely used solvents and in widening the range of raw materials from which such solvents can be made; the economic realisation of some of these ambitions may profoundly affect the situation and cause a shift in the area of production which may be detrimental to this country.

## Production of Alcohol from Ethylene

At the present time alcohol is the basis from which the most widely used cellulose ester solvents are made and the rapidly increasing use of alcohol as a fuel for internal combustion engines appears to be leading to the possibility of a shortage of suitable fermentable carbohydrate material from which alcohol can be made. Much work has been done on the production of alcohol from ethylene by absorption in sulphuric acid. The process has been exhaustively investigated in the past and has been actually used on a large scale, but it suffers from defects which render the process uneconomic unless profitable outlets can be found for the higher alcohols which are simultaneously produced. The higher alcohols produced by this method are mainly secondary and tertiary alcohols for which the demand is but small and this factor limits the production of alcohol.

Another method which does not suffer from these disadvantages has been studied during the last few years and consists in causing the direct union of ethylene with water by passing a mixture of ethylene and steam over catalysts at elevated temperatures and pressures. This method presents the fundamental dilemma that in order to obtain a reasonable speed of combination elevated temperatures are required, but with increasing temperatures the degree of conversion diminishes. Some success is now being had by using more suitable catalysts but the degree of conversion still remains at what appears to be an uneconomic figure. If, in this process, the higher olefines are substituted for ethylene the corresponding iso, secondary or tertiary alcohols can similarly be formed but the degree of conversion is again low and the method appears to be at a disadvantage with the sulphuric acid method which is now used for the large-scale production of iso propyl, secondary or tertiary butyl and amyl alcohols.

## Acetone and Higher Ketones

The large-scale production of acetone from alcohol has been carried out for several years by passing a mixture of alcohol and steam over oxides of iron, manganese or copper at about 500° C. Variations of the method have been disclosed, such as the passage of alcohol alone under diminished pressure over catalysts consisting essentially of oxides of chromium, manganese and iron at 400° to 500° C., yields of 90 per cent. of the theoretical being reached. Another method is that of passing alcohol continuously over barium hydroxide at about 420° C. or as a two-stage process by conducting the reaction first at 250° to 350°, so as to form barium acetate and then decomposing this at a higher temperature. Similar processes are recorded for the production of higher ketones of the type methyl ethyl ketone. This substance has been available for many years as a by-product of the wood distillation process for the manufacture of acetone, but the uncertain nature of the supply caused this ketone to fall into disfavour, accentuated by the fact that it compares badly with ethyl and butyl acetates as a solvent. The necessity for finding outlets for the secondary alcohols obtained by the hydration of petroleum olefines has resulted recently in the return of methyl ethyl ketone to the market as a solvent for cellulose esters. Methyl iso-butyl ketone produced in a similar manner is now available, but the more favourable boiling point of this substance is off-set by its diminished solvent power.

Another means of disposing of the secondary and iso-alcohols is by dehydrogenating or oxidising to aliphatic acids. This is effected by treatment with mixtures of caustic soda and potash at about 300°, methane and hydrogen being split off and an aliphatic acid produced having one less carbon atom than the original alcohol. Thus iso-propyl alcohol yields acetic acid in 93 per cent. yield while secondary butyl alcohol gives propionic acid in 95 per cent. yield.

## Partial Oxidation of Petroleum Hydrocarbons

The preparation of cellulose ester solvents by the partial oxidation of petroleum hydrocarbons has been explored by James for some years. The oxidation is effected by passing selected fractions of hydrocarbon distillate with air over complex catalysts of uranium, molybdenum, vanadium and cobalt at high temperatures. The product of the reaction, which consists of a mixture of alcohols, aldehydes, ketones and esters, is a powerful solvent, but the separation of the constituents is difficult. By treating the mixture with a small quantity of an inorganic acid the aldehyde and alcohol can be caused to unite to form acetals which can be removed by means of selective solvents. These acetals have powerful latent solvent properties and their production at an economic price is of considerable interest.

The use of ketene as an acetylating agent continues to receive attention. The highly reactive nature of ketene has necessitated the development of a special technique for its treatment. Ketene is produced by the pyrolysis of acetone and must be immediately diluted with an absorbing or diluting medium in order to prevent spontaneous polymerisation, which otherwise occurs with great rapidity. Acetone has been used for this purpose, the dilution being effected by passing an excessive proportion of acetone through the pyrolysing apparatus or otherwise. The ketene solutions thus produced are not very suited to subsequent use and it has now been found possible to introduce the ketene, under suitable control, directly into the material which it is desired to acetylate such as water, acetic acid, alcohol, ethyl glycol or aniline. By this means acetic acid, acetic anhydride and acetates of alcohols and amines may be produced without the production of any water of reaction and without the use of any esterification catalyst. Given good yields and a low price for acetone the process has merits of distinct economic importance.

## Cyclic Ethers

The cyclic ether, dioxane or diethylene dioxide, has been known for some time and is a useful "medium-boiling" solvent for cellulose acetate. The manufacture and use of other cyclic ethers has been receiving considerable attention and a number of such solvents ranging from "low-boilers" to plasticisers have been produced. One method of preparation is to treat dichloro-aliphatic ethers with lead oxide or other heavy metal oxides whereby ring-closure is effected; dimethyl dioxane can be obtained from  $\beta$ - $\beta$  dichloro dipropyl ether and methylene ethylene dioxide from chloromethyl chloroethyl ether. An alternative method consists in condensing aldehydes and glycols by means of etherifying catalysts—thus formaldehyde condensed with glycol by means of phosphoric acid yields methylene ethylene dioxide b.p. 78°. At the plasticiser end of the series are the cyclic ethers prepared by dehydrating various glycols, glycerine and sugars. As an example of these products diglycerine-trioxane may be quoted. Similar substances can be made by treating a suspension of glucose or erythritol in acetone with hydrochloric acid whereby a crystalline product of the polyhydroxy substance and acetone is formed having plasticising properties for cellulose acetate. A somewhat similar substance is furfural glycol, a cyclic acetal obtained by the condensation of furfural and glycol—this substance plasticises cellulose esters.

As usual there is a large number of new patents dealing with unusual substances as plasticisers. Many are of doubtful value and the patents seem to be merely blocking patents of no technical significance. Some of those worth considering, dichloroacetates of polyhydroxy alcohols prepared by the direct esterification of chloroacetic acid with glycerol, sorbitol or pentaerythritol, are said to be stable to light and other influences. The ethyl ester of phenoxy butyric acid has been recommended for plasticising cellulose esters and also the ethyl and butyl esters of methoxy benzoic acids prepared by the esterification of methylated salicylic acid or the other hydroxybenzoic acids. Crystalline effects can be obtained in celluloid sheet by using *p*-bi-phenyl benzoate, about 10 per cent. by weight being required and at this strength the sheet is not rendered unduly brittle. Many

other substances have been suggested as plasticisers. These are almost invariably obvious variations of the many possible esters which can be obtained and the inventive capacity shown is not of a high order.

Substances which can be used for plasticising gelatine, glyptal resins and cellulose nitrate have been prepared by etherifying polyhydroxy alcohols such as sorbitol, pentaerythritol and butylene glycol with ethylene oxide while the use of methylene diphenyl ethers has been proposed for plasticising benzyl cellulose to produce moulded products having high electrical resistance. A new plasticising solvent for cellulose esters which is now available in bulk is butylene glycol diacetate. This substance is stable to light and seems to prevent the discolouration of cellulose nitrate films, a property of considerable importance.

## The British Synthetic Fertiliser Industry

### Progress at Home and Overseas

It has become the custom to preface every modern statement on industrial matters with the hope, piously expressed, that things are now upon the upward grade, that the corner has been turned and that rewards for patience and perseverance are about to be garnered. Progress, however, is relative: the position of to-day must often be judged in the light of ground regained and consolidated. The synthetic fertiliser industry has shared with other great industrial ventures the difficulties of recent times and its progress in the past twelve months must therefore be measured by relative standards and not only by tonnage output. Where none has enjoyed prosperity, the fertiliser industry has continued to demonstrate its inherent security and strength.

British agriculture, by a process akin to nationalisation, has secured or is securing an ever-increasing share of the home market. For the fertiliser industry this has meant a steady trade at home. The agriculture of the Empire and overseas countries has not as yet been so prosperous. In these countries the fertiliser industry has still, in many cases, to contend with reduced purchasing power, difficult exchange conditions and a general falling off in demand.

#### Sulphate of Ammonia

The agricultural consumption of sulphate of ammonia in the British Isles for the year under review is estimated at about 210,000 tons. This is a decline on the high figure reached in special circumstances last year of some 12 per cent., the diminution in consumption being fairly evenly spread but occurring chiefly in England and Wales. It should be recorded here that at least half of this decline in sulphate of ammonia consumption was counter-balanced by an increase in the use of other nitrogen fertilisers and that the total consumption of fertiliser nitrogen in the British Isles was only about 4½ per cent. less last year than in the preceding twelve months.

Examination of the shipments to foreign markets reveals big increases in exports of sulphate of ammonia to Japan and India but declines in the trade with China, the West Indies and Australia. The Japan figure is particularly interesting as previous indications were that the country was now self-supporting in respect of nitrogen and the imports in recent years have been on a declining scale. The cause of the increase was shortage of water, the source of power on which Japan mainly relies for her fertiliser industry. The usual big trade was done with Spain, Portugal and the Canary Islands, the Portuguese figure showing a large increase on the previous year. Other noticeable increases were in the export figures for the Straits Settlements and South Africa.

On the technical side of sulphate of ammonia production there is nothing to say. There is now scarcely any badly-made sulphate obtainable. The improvements of recent years have resulted in a product of uniform excellence, capable of prolonged storage under most normal conditions.

Continued progress has been made, particularly in the home market where the trade is increasing very rapidly. The new marketing boards are stabilising the cash prospects for tillage crops, with the result that farmers everywhere are making

fuller use of complete dressings of plant food. The savings in freight and handling charges made possible by these concentrated fertilisers appeal very strongly to those engaged in growing crops that normally require heavy dressings per acre, and they have been quick to seize upon them as a means of effecting economies without stinting the land.

An increasing number of householders in Great Britain have become, as the Americans would say, "lawn-conscious" since the industry placed at their disposal the knowledge, gleaned from years of research, whereby the most weedy patch of grass may be welded into a homogeneous sward. The popularity of synthetic garden fertilisers for allotments all over the country may also be observed at any of the innumerable exhibitions of produce from these small holdings.

Nitro-chalk has increased its popularity. Each year a new sales peak is reached and the past year has been no exception. The word "nitro-chalk," originally coined as a trade name, has passed into the common agricultural vocabulary. All standard agricultural text books contain references to its excellence and the fertiliser is constantly being advocated for a variety of crops. Its importance to the commercial apple grower was stressed at the recent Fruit Growers' Exhibition at Leicester.

Last year, in a corresponding article, the opportunity was taken to mention the numerous pest control products which are so closely allied to the fertiliser industry. Mention may be made in the present retrospect of the new product, Nictagral, a nicotine insecticide, which has recently been developed. Organic mercurial dust seed-dressings, such as Agrosan, are constantly increasing in popularity for farm use and horticultural modifications are now making their appearance. Work on the control of fungous diseases and the various pests to which farm crops are liable was for many years regarded as the Cinderella of agricultural science. This state of affairs has now been remedied.

#### Agricultural Research

At Jealott's Hill the fertiliser industry continues to search for new knowledge and to demonstrate past achievement for the benefit of the interested agriculturist. Last year over 2,500 visitors from home and overseas went to the research station to exchange ideas with the staff there. In addition to the well-known grassland management experiments, such basic agricultural problems as silage making, the artificial drying of grass, the economic expansion of yields in suitable farm crops are under the closest observation at Jealott's Hill, which is linked by sentiment, the freemasonry of science and the training of its members with every other research station throughout the world.

This resumé of progress at home and overseas in the synthetic fertiliser industry began by deprecating a popular tendency to undue optimism in these difficult times. Consistency demands, therefore, that it shall close on a note of hope. Indeed, it may justifiably be pointed out that so intimately is the industry bound up with the destiny of this country that its reviving fortunes are a true reflection of the national prosperity.

# Fatty Oils and Soaps in 1934

By Professor T. P. Hilditch, D.Sc., F.I.C.

THE technical movements discernible during 1934 have been directed more to coping with economic pressure of one kind or another, than to the application of any strikingly novel principle; this applies especially, and not unnaturally, to reports appearing in the literature from German, American and Russian sources.

Consider, for example, the manufacture of soap. The soap-maker, in all parts of the world, has at the present time to watch a number of factors which were non-existent, or of relatively little importance, a few years ago. The progress made recently in the production of highly efficient detergents of the so-called "sulphonated" type must be regarded seriously; even if it is unlikely, on the ground of production costs, that these will be active competitors with ordinary soaps for some time to come. Apart from this, however, there continues a general increase in the demand for preparations of the flake or powder types, as distinct from the older bar-soap. Further, in some countries increasing pressure is being exerted to ensure the consumption of home-produced fats in soap-making, while in Germany recent regulations forbid the use of neutral fats for soap-making and also prescribe a maximum content of 50-52 per cent. fatty materials in grained soaps.

The German factories are paying more attention than ever to processes for distilling and recovering fatty acids from crude fats, and high efficiency with good quality of the product is claimed for several processes in which, as a rule, principles similar to those of the Wecker method are embodied. The desire to eliminate some of the time occupied in the conventional soap-boiling process and to effect still further reduction in costs is seen in various reports from America and Germany of rapid saponification methods, in which higher temperatures, or the use of more concentrated caustic lye than formerly, is recommended; and it is also shown by the interest maintained in what are termed "emulsification processes." The latter may be described briefly as developments of the old "cold process saponification" for nut oils or castor oil to other classes of fats, the saponification usually being effected at a temperature above atmospheric.

## Spoilage During Storage

Perhaps the most conspicuous advances during the year are concerned with the prevention of spoilage of fats and soaps during storage. The reader will be familiar with the increase in recent years in our knowledge of the onset of oxidative rancidity in fats, and of the manner in which such oxidation can be retarded by the addition of minute amounts of substances such as hydroquinone and some other compounds of a phenolic nature. The addition of substances of this kind is not altogether a satisfactory solution, especially where edible products may be concerned; but Greenbank and Holm have now found that certain unsaturated acids, especially maleic, are practically as effective antioxidants as hydroquinone. A more favourable line of approach, however, seems to have arisen from the earlier observations of these authors, and of Lea, that the oxidation process is markedly accelerated by light. Subsequent investigations showed that greenish light of wave-length 4900-5800 Å has but little, if any, tendency to accelerate oxidation of unsaturated fats, and Coe and Leclerc in America, and Davies in this country, have now recommended the use, in containers for fats, of green-coloured cellophane or glass. P. I. Smith similarly advocates the use of green-coloured wrappers for soaps in order to check rancidity. Whether, in soaps, rancidity is due really to oxidation of soap, or to oxidation of unsaponified fat is also present, is still, apparently, a matter of some dispute.

As regards edible fats, it seems now to be generally accepted that the delinquents which develop "rancid" or "fallow" flavour or odour are oleic glycerides and not the more unsaturated linoleic (or linolenic) groups. The latter, however, absorb oxygen more rapidly with formation of organic peroxides and other products, which are detected by the Lea or the Kreis tests, etc. So that it is easy to understand why even the "peroxide test" does not invariably correspond with the degree of rancidity as given by flavour. It may be added that the oleic compounds, to whose oxidation

the objectionable flavour is now ascribed, are said to oxidise much more rapidly when linoleic compounds are also present, the "peroxides" of the latter apparently acting as oxygen carriers to the oleic glycerides.

## Extraction of Fats from Seeds and Animal Tissue

New procedure in connection with the extraction of fats from seeds or animal tissues is always being suggested, and a good deal of space might be given to this. As regards oil-seeds, perhaps the most interesting items of the year are the protective action of carbon dioxide during storage of the seed (Kayukova) and the proposal by Rosenthal and Trevithick to use propane and butane from American petroleum gas for extraction of fat from seed-meal at low temperature under pressure. Besides ready removal of the solvent (as gas) from the extracted oil, the latter is said to be exceptionally pale in colour; but the process presumably requires the oil-mill to be adjacent, or, at any rate, not too far away from the oil-fields.

Reports from several research stations show that the problems of fatty oil production in different parts of the British Empire are being dealt with steadily and systematically. An account has appeared of the work so far undertaken by, and the special problems confronting, the recently-established Coconut Research Scheme in Ceylon. A Government committee has made a very full review of the conditions in the oil plantation industries of Malaya, and has recommended that, for the present at any rate, efforts should be mainly directed to consolidating and developing the production of coconut and palm oil; attempts to extend the industry to other kinds of oil-seeds are deprecated under the existing economic conditions. At home, the Paint Research Station is continuing its work and publications on tung oil. Dr. Jordan considers that separate specifications are desirable for the tung oils derived from the two varieties of *Aleurites*, *montana* and *Fordi*. British standard specifications for raw and boiled linseed oils, as well as for tung oils, are at present under discussion.

## New Demand for Fish Oils

Sustained interest is also displayed in fish oils, both in this country, America and Japan; and at home the Torry Research Station of the Food Investigation Board has continued active participation in this field. Perhaps the most conspicuous recent technical feature here is the development of the new demand for halibut liver oil on account of its high vitamin A potency: the production of halibut liver oil is, at the moment, a very profitable, although, naturally, not very large, industry. From recent reports it seems that the liver oil of the tunny is as exceptionally rich in vitamin D as halibut liver oil is in vitamin A. The reasons for these variations from one species to another, or for the seasonal variations in vitamin potency of livers of the same species, are little understood, but are being investigated by several groups of workers at different centres.

At the present time it is probably fair to say that extensions in our knowledge of the chemical constitution of natural fats are more prominent than new advances in technology. It would not be surprising if this continued for some time to come, since for many years fat technology developed far in advance of the structural organic chemistry of the subject.

Detailed analyses (due largely to Lovren) of fats from different kinds of aquatic animals and plants are now available in sufficient number to show certain characteristic differences between them, for example, the liver oils of marine and of fresh-water fish; while, in some of the larger marine animals (sturgeon, porpoise, etc.), the compositions of depot-fats from different parts of the body can now be compared. One curious fact which has emerged concerns the presence of mixed glycerides containing *iso*-valeric acid in some of the fats of dolphin or porpoise: the *iso*-valeric acid (combined for the most part with typical "marine" highly unsaturated



acids in mixed glycerides) is present only in certain depot-fats (blubber, head and jaw) and is absent from the fats of the liver and other organs. Further reports of work of this nature, which is being extended to many other cases of fats from aquatic sources, including phyto- and zoo-plankton, algae, etc., will be read with much interest.

Klenk has made detailed studies of the composition of an amphibian depot fat (that of the common frog) and has found that its component fatty acids are to some extent intermediate between those of fats of marine and land animals. The large variety of unsaturated acids characteristic of "aquatic" fats is present, but some of them are less prominent than usual, owing to the presence of much more oleic and linoleic acids than is found in fish fats. At Liverpool, the writer and his colleagues have recently observed that palmitoic acid (formerly only known as a "marine" fatty acid) forms about 7 per cent. of the depot fats of hens and of rats. There appears to be a more or less progressive change in the components of animal depot fats from the complex unsaturated type found in marine animals to the much simpler mixture present in body-fats of the larger land animals. But there is still room for a great deal more research and collection of data in this field.

### Synthesis of Oleic and Elaidic Acids

Perhaps the outstanding achievement in the pure chemistry of the fats during 1934 is the first complete synthesis of oleic and elaidic acids by Noller and Bannerot. The history of oleic acid epitomises rather well the rate of progress of

organic chemistry in this field as a whole. First definitely recognised by Chevreul in 1815, its conversion to an isomeric form (elaidic acid) was observed by Poutet in 1817. The position of its ethylenic linkage was not defined until Baruch established it as  $\Delta^{9,10}$ -octadecenoic acid in 1894. That oleic acid was the *cis*-, and elaidic acid the *trans*-, form of the pair of geometrical isomerides was only recognised in 1923, as the result of X-ray spectrographic study by Müller and Shearer. In 1925, Professor and Mrs. Robinson achieved an almost, but not quite, complete synthesis. They synthesised 10-ketostearic acid and also reduced stearolic acid to oleic acid, but were unable to convert the keto-acid into the acetylenic stearolic acid (the latter, however, can be transformed into ketostearic acid). Finally, in 1934, a complete synthesis of, firstly, heptadecenylchloride, and then oleic acid has been obtained by a somewhat complex series of reactions, starting from 9-chlorononylaldehyde.

In the methods of technical analysis, discussion has continued with reference to the comparative merits of different procedures for the determination of iodine values and of the acetyl values of fats. Greene has put forward cogent arguments in favour of the more general and more developed use of microscopical examination of fats and soaps. A method for approximate estimation (as elaidic acid) of oleic acid in mixtures of the latter with linoleic and linolenic acids has been devised in the Liverpool laboratories; the procedure, which is somewhat lengthy, gave, with a series of oils, results in excellent agreement with those determined by the thiocyanometric method, thus confirming the validity and utility of the Kaufmann process.

## New Company Progress for the Past Three Years

By S. HOWARD WITHEY, F.C.I.

(Senior Fellow of the International Accountants' and Executives' Corporation)

SINCE 1931, many of the well-established chemical and directly allied companies have had more liquid resources than at any time during their history, and as those concerns whose finances were not in a very healthy condition have not been encouraged to seek additional capital under the existing economic and trading conditions, new issues and new formations have been relatively few.

In the wholesale and export druggists section, Wright, Layman and Umney (1932), Ltd., was registered in June, 1932, with an authorised share capital of £340,000, all of which has been issued and fully paid, consisting of £280,000 in the form of 7 per cent. cumulative preference £1 shares—the dividend on which is paid half-yearly in April and October—and £60,000 in ordinary £1 shares. During the financial year immediately preceding the formation of the new company, a net profit of £40,039 was realised on a share capital of £180,000, the latter consisting of £90,000 in 6 per cent. preference shares, and £90,000 in ordinary shares, which enabled a dividend of 4½ per cent to be paid. In the following year, the balance of net profit reached the record figure of £40,302, but in view of the fact that in a complete year the dividend on the preference shares absorbs £19,600 gross, the directors very wisely adopted a conservative policy, and declared an ordinary dividend of 10 per cent., which left a balance of £24,534, of which the sum of £17,500 was allocated to reserve. During 1933, the trading profit amounted to £44,745, and after providing for income-tax, the net profit worked out at £30,705, enabling the dividend of 10 per cent. to be maintained, and £10,000 to be transferred to the reserve fund, which now totals £27,500. The next accounts will probably be submitted in March, and there is a credit balance of £8,139 to be brought in.

The first accounts of National Drug Industries, Ltd., covered a period of thirteen months to March 31, 1933, and revealed a net profit of £2,601. This company was formed for the purpose of acquiring the undertakings of C. R. Harker, Stagg and Morgan, Ltd., and Thos. Hodgkinson, Prestons and King, and owns works and laboratories at Emmott Street, London. The authorised capital is £80,000, of which £63,065 has been issued and fully paid, comprising £31,250 in the form of 6 per cent. cumulative preference £1 shares; £31,315 in ordinary £1 shares, and £500 in deferred shares of 1s.

denomination. For the initial period, the preference dividend was paid, leaving £640 to go forward, and during the twelve months ended March 31, 1934, a net profit of £4,266 was realised, enabling the dividend on the preference shares to be maintained, and the sum of £309 to be allocated to a leases depreciation reserve, and £2,724 to be carried forward to the next account.

In 1932, British Plaster Board, Ltd., was made public, the authorised capital being £250,000, of which a total of £208,000 has been issued in the form of ordinary 5s. shares of one class. Great progress was made during the financial year to April 30, 1933, the trading profit amounting to £54,342, and after transferring £11,521 to the tax reserve, and debiting expenses in connection with the increase of capital, etc., the net profit proved to be £41,524. The demand for the company's products necessitated the installation of additional plant, and during the twelve months to the end of April last a trading profit of £100,387 was realised, the net profit working out at no less than £86,617. Consequently, the dividend of 30 per cent. has been maintained, 15 per cent. being payable on £100,000, and 15 per cent. payable on £208,000, absorbing £46,200, as compared with £30,000 in 1932-33, and after placing £35,000 to the reserve—which now amounts to £50,000—and allocating £3,142 to obsolescence, there remains a balance of £5,691 to go forward to 1934-35, as against £3,415 brought in from 1932-33. The company is well equipped to meet any competition.

In October, 1932, English Clays, Lovering, Pochin and Co., Ltd., was registered for the purpose of acquiring the china clay businesses of English China Clays, Ltd., Lovering China Clays, Ltd., and H. D. Pochin and Co., Ltd., and their respective subsidiaries. The first accounts were made up to the end of September, 1933, and were submitted in March last, showing a profit of £28,417. Of an authorised capital of £2,570,225, a total of £2,565,225 now ranks for dividend, comprising £351,873 in the form of 7 per cent. cumulative preference shares, and £2,213,352 in ordinary shares, all of £1, and after meeting the preference dividend to March 31, 1933, and allocating £15,474 to depreciation account, there remains a credit balance of £5,633 to be carried forward to the next account.

# Standardisation in the Chemical Industry

## Activities of the British Standards Institution

PROGRESS has been made during the year in the work of the committees of the Chemical Division of the British Standards Institution and it was a source of considerable gratification to the chemical industry to know that Dr. E. F. Armstrong, chairman of the Chemical Divisional Council, was elected chairman of the Institution. A considerable step forward has been made in dealing with the comprehensive programme of work outlined in the last annual review and specifications have been issued for the following:—

Technique for determining the Rideal-Walker coefficient of disinfectants (B.S.S. No. 541); determination of phosphorus in coal and coke (568); report on standard temperature of volumetric glassware (554); distillation flasks (571); interchangeable conical ground glass joints (572); linseed oil putty (544); nickel anodes (558); nickel ammonium sulphate and nickel sulphate (564); diacetone alcohol (549); normal butyl acetate (551); amyl acetate (552); ethyl acetate (553); dibutyl phthalate (573); diethyl phthalate (574); carbon tetrachloride (575); glacial acetic acid and dilute acetic acid (576); hexachlorethane (hexachloroethane) (577); technical acetic acids (578); technical ether (579); trichloroethylene (trichloroethylene) (technical and stabilised) (580).

The Rideal-Walker method of comparing disinfectants, first published in 1903, has been varied largely by other workers leading to a great deal of confusion and numerous disputes. The object of the standardised technique is to ensure that the buyer and seller, when a particular Rideal-Walker coefficient is part of the requirements of the disinfectant, both use precisely the same method. Its chief application is for coal tar disinfectants. Experience has shown that the standardised technique, if carefully followed, will give concordant results in the hands of competent workers and it is therefore recommended for general adoption by all who purchase and supply disinfectants on the basis of the Rideal-Walker coefficient. The method for the determination of phosphorus in coal and coke is a further development of the standardisation work carried out in the last few years in connection with the setting up of standard methods of sampling and analysis for coal and coke. The present standard is based upon a comprehensive survey of different methods for the testing of a large number of coles from different localities carried out by the Northern Coke Research Committee, nine methods being selected as representative of the methods in use amongst coke producers and users in Great Britain. The nine representative methods were for the determination of phosphorus in a large number of coles from this country and from the Continent, and it was found that seven of the methods gave low results shown to be attributable to the loss of phosphorus in the residues normally rejected during the determination. The method selected as standard is one of the two found to give consistent results and of these two is the more simple and economical in type.

### Scientific Glassware

The specifications for interchangeable conical ground glass joints and distillation flasks represent the first specifications prepared by the Scientific Glassware and Laboratory Ware Committee which is working in close co-operation with the National Physical Laboratory.

The Chemical Divisional Council is steadily developing the application of national standards to those cases where the preparation of British standards will be of service to the industry. The Scientific Glassware and Laboratory Ware Committee has at present six committees actively working on the preparation of specifications for scientific and laboratory ware, including thermometers, hydrometers and volumetric glassware. Consideration is being given to the standardisation of qualities of the resistance of glassware and to the question of laboratory porcelain.

The Vegetable Oils Committee, under the chairmanship of Mr. Bolton, has almost completed its series of specifications for vegetable oils, and standard methods are being suggested for the sampling of vegetable oils and fats in packages or in bulk. The Coal Committee is working on methods for the ultimate analysis of coal and for the analysis of coal and coke ash, and it is hoped to issue early in the new year a method for determining the agglutinating value of coal.

The Disinfectants Committee is endeavouring to set up a standard for the Chick-Martin test, while the Tar Acids Committee has been engaged in the revision of the specifications for crude carbolic acid, distilled carbolic acid and refined cresylic acids.

Perhaps the most important development in the chemical work of the Institution is the setting up of a Dairy Standards Committee to deal with the question of standardisation in the dairying industry. This work is a development of the work of the Dairy Research Committee of the Empire Marketing Board. With the discontinuance of the Board in 1933 it was left to the Imperial Agricultural Bureau to consider what steps should be taken to provide for the future of schemes initiated by the Board. After consideration, the Imperial Agricultural Bureau asked the British Standards Institution to set up a Technical Committee for Dairy Standards to continue the work carried out under the Dairy Research Committee, and to deal with any further standardisation which might be required by the dairy industry.

Amongst the other new work that is being undertaken, reference should be made to the newly formed Glues and Leather and Tanning Materials Committees. The Glues Committee is attempting to set up standard methods for testing glues, while the Leather Committee has completed a preliminary draft specification for vegetable tanned sole leather.

## Federal Council for Chemistry

### International Problems

THE objects of the Federal Council for Chemistry are to foster and advance the scientific interests of chemistry in all its branches and (1) to represent the views of British chemists, both nationally and internationally, (2) to co-operate with those bodies in Great Britain and the British Empire representing the science of chemistry and to co-ordinate their scientific activity, and (3) to enter into scientific relations and to co-operate with similar bodies in other countries, and, in particular, to represent British chemistry in the International Union of Chemistry. The council consists of representatives appointed by each of the constituent organisations and of such other members as the council may from time to time elect.

Several matters of international importance have occupied the attention of the Federal Council during 1934. The IXth International Congress of Pure and Applied Chemistry (postponed from 1932) was held in Madrid from April 5 to 11 to which the Federal Council sent ten delegates. A number of its constituent organisations were also represented. The XIth Conference of the International Union met at Madrid at the same time as the Congress, to which delegates were appointed. Among questions of importance considered was that of chemical nomenclature. Delegates of the Federal Council were also present at the following functions held in Paris: The IIIème Congrès Internationale Technique et Chimique des Industries Agricoles (March 25 to 31), Quatorzième Congrès de Chimie Industrielle (October 21 to 27) and the official opening of the Maison de la Chimie (December 1).

The reports of the International Commission on Atomic Weights for 1934 and of the International Commission on Thermochemistry have been received from the International Union and steps taken to give necessary publicity to these reports in the technical Press in this country.

The Federal Council continues to give attention to the promotion of a scheme for the unification of the professional and scientific welfare of chemists to which reference was made in the review of last year. Following meetings of representatives of the Federal Council for Chemistry, the Chemical Society, the Institute of Chemistry, the Society of Chemical Industry and the Association of British Chemical Manufacturers (representing industry) a "Chemical Council" has been formed to put into practice a scheme of co-operation between three of the principal organisations representing chemistry in this country.

# The British Association of Chemists

## A Steady Advance Throughout the Profession

ONE of the outstanding features of the work of the British Association of Chemists during the year has been the increased facilities of the employment bureau. The welcome fall which has taken place in the unemployment benefit paid is not due alone to the improvement in general conditions. A great deal has been done by the Association to find employment of a satisfactory kind for its members. In many cases members have obtained highly remunerative appointments. This is to a large extent due to the propaganda carried out over a number of years which has aimed at impressing employers with the economic advantages of employing a chemical staff. After many years of consistent work the results are becoming apparent in the increasing number of applications made to the bureau by employers who either have not previously employed a chemist or who have decided that an increase in the chemical staff would actually result in economies being effected.

### Salaries

For this reason the Association continues to insist upon a salary of £350 per annum as a bare minimum. The Association has always been interested in maximum rather than in minimum salaries but some provisional minimum has to be fixed. It is the view of the council that except in special circumstances no chemist should accept a post at a salary below this minimum. With few special exceptions members are able to command this initial minimum and in many cases a great deal more. The increase in the membership of the Association during recent years has greatly strengthened the Association's ability to negotiate in regard to economic conditions. A great deal more can be done when it is fully realised by chemists that it is only by supporting a society with specific powers to deal with economic questions that satisfactory conditions can be secured for the whole profession.

### Legal Aid

The legal aid department has done valuable work for members. £809 has been recovered for members during the past year. A number of other cases have since been settled by negotiation so that the amount recovered to date is considerably in excess of this sum. The principal aim of the department, is, however, prevention rather than cure. Owing to the careful attention given by the department to the review of agreements, a large number of members had clauses in agreements modified or removed so that possible causes of litigation have been eliminated. While this activity necessarily receives little publicity the work has been steadily developed and continues to increase. Owing to the increasingly strong position of the unemployment benefit fund, the unemployment special purposes committee has under consideration ways and means by which additional benefits might be made available for members. It is not yet decided what form these benefits should take but alternative schemes for additional benefits will be submitted for the consideration of members.

### Active Local Sections

Excellent work has been done by the local sections. The committees have been energetic in arranging meetings and providing special speakers. In a number of instances joint meetings have been arranged with the local sections of other societies. The importance of a closer co-ordination between the various chemical societies is fully appreciated by the council. If the Association is not in full agreement with many suggestions which have been made for the closer co-ordination of chemical organisations, it yet welcomes all genuine efforts which are being made in this direction. Work in the local sections is doing a great deal to establish closer contact between the societies, and joint meetings which the local sections have been active in endeavouring to foster give opportunities for discussing the local problems and their bearing on the question of co-ordination.

The council is of opinion that the general problem of registration is closely bound up with the question of co-ordina-

tion. Its scheme for registration, which has been worked out in some detail, requires as a first step the setting up of a provisional council or at least a co-ordinating committee which would make it possible for all societies directly or indirectly interested in the question to explore it. Until this is done it is not possible to deal further with the question of registration. But there is evidence that the rank and file of the profession are still interested in the matter. The responsibility for the next step, however, lies not with the Association alone but in closer co-operation between the societies.

The work of the nominations committee this year has been exceptionally heavy, first on account of the large number of applications, and, secondly, by reason of the great care with which applications have to be scrutinised. The Association has definite regulations which govern admission to full membership and those who do not obtain the required standard cannot be admitted in the capacity of full members. The British Association of Chemists is the only society which requires besides evidence of sufficient general and scientific education satisfactory proof of competency in chemical practice.

The activities of the Association in regard to publicity have been maintained through its journal and in the technical and public Press. The increase in its membership and its steadily-improving financial position are due to the fact that the benefits of membership of the Association become increasingly widely known.

## Society of Glass Technology

### More Work: Increased Subscriptions

THE activities of the Society of Glass Technology have been well maintained during the year. The programme included nine ordinary general meetings, four in Sheffield, two in Leeds, two in London, and one in Stourbridge. At the annual general meeting at Sheffield in April, Mr. G. V. Evers, of Stourbridge, was re-elected president for a second year. Several alterations were made during the year in the Society's constitution and rules. The annual subscription of collective members (firms and institutions) was increased, as from January, 1935, from £3 3s. to £5 5s. The annual subscription of ordinary members was also increased from £1 10s. to £2 2s., but in this case the increase will not become operative until January, 1936. In addition, the former grade of student membership was replaced by a new grade of associate membership. This is open to persons under 25 years of age, the annual subscription being 7s. 6d.

The quarterly "Journal of the Society of Glass Technology" appeared as usual during the year, and included some thirty original papers and reports. Of the other publications of the Society, the opportunity was taken of thoroughly revising its "Directory for the British Glass Industry," and the new (third) edition was published in September. It contains over 400 pages. Four of the Society's technical committees have been active, namely, (a) Standards Committee, (b) Furnace Committee, (c) Refractories Committee, and (d) Committee on Form, Design and Process Work. A development of the work of the last-named committee was the institution of an Art Group of the Society, consisting of members interested in the work of this committee. The May meeting in London was devoted to a symposium on the form, design and decoration of glass.

Early in the year a Yorkshire section was successfully inaugurated. The Society therefore now has three flourishing sections, (a) London Section—hon. secretary, Mr. T. C. Crawhall, M.Sc. (The Science Museum, South Kensington, London, S.W.7); (b) Midlands Section—hon. secretary, Mr. G. W. Stuart (Stuart and Sons, Ltd., Red House Glass Works, Stourbridge); and (c) Yorkshire Section—hon. secretary, Mr. A. Garstang (King, Taudevin and Gregson, Ltd., Cambridge Street, Sheffield). The headquarters of the Society are at Darnall Road, Sheffield, 9, the hon. secretary being Professor W. E. S. Turner, O.B.E., D.Sc.

# The Institution of Chemical Engineers

## Membership Exceeds Eight Hundred

DURING the year the membership of the Institution of Chemical Engineers has passed the eighth hundred, a sign of continued youthful vigour and useful work. At the twelfth annual corporate meeting, held in February, Mr. W. Macnab, C.B.E., was elected president; Dr. H. Levinstein and Mr. H. Talbot were re-elected vice-presidents and Mr. H. W. Cremer and Mr. F. A. Greene, respectively, hon. secretary and hon. treasurer. New elections to council included Dr. W. R. Ormandy, Mr. C. S. Robinson, Dr. A. J. V. Underwood and Mr. C. Chapman. The address by the retiring president, Viscount Leverhulme, dealt with "Chemical Engineering and the Edible Fat Industry," in which the part played by the chemical engineer in building up this comparatively modern industry was clearly demonstrated.

The Moulton Medal for 1933 was presented to E. W. Colbeck, W. E. MacGillivray and W. R. D. Manning for their papers: "The Mechanical Properties of some Austenitic Stainless Steels at Low Temperatures," and "The Mechanical Properties of Metals at Low Temperatures, Part II—Non-Ferrous Materials"; the Junior Moulton Medal and prize of books was awarded to Dr. E. H. T. Hoblyn for a paper on: "The Solvent Extraction of Sulphur from Sicilian Ores." The Osborne Reynolds Medal, for meritorious service for the advancement of the Institution, was awarded to Mr. H. W. Cremer, the hon. secretary.

The papers read before meetings of the Institution have covered an unusually varied field, and three of the meetings were held jointly with other bodies. The opening paper of the session, "Legal Pitfalls for the Chemical Engineer," by Mr. J. Davidson Pratt and Mr. G. S. W. Marlow, gave a comprehensive account of the legal obligations demanded of the chemical engineer and was of so outstanding a character that it has been made available as a separate booklet, and is

on sale at the price of 3s. per copy. Other papers during the year included: "The Washing of Gas," by H. Hollings and L. Silver, and "Multi-Stage Washers," by L. Silver; "Modern Methods of Attacking Heat Transmission Problems," by Professor C. H. Lander; "The Practical Testing of a Continuous Petroleum Still," by A. H. Goodlife; "The Determination of Plate Efficiency in Fractionating Columns for Complex Mixtures," by Dr. A. J. V. Underwood; "The Economic Generation of Steam," by F. H. Preece and B. Samuels, and "The Economic Use of Steam," by W. F. Carey and A. H. Waring; "Crushing and Grinding," by W. F. Carey, and "Crushing and Grinding Appliances—the connection between Type and Purpose," by Professor Bernard W. Holman; "Separation of Solids from Liquids—Thickening," by W. Cullen and H. T. Durant. The public lecture of the year was delivered on October 26 by Professor C. H. Desch, F.R.S., on "The Influence of Texture on the Chemical Resistance of Materials," and attracted a large audience. Seven meetings were held by the Graduates and Students Section during the year. At the annual meeting of the Section in May, Mr. K. L. Emler was re-elected chairman and Mr. D. G. Bagg was elected hon. secretary.

The president's reception, held at Sutherland House, Mayfair, on November 29, again attracted a large and distinguished gathering, some four hundred guests being received by the president. The Associate-Membership examination for 1934 followed the lines of the last few years. Of seven candidates, six satisfied the examiners.

Steps were taken to compile an index of experience to cover the whole range of chemical engineering experience of corporate members of the Institution. This index has been of great assistance in answering requests for expert help on specific subjects.

## The Institute of Chemistry

### Signs of Better Conditions in Industry

PROFESSOR JOCELYN F. THORPE entered upon his second year of office as president of the Institute of Chemistry at the annual general meeting in March. Professor Morgan's lectures on inorganic chemistry, published in 1933, concluded a series which was intended to form the basis of a "refresher" course in pure chemistry for the benefit of chemists who had left the university for several years. During the year a further series of lectures has been given dealing mainly with "methods." This included the following, which have already been published: "Electrometric Methods in Physical and Analytical Chemistry," by Dr. Samuel Glasstone; "Micro-chemical Methods Suitable for General Analytical Practice," by Professor H. V. A. Briscoe and Dr. Janet W. Matthews; and a further lecture, "New Indicators and Other Reagents employed in Volumetric Analysis," by Dr. A. D. Mitchell, is now in the press.

The sixth Gluckstein Memorial Lecture was delivered by Lord Melchett, who took for the title of his address the question "Is Nature Wiser than Man?" The seventeenth Straetfeld Memorial Lecture was given by Mr. George E. Shaw, who dealt with "Quinine Manufacture in India." This will be published as a separate monograph.

The eighth edition of "Official Chemical Appointments," which was published in April, was extended to include appointments connected with gas, water, sewage, transport and other public utility services, as well as an enlarged summary of legislation, Government departmental regulations, recommendations, etc., affecting the profession of chemistry. A new edition of the register of the Institute was published in August, showing that the membership at that date was 6,212, and the number of registered students, 783. Many valuable papers have been read at the meetings of local sections, frequently held jointly with other bodies; and other meetings have been devoted to discussions on professional subjects, and

to demonstrations and exhibitions of apparatus, visits to works, etc. The conference of hon. secretaries of local sections held at the Institute in June was found so useful that it has been decided to make this an annual event. Arrangements are in progress for the formation of a section in India.

Examinations of the Institute have been held at headquarters, and at Glasgow, Manchester, and other centres. The examinations for National Certificates in Chemistry have, as usual, been conducted jointly by the Institute with the Board of Education (England and Wales), the Scottish Education Department, and this year, for the first time, with the Ministry of Education for Northern Ireland. The Meldola Medal for 1933 was awarded to Dr. Maurice Stacey; the Frankland Medal and Prize to Mr. Walter Lee; and the administrators of the Sir George Beilby Memorial Fund (comprising officers of the Institute, the Society of Chemical Industry, and the Institute of Metals) have made awards to Dr. W. Hume-Rothery and to Dr. E. A. Rudge.

The appointments register committee has reported that the number of members known to be unemployed shows a substantial decrease, which is indicative of the better conditions obtaining in industry. The benevolent fund committee, on the other hand, has found it necessary to disburse nearly £700 more than in 1933, actually more than twice as much, in the assistance of widows and orphans of members, and in helping those who have fallen on bad times.

In co-operation with other bodies, the council has been concerned with many matters of importance affecting chemistry and chemists as a whole, including those relating to the Patents and Designs Act, the Pharmacy and Poisons Act, the Arbitration Act, the Fertilisers and Feeding Stuffs Act, the Food and Drugs (Adulteration) Act, British Standard Specifications, chemistry in general education, expert witnesses, and the promotion of co-operation among chemical societies.

## The Chemical Society

### Progress of Provincial and Joint Meetings

DURING 1934, the council of the Chemical Society has continued its policy of holding lectures, discussions and ordinary scientific meetings outside London as well as in London. On February 22, Professor Dr. Hans Fischer delivered the fourth Pedler Lecture entitled "Chlorophyll": on April 19, an ordinary scientific meeting was devoted to celebrating the centenary of the birth of Mendeléeff, when Lord Rutherford of Nelson lectured on "The Periodic Law of Mendeléeff, and its Interpretation," and on October 18 a lecture on "Induced Radioactivity" was given by Professor P. M. S. Blackett. The following discussions were held: "Some Aspects of the Electronic Theory of Valency," opened by Professor J. E. Lennard-Jones; "Unicellular Chemistry," opened by Dr. J. Vargas Eyre, and "Chemical Problems in Agricultural Science," opened by Sir John Russell.

Six ordinary scientific meetings were held at which 27 papers contributed by Fellows were read and discussed. On June 7, the ordinary scientific meeting held by invitation of the president, Professor G. T. Morgan, at the Chemical Research Laboratory, Teddington, was devoted to a discussion on "Chemical Syntheses under Pressure."

#### Meetings Outside London

Five lectures were held outside London. On January 22, Professor R. Robinson lectured at Birmingham on "Some Recent Work in the Alkaloid Group"; on February 23, Professor Dr. Hans Fischer repeated at Manchester his lecture on "Chlorophyll" on March 13, Professor W. C. McC. Lewis lectured at Liverpool on "The Experimental Study of Some Gas Reactions"; on November 5, Mr. C. N. Hinshelwood gave a lecture on "The Development of Chemical Kinetics" at Birmingham, and on November 15, Professor W. E. S. Turner delivered a lecture on "The Surface of Glass" at Sheffield.

Two discussions were held, the first on "The Ignition of Gases" being arranged by Professor R. V. Wheeler, on January 25, at Sheffield. The second, on "Applications of X-rays and Spectroscopy to the Elucidation of Chemical Structure" was held at Manchester, on November 9 and 10.

Ten meetings of the Society jointly with other organisa-

tions were arranged; on February 2, March 9 and November 16 and 26, with University College of North Wales (Bangor) Chemical Society; on January 26 with the local sections of the Institute of Chemistry and the Society of Chemical Industry at Bristol; on February 9 with the Glasgow sections of the Institute of Chemistry and the Society of Chemical Industry; on March 12 with the Students' Chemical Society of the University College of Swansea; on March 15 with the University of Sheffield Chemical Society; on March 5 with the University of Birmingham Chemical Society, and on November 3 at Liverpool with the Science Masters' Association. Fellows were also invited to attend four Bedson Lectures at Armstrong College, Newcastle-on-Tyne.

#### Anniversary Meetings

The 93rd annual general meeting and anniversary dinner were held at Birmingham in March. The Lord Mayor and Corporation held a civic reception in honour of the Society. The president's address was on "Unification of the Chemical Profession." The principal guest at the dinner was the Rt. Hon. Sir Austen Chamberlain.

In 1934, 218 new fellows were elected. The "Journal" (published monthly) contained 443 original papers and 34 notes, in addition to the special lectures delivered before the Society. Volume XXX of the Annual Reports on the Progress of Chemistry, which summarises the important advances in pure chemistry made during the previous year, was published in March. It contained, in addition to the usual reports, special reports on crystallography, and radioactivity and sub-atomic phenomena. British Chemical Abstracts "A" contained 1,422 pages, compared with 1,344 in 1933.

There were 81 applications for grants from the Society's Research Fund available for the assistance of chemical research, and grants amounting to £672 8s. were distributed to 77 applicants. The use of the extensive reference and lending library of the Society steadily increases. The number of volumes is now over 38,500 and these deal with every aspect of chemistry, and with many branches of related sciences. In 1933, there were 8,399 attendances and the number of books borrowed was 5,518.

## The National Paint Federation

### Success of the Co-operative Advertising Campaign

THE past twelve months have witnessed the consummation of important activities which have engaged the attention of the National Federation of Associated Paint, Colour and Varnish Manufacturers for some considerable time, outstanding examples being the concessions connected with tariffs which have been obtained, in the addition of rosin (colophony) and tung oil (china wood oil) to the Free List and the introduction of a workable and more comprehensive linseed oil drawback scheme. The scheme now in operation provides for an increase in the original rate of drawback from 30s. to 60s. and an extension of the scheme to linseed oil used in the manufacture of putty, mixtures of linseed oil and driers, and heat-treated and refined linseed oil. In addition, a "substitution" scheme, the adoption of which has always been advocated by the Federation, has been accepted by the Customs authorities in a modified form, so that it is now unnecessary to segregate duty paid and non-duty paid oil used in the manufacture of paints and varnishes. The application for the inclusion of copal gums in the Free List, which was turned down last year has been re-submitted after successful representations had been made by the Federation to the New Zealand Government for reconsideration of the Ottawa Agreements Act so far as it affected the Kauri gum position.

Research has been stimulated by an offer from the Government of increased grants to research associations whose members were prepared to increase their subscriptions. The members of the Research Association of British Paint, Colour and Varnish Manufacturers have taken up the challenge, with

the result that the Association is assured of adequate financial support over a period of years. The co-operative advertising campaign advocating the use of more paint, which was launched in the spring and continued in the Autumn, has been a remarkable feature. The campaign will be continued in 1935 and 1936.

The Joint Industrial Council for the industry has completed a year of active and progressive work. Interest among the employees in the technical side of the industry appears to be increasing, 52 certificates having been awarded by the City and Guilds of London Institute to candidates in paint and varnish technology. From the export point of view the story is not quite such a happy one, although concessions with regard to drawback have mitigated the situation considerably. On the other hand, the menace of Japanese competition in practically all export markets is serious and appears to be increasing. In this connection, the Federation has taken every opportunity of representing to the Government the views of the industry and will continue to do so in the future. Revision of the customs tariffs by Australia and New Zealand has been of some assistance, but it is doubtful whether the small concessions granted will make it any easier for manufacturers generally to compete with domestic manufacturers in those markets, or whether the Governments concerned have gone as far as they might have done to carry out the spirit of the Ottawa Agreements Act. The industry has shared in the general upward tendency which has been noticeable during the year.



## The Voice of the Industry

### A Retrospect of 1934 and a Glimpse of the Future

We recently invited our advertisers to tell us how they had fared in relation to the improvement in trade which had become apparent in many directions during the past twelve months, and what were their prospects for 1935. Below we give extracts from their replies.

#### Metropolitan-Vickers Electrical Co., Ltd. Davey, Paxman and Co., (Colchester) Ltd.

DURING the past year the shops of the Metropolitan-Vickers Electrical Co., Ltd., both at Trafford Park, Manchester, and Attercliffe, Sheffield, have experienced a considerable increase of business. The most powerful steam turbo-generator in Europe is being built at the former works, and the latter have many contracts for electric traction equipment, ranging from tradesmen's delivery vans and trolley buses to railway electrifications. Readers of THE CHEMICAL AGE will perhaps be most interested in a series of electrodes which have for the most part been developed in the past year for use in arc welding. Many types are available, each with metal and flux properly proportioned and chosen for the numerous different classes of work now carried out by arc welding.

#### British Tar Products, Ltd.

It is reported by British Tar Products, Ltd., that the tar products market had commenced its recovery in certain respects earlier than 1934, and, strange as it may seem, is even now feeling something in the nature of a reaction inasmuch as prices of certain products, such as toluol, xylol and pitch during the latter part of 1934 have fallen from the high levels enjoyed by them during the latter part of 1933 and early 1934. Those closely engaged in the trade have the opinion that lighter products will recover in the near future although the position of pitch is yet not certain: on the other hand, one of the company's main products, creosote, has gradually increased in demand and value during this period. No new product has been evolved during 1934 but as far as the finer chemicals are concerned, it may be said that the tendency all round seems to be to stiffen up qualities and aim at standards of purity not previously demanded. This means that only by keeping well abreast of the latest developments can a producer hope to enjoy the best markets and this appears to be the keynote for 1935.

#### Aluminium Plant and Vessel Co., Ltd.

A YEAR of considerable development—particularly in the distillation field—is announced by the Aluminium Plant and Vessel Co., Ltd. Plants have been constructed in non-ferrous metals for the distillation of organic acids, ketones, esters and alcohol. Some idea of the magnitude of the work can be gauged from the fact that within a matter of twelve months stills with a combined daily (24 hours) capacity of 900,000 gal. have been constructed. Some of these stills are of immense size and represent a class of work which was previously imported from France or Germany. A particularly interesting development is a patent type of distillation plate for fractionating columns. Three such columns have been built by this concern during the past year.

The demand for the A.P.V. patent plate type of heat exchanger continues to grow and machines have been constructed for use on aldehydes, alcohol, and polymerised oil. They have also been introduced in distillation plants for heating boiler feed water by waste heat contained in the effluent from the stills, etc. Other important chemical plant recently constructed by this very progressive company includes special plants in monel metal for the evaporation of certain solid organic acids, large final evaporators in nickel for caustic soda evaporation, soaking and vacuum washing plants in aluminium for the viscose silk industry, and many reaction vessels for acetic acid and anhydride processes. A great deal of interesting plant has also been made for the varnish industry, among which should be mentioned a complete pilot plant in aluminium, copper and staybrite steel for the manufacture of cellulose esters, large plants for the polymerisation of drying oils (one heated electrically, and others by the special A.P.V. oil fuel firing system) and special varnish plants each of five tons gross capacity for the bulk melting and esterification of copal and other natural resins.

STEADY increase in the manufacture of plant for the chemical and metallurgical trade is reported by Davey, Paxman and Co. (Colchester), Ltd. The machine which has been in greatest demand is the Paxman rotary vacuum filter with its "streamlined" cell construction. To illustrate the variety of uses for this filter, the following cases can be cited where Paxman filters have been sold or installed during the year: Filtration and washing of caustic lime mud in paper mills and tar distilleries, of lime sludge in the manufacture of bleach liquor, of potassium salts recovered from the Dead Sea, of gold cyanide slimes in the metallurgical industry, the dewatering of coal slurries in coal washeries, of precipitated chalk, of sludge from water purification plants and the treatment of trade effluents, including the recovery of pulp from paper mill backwater. In addition to the plants supplied to users in this country, a number of Paxman filters have been exported.

#### The Thermal Syndicate, Ltd.

It was to be anticipated that an increase in the production of chemicals would lead to an improved demand for Vitreosil (pure fused quartz or silica), which is made by the Thermal Syndicate, Ltd., and this has been the case. For over 25 years Vitreosil has been known to be an outstanding material for resisting acid attack and temperature change, and the confidence of chemists and chemical engineers in this equipment continues and increases. In addition to the standard forms supplied for chemical plant, for acid gas absorption and for acid distillation, cooling and condensation, many pieces have been manufactured to purchasers' own designs to suit special applications. All this work has necessitated additions to existing equipment for the manufacture of Vitreosil chemical plant, and the various departments of the Vitreosil works continue to be kept in a high state of efficiency to enable orders for standard or special pieces of equipment to be supplied with the utmost despatch.

Special mention may be made of the improvements which have been effected in the production of transparent Vitreosil so that high-grade optical material free from striae, strain or fog is now available. These advances have also led to increased activity in the production of mercury vapour burners of all types and for all purposes, including industrial uses. Besides the standard forms generally used for actinotherapy, larger units are now available for continuously irradiating and sterilising liquids, including milk and water supplies. Considerable attention is being given to the development of the discharge type of burner, which can be made of more robust construction than the arc type and has the added advantage of being self-starting.

#### Staveley Coal and Iron Co., Ltd.

DURING 1934 there was a little fluctuation in trade and it can be said that the improvement noted towards the end of last year has been well maintained. Judging by the demand for chemical products made by the Staveley Coal and Iron Co., it would appear that most of the trades have benefited to some extent by the improved conditions. The heavy chemical section has been fully employed and the demand has remained good, but in spite of the heavy demand prices have not been raised.

The demands this company has experienced would seem to indicate that the textile trade has certainly had a better year, and the same remarks apply to many other trades. Although the demand for sulphuric acid has been somewhat better there is still room for considerable improvement, but it is doubtful whether we are likely to see the sulphuric acid plants of the country fully employed again until such time as there is appreciable improvement in the world value of nitrogen, thereby enabling coke-ovens and gasworks to make sulphate of ammonia profitably. By-products and tar products are

still true to their reputation in that the markets for them fluctuate considerably. The high price ruling at the beginning of the year for pure toluol has resulted in an increase in the output and the price has now come back to a more reasonable level. The same remarks apply to xylol. It is fortunate for the coke-ovens interests that, as so many of the rubber works are now using white spirit instead of solvent naphtha, the cellulose lacquer trade has developed so well and is absorbing so much of what would have been made into solvent naphtha in the form of xylol.

In the tar products market, tar acids have not done very well for the producer and the pitch market is developing considerable weakness, owing to the increased production and the larger quantities of pitch which are now being shipped from America to the European markets. Not only has America shipped pitch to Europe but she has also shipped considerable tonnages of tar, as, owing to the low prices ruling in America for fuel oil, it has been found more profitable to export tar and pitch than to burn the tar in the steel furnaces as was formerly done. Fortunately for the tar distillers the creosote market has improved considerably. Large quantities have been sold for burning purposes in this country to replace imported fuel oil and a considerable quantity will also be hydrogenated. In addition, there is now an improved demand from America and but for the strength of the creosote position the immediate outlook for tar would be very poor indeed. It is perhaps a little encouraging to reflect that during the slump period, tar products did not suffer nearly so badly as most of the other markets, and, of course, with an improvement in the iron and steel trade there is an automatic increase in the output of tar; and unfortunately the demand for tar products, apart from creosote, has not kept pace with increased output. However, the tar trade is quite used to these fluctuations.

Whilst the Staveley Coal and Iron Co. has made no additions to its chemical products during the past year, they are at the moment seriously considering extensions, and the addition of new manufactures.

### F. Jahn and Co.

THIS firm reports that there has been a marked increase in inquiries for specially designed modern plant for a variety of purposes, including quite a few from foreign countries and the colonies. They have shipped one of their latest type London-built evaporating plants to Australia. On account of patented features, the Australian Government granted a licence for import free of customs duty. Several vacuum evaporating plants for large output, including such in quadruple effect were built during the year for home factories. Prospects for further contracts, including such from abroad look promising for the new year. The difficulty of welding stainless steel tubes into stainless steel tube plates at both ends and allow for barrel expansion has been overcome. This is considerable advance for milk cooling with ammonia from the compressor at 300 lb. pressure in a cooling unit.

### Johnson, Matthey and Co., Ltd.

DURING the past few years the chemical industry, by virtue of increased efficiency in manipulation by plant manufacturers, is realising the benefits gained by the use of silver as a lining material for much of their plant. As regards its resistance to corrosive attack, sterling silver with its purity of 92.5 per cent. cannot be compared with the product which is 99.97 per cent. pure, known as "Mattheylic" silver, which is electrolytically refined by Johnson, Matthey and Co., Ltd. Special processes developed by this company have made it possible to apply "Mattheylic" silver in the form of a thin lining in such a way that the lining becomes entirely and intimately united with the base metal from which the plant is constructed. This method of lining plant with homogeneous sheet silver must not in any way be confused with electro-deposition of silver, which is an entirely different process and which is porous. Where joints are unavoidable, the autogenous welding method obviates all risks of local corrosion and contamination which occur with soldered or brazed joints. Among other important contributions to the chemical and allied industries is the research work done by Johnson, Matthey and Co., Ltd., on solders. The addition of silver to the brazing alloys introduces advantages in that the solders have lower melting points, increased tensile strength and

greater resistance to corrosion. Considerable success has attended these endeavours and "Sil-fos" is satisfactory for copper and brass; others for the jointing of stainless steels and steel have given highly satisfactory results. Work has also been done on soft solder with the object of producing alloys which melt at slightly higher temperatures than the ordinary tin lead compositions and which at the same time will possess greater tensile strength and corrosion resistance. Solders of this order are of particular interest to the electrical industry where loads on the low melting point soft solders may result in breakdowns. In preparing for 1935, the company has entirely re-designed and enlarged its shops for the construction of process plant in "Mattheylic" silver, and its technical staff has been considerably augmented to investigate the problems arising both from the application of silver for the chemical and allied industries and problems which arise in connection with the jointing of metals.

### Alcock (Peroxide), Ltd.

THIS firm specialises in the manufacture of sodium metasilicate, which it first introduced to British industry several years ago. The year 1934 has seen a steadily increasing demand for this product. The laundry industry in particular has been quick to seize upon the advantages derived by its use. The powerful detergent properties of metasilicate are tempered by the silica content which protects the fibres against loss of strength, prevents the redeposition of dirt on the goods, and reduces the possibility of staining by iron rust to a minimum. These properties promise to be of service for some of the cleansing operations in the textile industry, and whilst it is too early to say what the possibilities are in this direction, it is believed that the investigations proceeding in this country and abroad will lead to increased consumption.

Another direction which shows promise of expansion is in the preparation of proprietary cleansing compounds, as manufacturers of such products are beginning to appreciate that the addition of metasilicate offers advantages not hitherto available. For the removal of oil and grease from metal without danger of corrosion sodium metasilicate has found a welcome place in some of the largest car factories in the country.

During 1934 the factory of Alcock (Peroxide), Ltd., has been enlarged and plans are ready for a further increase of production at short notice.

### The British Drug Houses, Ltd.

CHEMICAL research and modern analytical practice make it extremely important only to employ reagents which conform to definite and precise standards of purity. During the past year, two British manufacturers, The British Drug Houses, Ltd., and Hopkin and Williams, Ltd., have published, under the title "Analar" Standards for Laboratory Chemicals," full details of tests and specifications for the characterisation of 220 chemicals chosen for their importance in scientific work. This publication marks a very real step forward in the definition of the purity of chemical reagents, and it has received warm compliments from every quarter. These specifications are definite and precise, and denote throughout an accurate knowledge of the recent advances in analytical technique. The British Drug Houses, Ltd., have issued for sale these 220 chemicals guaranteed to be in conformity with the new specifications, and these are sold under the name "Analar" chemicals.

Another useful volume for laboratory work, lately issued by The British Drug Houses, Ltd., is "The B.D.H. Book of Reagents for 'Spot' Tests and Delicate Analysis." This contains details of 60 valuable and interesting organic reagents for use in micro-analytical work. The B.D.H. also issue a compact spot test outfit which provides chemists with a collection of 27 of these reagents and the accessories required for their use. Analytical chemists and all who are engaged in schools and colleges and desire to demonstrate the technique of micro-analytical work will find this outfit useful.

The colorimetric determination of  $\beta\text{H}$  is becoming increasingly important in the control of many industrial operations. The Lovibond Comparator, designed jointly by The Tintometer, Ltd., and The British Drug Houses, Ltd., provides chemists with permanent colour standards prepared with Lovibond glasses. The standards have been established by comparison with solutions of known  $\beta\text{H}$  and

B.D.H. indicators. The B.D.H. also supply a comprehensive range of oxidation-reduction indicators for bacteriological use and for metallurgical use. Chemists interested in the use of these indicators should apply to them for a copy of a 16-page booklet describing the theory and use of these indicators. The booklet is entitled "The Colorimetric Determination of Oxidation-Reduction Balance," and is supplied free of charge.

Among other novelties included in the latest edition of the B.D.H. Price List of Laboratory Chemicals is a remarkable new method of determining minute traces of chlorine by the use of a special solution sold under the name of the B.D.H. Chlorotex reagent. It affords a simple means of determining the amount of residual chlorine in swimming pools and in drinking water purified by chlorination. Five cc. of the Chlorotex reagent is mixed with 50 cc. of the water and the amount of chlorine present is indicated by the colour of the solution. In the absence of chlorine, a slightly milky solution with a blue fluorescence is produced; with 0.2 to 0.5 part per million the colour is from pink to red, with 0.6 part, the colour is purple, while with 1 part a blue colour is formed.

### International Pulverisers, Ltd.

THIS company manufactures the well-known Pulmac mill, and is now completing its second year of existence. It will be remembered that they purchased the patents of the International Pulverising and Grinding Machines (Parent) Corporation, Ltd., who went into liquidation in 1932. When the company was registered, the Pulmac mill was manufactured in Belgium, but this mill is now entirely British-made. During the last year important contracts have been placed for the Pulmac mill not only in this country but also abroad, notably in Algiers, France, India, New Zealand, America, Norway, Singapore and Australia. The company are now supplying a laboratory size Pulmac mill, which will fulfil a long-felt need not only in the chemical industry but in every other industry that requires materials reduced to a fine powder or granulation. They have a demonstration plant in London and welcome the opportunity of showing their full range of mills and demonstrating capabilities. The managing director of this company (Mr. A. L. Dix-Perkin) is a great-nephew of Sir William Perkin, who was a pioneer in the making of coal tar dyes.

### Negretti and Zambra

DURING the past year, temperature recorders have received special attention from Negretti and Zambra. The "Mersteel" temperature recorder is constructed on the mercury expansion principle and is suitable for temperatures between  $-39^{\circ}$  and  $1,200^{\circ}$  F. It is twelve years since this firm produced its first patent mercury-in-steel temperature recorder. Many hundreds of these temperature recorders are now in use, and it is safe to say that the only bar to their universal adoption has been the comparatively high cost. The firm has therefore introduced a new pattern, which, though constructed on the same mercury expansion principle, is of an entirely new design with the object of more economical production consistent with equal accuracy and reliability. This new recorder is a triumph of advanced instrument making as the production methods of each part have been studied and no expense has been spared in obtaining the most suitable plant in order to effect a large reduction in cost without a sacrifice of quality. The special merits of the "Mersteel" temperature recorder are moisture-proof case, rust-proof movement, micro-bore connecting tubing, grooved-edge bourdon tube, and artificial ageing of the movement.

### Boots Pure Drug Co., Ltd.

THE improvement in general trade during 1934 has reflected itself in increased production in all the manufacturing departments of Boots Pure Drug Co., Ltd. In particular, the output and sales of fine chemical and special medical products has shown quite a considerable increase over last year's figures. Such well-known products as aspirin, chloroform, Glauber salts and bismuth preparations are now manufactured in very large quantities. Perhaps the most important developments during the year are associated with Boots special medical products for use under medical direction. In connection with these products much research work has been carried out for the improvement of products and manufacturing processes. Insulin (Boots) is manufactured under the most strict scientific-

ally-controlled conditions which ensures that a product of the highest possible purity is available for the treatment of diabetes. The staff of the ampoule filling and packing departments has been considerably increased to meet the rapidly developing demand for sterile ampoule products. Recently issued new products are an improved Stabimol, which is a solution of a new bismuth compound in olive oil for the treatment of spirochaetal diseases, and Burnol—a creamy emulsion containing acriflavine—the powerful antiseptic which is unequalled for use as a first-aid dressing for burns, wounds, and general septic conditions. Burnol is supplied in handy collapsible tubes. The institution of the 5-day week has been a great success. All aspects of its working have been dealt with in a report drawn up by Sir Richard Redmayne.

### Sofnol, Ltd.

THE 1934 trade revival has affected this company in so far as it has been doing increased business in all products during the past twelve months, both in the supply of materials for water treatment and water testing, and also in general horticultural business. Considerable extensions have been made to the works in Anchor and Hope Lane, Charlton, in order to cope with increased business, and new and more up-to-date plant has been installed both at Charlton and also at the works at Greenwich, where soda lime is manufactured in various grades. Both of the company's factories have been kept busy in order to cope with the increased demand for this product.

### British Hydrological Corporation

CLEANSING and sterilising compounds for various industries are manufactured by this company. It reports that 1934 has been the largest trading year in its history. Extensions of plant have been made during the current year and a 50 per cent. increase is scheduled to operate as from the beginning of January, 1936. Whilst the whole of this extension will not be made use of immediately, the greater portion of it will, and the history of the past year suggests that the output will continue to increase and that the complete plant will be absorbed completely during the next twelve months. Increase in output has not been concerned only with home trade, since the export trade has very considerably increased, too, and at the moment the managing director of the firm is now in the United States in connection with further extensions of the branch of the organisation over there.

### John W. Leitch and Co., Ltd.

THIS firm, which manufactures intermediate products and dyes, has made substantial progress during the year in developing the dyestuffs section of the business. This has been the case, particularly in respect to the fast bases (ice colours), of which a most comprehensive range is now available. Dyestuffs produced on the fibre from these bases are finding an ever-extending market, largely due to the very good fastness to light and washing which such colours possess. This method of dyeing is now also available to wool by Leitch's patented process, thus making possible the production of shades on wool equal in brightness to the acid dyestuffs, with the fastness properties of the vat or best chrome colours. It is expected that the new plant will be in operation early in 1935. An up-to-date and efficient laboratory is installed to assist customers in their choice of products and to test the materials at every stage of manufacture.

The older side of the business of John W. Leitch and Co., Ltd., which is engaged in the manufacture of intermediate products for coal tar dyes and nitro products for the explosives and allied trades is not being neglected, and new products are put on the market as occasion arises.

### George Kent, Ltd.

THIS firm has been specialising in flow metering for the past fifty years, and are now the largest organisation of their kind in Great Britain. They have developed a complete range of flow, pressure, temperature and level recorders, designed with a view to obtaining uniform appearance when mounted on instrument panels. The success of these meters, coupled with the handsome appearance of the steel panels, has led to rapidly increasing demand. At their works at Luton, a new assembly and testing bay is being constructed for complete metering schemes.

George Kent, Ltd., offer three alternative finishes for their instrument panels, namely, black semi-matt, black glossy or crystallate, the first being generally recommended as the most serviceable. The edges of the panel are bound by a highly polished stainless steel beading. The Kent automatic boiler controllers are designed to fit into this scheme and a notable example of such an installation is at Roath Power Station, Cardiff, where the panel containing the master controller and fan speed regulators is situated on the firing floor. Another example of Kent automatic boiler control combined with meter panel schemes are to be found at Thomas Headley's soap factory, Manchester. A type of meter which is widely used for checking and measuring the distribution of steam is the Kent shunt steam meter. Many recent orders have been received, including 10 meters for the British Dyestuffs Corporation, and 8 meters for the General Electric Co., Coventry. At the present moment there are orders for over 100 steam meters on hand.

### The International Electrolytic Plant Co.

VERY satisfactory progress during 1934 is reported by The International Electrolytic Plant Co., and there is every reason to expect that the rate of progress will be maintained during the coming year. The demand for pure hydrogen and oxygen is steadily increasing and important contracts are in hand for countries as far distant as Mexico, Peru and South Africa. By far the largest use for hydrogen is in the production of synthetic ammonia for artificial fertilisers, but since 1933, when a large Knowles electrolytic plant was put into operation in Japan for this purpose, there has been little demand from that industry. On the other hand, requirements for the oil hardening industry show an upward trend. Recent developments in the metallurgical industry have created a demand for both hydrogen and oxygen of the highest degree of purity. The Knowles cell, which is the only electrolyser for the decomposition of water manufactured in this country, produces the two gases of the quality required for the most delicate catalytic and metallurgical processes. Research work is continually in progress with the object of increasing efficiency while maintaining simplicity of design.

### The Steel Barrel Co., Ltd.

DURING the past year, the Steel Barrel Co., Ltd., has doubled its production capacity and has had very great success, owing to putting in the most modern equipment. A new double seaming machine for 10-gallon drums has been installed, which is capable of an output of 2,500 drums per day and a smaller machine to cope with the demands for 5-gallon drums with the same output. There has also been installed a new double seaming machine for the output of 40- and 45-gallon drums with a capacity of 1,000 drums per day. Many large contracts have been secured from the principal oil companies and important Government Departments.

The Steel Barrel Co., Ltd., has had over 40 years' experience in the manufacture of containers of all kinds and has originated a great many of the designs which to-day are the practice for fluid transport. It has brought out a type of drum for carrying powder, which has been largely used in industries of this description. The manufacture of heavy steel barrels has been increased during the past year and new purposes are being found for very heavy containers which have a life of over 20 years, and the company is at present repairing some of its barrels manufactured 25 years ago. The company has also experimented, in connection with many of the chemical manufacturers, in the manufacture of stainless steel barrels and has now brought out a successful type which is meeting with an increasing demand for these specialised trades.

### Peter Brotherhood, Ltd.

THE year 1934 has been a very busy year for this company, who have found a ready market, both home and abroad, for "Brotherhood" compressors and turbines, steam engines and refrigerating plant, and "Brotherhood-Ricardo" high-speed diesel engines. During 1933, when they were not so busy, they took the opportunity of bringing all their manufacturing, testing and power plant up-to-date. Some shops were rearranged, many new machine tools and welding sets were bought, several Brotherhood-Ricardo diesel engines were

installed in the power house to provide the power for the shops at the cheapest possible rate, and, to enable the works to test steam power units using high pressure steam, a water-tube boiler having a normal working pressure of 600 lb. per square inch, and fitted with a superheater to raise the temperature of the steam to 750° F., was installed. These preparations have enabled the company to cope with the large amount of work that has subsequently come their way.

Important gas and air compressor contracts, either just completed or in hand, include machines for The Union Cold Storage Co., Ltd.; The Whitwood Chemical Co., Ltd.; I.C.I. (Alkali), Ltd.; Scottish Oils, Ltd.; The Liquid Oxygen Co., Ltd.; The Mond Nickel Co., Ltd.; The Washington Chemical Co., Ltd.; British Titan Products, Ltd.; Tata, Ltd. and The British Xylonite Co., Ltd. Contracts have also been received for a great deal of fabricated steelwork, a number of water circulating pumps, fans, water cooling towers, and homogenisers.

### W. M. Fuller, Junr.

THE firm of W. M. Fuller, Junr., manufactures end-runner grinding mills, sample crushers and sample grinders. The patented end-runner grinding mill is really a mechanically-operated pestle and mortar. The smaller machines are hand-operated, the larger ones are run by power and have ball bearings. They are suitable for manufacturing chemists, colour manufacturers, enamels, food products, toilet pastes and creams, etc.; for grinding and mixing in dry, wet or plastic condition; and for use in preparing samples of solid fuel for analysis. The Breakwell sample crusher is used for reducing all kinds of ore and fuel samples to  $\frac{1}{4}$  in. or less; it can be had in two sizes, has steel wearing parts, and can be operated by hand or power.

### Daniel Adamson and Co., Ltd.

THIS firm reports considerable demand during the year for its complete power station equipment, comprising Lancashire, super Lancashire, Cornish and Dryback boilers, steam turbines, turbo alternators, turbo generators, turbo compressors, condensing plant, steam raising plant, sewage lifting plant and auxiliaries. The Adamson 84 years' record is one of which any firm may be proud, and what is remarkable is that with all its long and splendid history it continues to be up to date in every respect, its factory equipment being of the most modern type. Its technical service is freely at the disposal of power plant engineers and others, with regard to the selection of the most suitable type of plant for any particular purpose, and no trouble is too great to render Adamson products adequate to the needs of users. Wherever steam power plant is used the name of Adamson is held in the highest regard by reason alike of the high quality of their plant and the reliability of its service. The year has been notable for the introduction of small steam turbines and rotary air compressors for which there has been quite an appreciable demand. The company has a number of large contracts in hand and the prospects for 1935 appear to be bright.

### John Thompson (Dudley) Ltd.

THE John Thompson group deals with vessels for chemical plants and processes, and has been considerably busier during 1934, so that they have been able to re-absorb all their skilled workers and have had to start training more. The homogeneous lead lining department has more than doubled the output of its previous records, and has employed an increased number of men and for much longer periods than before; this in itself is a tribute to the maintenance of the very highest quality in spite of trade depression in the slack period. The output of stainless steel and heat-resisting steels continues to increase, and during 1934 the firm has made what it believes to be the largest monel metal electrically-welded vessel ever made in this country, this being 9 ft. 6 $\frac{1}{2}$  in. diameter by 12 ft. 6 in. total length. It is being found that nickel-clad steel is now a definite commercial proposition, and orders for larger equipment have been booked this year than in 1933. The demand for vessels in Inconel (the latest product of Henry Wiggin and Co.) is also increasing, two large vessels having been made in this material. High-pressure

work has likewise received attention, riveted vessels being constructed of large size to withstand 400-lb. working pressure. The question of heavy welding has received considerable investigation during 1934, a vessel 12 ft. diameter by 24 ft. long, from material  $1\frac{1}{2}$  in. thick, being an example of this class of work.

### Henry Wiggin and Co., Ltd.

THE market for malleable nickel, and the nickel alloys, Monel metal and Inconel, has expanded during 1934. Further orders for Monel metal equipment for use in the manufacture of tartaric acid have been completed, while both Monel metal and nickel equipment have been supplied for the production of aspirin. Developments in the canning and food industries have resulted in substantial orders for both Monel metal and Inconel plant, the former for various established uses, such as syrup boiling pans, the latter mainly for special products involving particular corrosion problems. The manufacture of pharmaceuticals, cosmetics and tooth paste has also necessitated new Monel metal and nickel equipment. During the year the first important piece of British-built Inconel equipment installed in a domestic fruit juice factory was examined and after two years no sign of corrosion was manifest. The

number of inquiries received regarding this material shows there are many corrosion problems where its properties appear likely to be of value. Prospects for the coming year are indicated by the fact that duplication of two or three of the largest installations made during 1934 are planned.

### Hedley and Co. (Leytonstone), Ltd.

THROUGHOUT the past year the firm of Hedley and Co. (Leytonstone), Ltd., has continued its manufacture of the halogen derivatives of the hydrocarbons. In bulk quantities these compounds find wide application in the dye, fine chemical and refrigerating industries, whilst the pharmaceutical department of the firm retains its activity in preparation on a large scale of ethyl chloride tubes of various sizes and construction for "local" and "general" anaesthesia. The "Dridustsol" preparations are also worthy of mention; these consist of solutions of various drugs in ethyl chloride, which are sprayed and deposited at will in a fine dry film on the surface to be treated. Reference to the extraordinarily effective fire extinguisher which Hedley and Co. are just beginning to market must not be omitted. Many testimonials from satisfied users have been received with reference to this original self-acting fire extinguisher.

## Key Industry Duty

### Renewal of Exemptions

THE Treasury has made an Order under Section 10 (5) of the Finance Act, 1926, continuing the exemption from Key Industry Duty till December 31, 1935, of the following articles. The Treasury Order will shortly be published by H.M. Stationery Office.

Lamp-blown glassware.—Dolls' eyes.

Scientific instruments.—Integrators (planimeter type); fermentographs, being instruments for measuring and recording carbon dioxide evolved during dough fermentation.

Vacuum Tubes.—Mercury vapour rectifiers having mercury cathodes, of a capacity of more than 300 amperes, and having more than three main anode terminals.

Compounds of rare earth metals.—Celtium oxide; dysprosium oxide; erbium oxide; europium oxide; gadolinium oxide; holmium oxide; lutecium oxide; neodymium oxide; praseodymium oxide; samarium oxide; scandium compounds; terbium oxide; thulium oxide; Ytterbium oxide; Yttrium oxide.

Synthetic organic chemicals, analytical reagents, other fine chemicals and chemicals manufactured by fermentation processes.—Acid adipic; acid flicic; acid propionic; acyl derivatives of urea, the following: acid isobutyl allyl barbituric, sodium ethyl methyl butyl barbiturate; alcohol amido ethyl; amido guanidine sulphate; amidopyrin (pyrimidin; dimethylamidoantipyrine); ammonium perchlorate; barbitone (veronal, malonal, malourea, acid diethyl barbituric, diethylmalonylurea, hypnogen, deba); bromural (dormigene); butyl esters, the following: butyl methyl adipate; calcium gluconate (calcium glyconate); cellulose ethers, the following: ethyl cellulose, methyl cellulose; chinoline (quinoline); chinisol; cocaine, crude; cyclohexanol esters and alkyl cyclohexanol esters, the following: methyl cyclohexanol methyl adipate; dial (acid diallyl barbituric); dicyandiamide; didial (ethyl morphine diallyl barbiturate); dimethyl sulphate; diphenyl; diphenyl oxide; elbon (cinnamoyl para oxyphenyl urea); ethyl esters, the following: ethyl abietate; ethylene bromide; ekodal; fufural; germanium oxide; glycol ethers; guaicol carbonate (duotal); lead tetraethyl; lipoidin; metaldehyde; methyl esters, the following: oxymethyl para-oxyphenyl benzylamine methyl sulphate; methyl sulphonol (diethylsulphonemethylmethane, trional); methylene chloride; nickel hydroxide; organo-arsenic compounds, the following: copper methyl arsenate; phenazone (antipyrine, phenyl dimethylpryrazolone, analgesin, anodynine, dimethyl oxychinin); phenetidine, para-; phloroglucine; phytin; piperazine (diethylene-diamine, dispermin); R. potassium chlorate; potassium ethylxanthogenate (potassium xanthogenate); potassium guaicol sulphate; R. potassium hydroxide (R. potassium caustic, R. potassium hydrate); R. potassium permanganate; pyrimidin-veronal; quinine ethyl-carbonate; radium compounds; salol (phenyl salicylate); strontium carbonate, strontium nitrate, sulphonol; synthalin; urea (carbamide).

Amorphous carbon electrodes over three feet long, the cross section of which exceeds 12 inches in both length and breadth with longitudinal slots exceeding two inches in width and four inches in depth.

Vanadium compounds.—Vanadium-silica compounds specially prepared for use as catalysts for sulphuric acid manufacture.

## British Industries Fair, 1935

### Preliminary List of Chemical Exhibitors

PREPARATIONS are already well in hand for the British Industries Fair, 1935, and preliminary lists of exhibitors have been issued. Those in the chemical section at Olympia will include the following:—

Allbright and Wilson, Ltd.  
Association of British Chemical Manufacturers.

A. Boake Roberts and Co., Ltd.

British Drug Houses, Ltd.

British Titan Products, Ltd.

W. J. Bush and Co., Ltd.

Alex. Finlay, Ltd.

Gas Light and Coke Co.

General Chemical and Pharmaceutical Co., Ltd.

High Speed Steel Alloys, Ltd.

Hopkin and Williams, Ltd.

Howards and Son, Ltd.

Imperial Chemical Industries.

Imperial Smelting Corporation,

Ltd.

Johnson and Sons Manufacturing Chemists, Ltd.

Kestner Evaporator and Engineering Co., Ltd.

B. Laporte, Ltd. (sharing with

Malehurst Barytes Co., Ltd.,

National Titanium Pigments,

Ltd.).

In the druggists' sundries

section the exhibitors will

include:

Anzora Perfumery Co., Ltd.

L. Batley and Co.

Beaucaire Laboratories.

British Fumigants Co., Ltd.

G. Cooper Beauty Preparation,

Ltd.

Corfield, Ltd.

P. B. Cow and Co., Ltd.

Cussons, Sons and Co., Ltd.

Drayton Paper Works, Ltd.

Druggists Specialities, Ltd.

Floragen Works.

Gen. Kaputine Syndicate, Ltd.

Adelaide Grey, Ltd.

Hopes Soaps.

F. Hulse and Co., Ltd.

Newton Chambers and Co., Ltd.

Pioneer Magnesia Works.

Powell Duffryn Steam Coal Co.,

Ltd.

Society Chemical Industry.

South Metropolitan Gas Co.

Spencer Chapman and Messel,

Ltd.

D. Thom and Co., Ltd.

T. Tyrer and Co., Ltd.

Viscose Development Co., Ltd.

Watson Laboratories, Ltd.

Willifen and Sons, Ltd.

Williams (Hounslow), Ltd.

Firms showing with Imperial

Smelting Corporation, Ltd.—

Cuprinol, Ltd., The Delaville

Spelter Co., Ltd., Fricker's

Metal and Chemical Co., Ltd.,

Improved Metallurgy, Ltd.,

National Alloys, Ltd., The

National Smelting Co., Ltd.,

Orr's Zinc White, Ltd., Nor-

thern Smelting and Chemical

Co., Ltd.

Jetglaze, Ltd.

Manon Freres, Ltd.

Meritex, Ltd.

Chas. Midgley, Ltd.

Ozonal Laboratories, Ltd.

Potter and Moore, Ltd.

Reliance Rubber Co., Ltd.

Sharmek Biological Cosmetics,

Ltd.

Skuse and Co., Ltd.

Solport Bros., Ltd. (sharing with

C. Keith Thomas, Ltd.).

A. Stockton, Ltd.

Talbot Manufacturing Co., 1926,

Ltd.

Zenobia, Ltd.

SULPHURIC acid production in Canada has doubled in the last decade to 148,142 short tons in 1933, as compared with 136,846 tons in 1932 and 71,091 tons in 1924. Of the 1933 total, 65,927 tons were made for sale and 82,215 tons for use of the makers in their own plants.



## Personal Notes

MR. FRED W. BAIN has been appointed chairman of the General Chemicals Group of Imperial Chemical Industries, Ltd., in succession to Mr. Holbrook Gaskell.

MR. JOHN BAIRD, Ellinor Cottage, Linlithgow, has died at the age of 70. He was formerly chief foreman in Nobel's Regent factory and retired in May, 1929, when he received a presentation after 21 years' service.

MR. W. W. WATT, chairman and managing director of Ogston and Tennant, Ltd., of Renfrew and Aberdeen, has accepted the vice-chairmanship of the management committee of Lever Brothers, Ltd., at Port Sunlight.

### Held Over

Owing to pressure of space we have been compelled to hold over until next week a review of "Chemistry in Agriculture in 1934," by Mr. H. J. Page, controller of the I.C.I. agricultural research station at Jealott's Hill, and an article on "Processing Plant in the Food Factory," by Mr. P. A. Farmer.

MR. DUNCAN MCGLASHAN, M.P.S., of Kingscroft, Barnton, Midlothian, and of Cramond Bridge, Edinburgh, manufacturing chemist, founder of Duncan McGlashan, Ltd., abidine works, Edinburgh, left personal estate valued at £38,961.

MR. ROBERT RATTRAY TATLOCK died on December 22. He was 97 years of age and was public analyst for Glasgow for 50 years, serving in a similar capacity the county of Dumbarton, Stirling, the city of Perth and a number of other burghs. He was well-known for his contributions to chemical literature, and was greatly sought after for expert evidence in legal cases. In this connection he played an important part in the famous case of Nobels versus the Government, and also in a historical gold dispute in South Africa. While associated with Dr. Penny, of Anderson's College, he was responsible for conducting the intricate analyses required by the Crown authorities in the trial of Dr. Pritchard.

## Chemical Trade Inquiries

The following trade inquiries are abstracted from the "Board of Trade Journal." Names and addresses may be obtained from the Department of Overseas Trade (Development and Intelligence), 35 Old Queen Street, London, S.W.1 (quote reference number).

**Canada.**—A firm of metal importers and manufacturers' agents at Montreal desires to obtain United Kingdom agencies for cream of tartar, citric and tartaric acids, and lithopone, presumably on a commission basis, throughout the Dominion.

**Holland.**—An agent recently established at Amsterdam wishes to obtain the representation on a commission basis of United Kingdom manufacturers of chemicals, colours, oils and greases.

**Brazil.**—A firm of commission agents established in Brazil wishes to obtain the representation of United Kingdom manufacturers of industrial and pharmaceutical chemicals and drugs.

## New Companies Registered

**Analytical and Synthetic Laboratories, Ltd.**, 5 Water Lane, London.—Registered December 20. Nominal capital £600. Analytical, synthetic, technical, research and consulting chemists, manufacturers, importers, and exporters of and dealers in all chemical, metallurgical, pharmaceutical, petroleum and coal tar products, etc. Directors: Henry J. Fitz Weiss, Charlotte T. Werner.

**Granville Wood and Co., Ltd.**, Ashcroft Street, Oldham.—Registered December 20. Nominal capital, £10,000. To acquire the business of a manufacturing chemist now carried on by Mrs. Ann Wood at Oldham, as "Granville Wood & Co." Directors: Walter E. Williams, Edward C. B. Shelbourne, Robert E. Pearmund.

**Richard Poolton and Sons, Ltd.**, 80 Hurst Street, Birmingham.—Registered December 15. Nominal capital £3,000. To acquire the stock-in-trade, plant, etc., of tanners of all kinds of copper, brass and iron tubes, sheet metal and wire and small work of every

## Mining Research

### Progress at Birmingham University

ACCORDING to the annual report on the work of the Mining Research Laboratory of Birmingham University during 1933 investigations have been continued of the quantity and chemical composition of the fine dust suspended in mine air, and tests to ascertain the quantity and free silica content of the dust have been carried out in a number of pits in the Midlands, Lancashire and South Wales. The accumulation of the desired data is of necessity slow owing to the time taken to collect sufficient dust for an analysis, and to obtain a quantity sufficient for one analysis by the modified "micro" method of rational analysis the dust collecting plant has frequently had to be run for 500 hours, which is equivalent to about eleven weeks, if only one shift is worked per day.

### Compressed Gas for Transport

In last year's report it was pointed out that the use of compressed coal gas for motor vehicles propelled by internal combustion engines could be made more certain of success if by a reasonably cheap method the calorific value could be raised much over that of the ordinary town's gas supply. With this object various experiments were undertaken to increase the methane content of coal gas.

Investigations on spontaneous combustion and the occurrence of dangerous atmospheres in underground workings have included the continuation of laboratory tests to measure the oxidisability at different temperatures of various sections of strata from the neighbourhood of seams in which spontaneous heating has occurred, the production of oxides of carbon relative to oxygen absorption, the influence of moisture on the oxidation of coal and correlation of the results with underground analyses.

Other investigations have shown that the oxidation of anthracite is much increased by the presence of moisture, the carbon dioxide production expressed as a percentage of oxygen absorbed being on the average about doubled and the CO production much reduced with an atmosphere saturated with moisture.

description, lately carried on at 80 Hurst Street, Birmingham, by G. E. Poolton and G. E. Poolton, junr., as Richard Poolton and Sons. Directors: George E. Poolton and George E. Poolton, junior.

## Forthcoming Events

### BIRMINGHAM.

**Jan. 3.**—Electrodepositors Technical Society. Joint meeting with the Midland Metallurgical Societies. "The Microstructure of Electrodeposited Coatings." A. W. Hothersall. 7.30 p.m. James Watt Memorial Institute, Gt. Charles Street, Birmingham.

### BRADFORD.

**Jan. 3.**—Society of Dyers and Colourists (West Riding Section). "Experimental Researches on Lubrication—Some Properties of Thin Oil Films." J. E. Southcombe. Bradford.

### LIVERPOOL.

**Jan. 4.**—Society of Chemical Industry (Liverpool Section). "Food as Colloid Systems." Dr. W. Clayton. 6 p.m. University, Liverpool.

### MANCHESTER.

**Jan. 4.**—Society of Chemical Industry (Manchester Section). "The Chemical Engineer and His Training for Industry." Dr. A. J. V. Underwood. 7 p.m. 17 Albert Square, Manchester.

### TROWBRIDGE.

**Jan. 2.**—Institution of the Rubber Industry (West of England Section). "Raw Rubber Production." G. E. Coombs. Town Hall, Trowbridge.

### WOLVERHAMPTON.

**Jan. 3.**—Institution of the Rubber Industry (Midland Section). "Modern Foremanship." F. E. J. Hunt. Victoria Hotel, Wolverhampton.

## From Week to Week

THE INTERNATIONAL ZINC CARTEL, it is understood, will definitely terminate on December 31.

THE NOMINAL CAPITAL of Chemical Building Products, Ltd., has been increased by the addition of £1,400 beyond the registered capital of £2,000.

THE MAGNESIUM METAL CORPORATION, LTD., has increased its nominal capital by the addition of £100, which is divided into 100 "A" shares of £1.

"THE JOURNAL OF SCIENTIFIC INSTRUMENTS" will, after January 1, be published by the Institute of Physics, 1 Lowther Gardens, Exhibition Road, London, S.W.7.

BUSINESS VISITORS to Newfoundland will be aided by a memorandum for their use which has been issued by the Department of Overseas Trade.

QUININE WORTH £18,750 has been lent to Ceylon by the Indian Government in order to help the fight against the malaria epidemic which has broken out there.

THE UNITED MOLASSES CO., LTD., held its ninth annual general meeting at Irving Hall, Bush House, London, on December 19, when the chairman, Mr. F. K. Kielberg, announced a 6 per cent. dividend.

THE COUNTY CHEMICAL CO., LTD., of Birmingham, is to have a large extension to its premises on the North Circular Road, N.W. A contract has just been placed with Commercial Structures, Ltd., who will commence operations immediately.

A NEW POISON, reported by Mr. Sam O. Williams, of Newcastle-on-Tyne, would, he states, rid South Africa of locusts within three years. The spraying of locusts with arsenite of soda is only partly satisfactory, and Mr. Williams is returning to Africa in February to continue negotiations for the sale of the poison by William Cail and Partners for £500,000.

NARROW ESCAPES WERE EXPERIENCED by several workmen at the Istook Brick and Pipe Co.'s works in Leicestershire on December 20, when the skeleton of a steel building 200 ft. long and about 30 ft. high, weighing 25 tons, collapsed. The accident to the structure, the framework of a new tunnel kiln, is believed to have been due to a strong gust of wind.

THE COMMERCIAL SECRETARY to the Residency, Egypt, reports that the Department of Public Health is calling for tenders, to be presented in Egypt by March 4, 1935, for the supply of 32 microscopes and accessories. Firms desirous of offering instruments of United Kingdom manufacture can obtain further particulars, together with particulars of the Department's Special Register service of information, upon application to the Department of Overseas Trade, 35 Old Queen Street, London, S.W.1. Reference number B.Y. 7943 should be quoted.

WEST RIDING INSURANCE COMMITTEE, meeting at Wakefield on December 20, agreed to the following resolution:—That there should be an addition to the terms of service for persons supplying drugs and appliances whereby a chemist shall notify the Insurance Committee of any arrangements which he has made with another chemist for the dispensing of medicines when he is unable to undertake the work himself; and that he shall not absent himself from his shop for more than one week without first informing the Committee of his proposed absence and of the name of the chemist responsible for undertaking the dispensing of medicines during such absence.

THE INSTITUTE OF CHEMISTRY (Newcastle and North-East Coast Section) held a discussion on the report of the Government Commissioner on the Durham and Tyneside distressed area on December 13, following an informal dinner. The principal speakers were Dr. P. L. Robinson, Mr. J. W. Craggs, Dr. A. A. Hall and Professor G. R. Cleme, and after an interesting discussion the following motion was carried unanimously:—"That the hon. secretary be directed to approach the Newcastle Section of the Society of Chemical Industry with a view to forming a joint committee to seek to increase the application of science to the co-ordination and development of industry in this area."

VARIOUS ASPECTS of the application of chemistry to the uses of the community were dealt with by Mr. A. R. Jamieson, City Analyst's Department, when he addressed the members of the Glasgow Business Club on December 20 at their weekly luncheon meeting. Mr. R. A. Raphael presided. Referring to the production of oil from coal, Mr. Jamieson said that this development would undoubtedly revolutionise both the coal industry of this country and the petroleum industry. The plant in course of construction, however, would only be capable of producing something like one-thirtieth of our home needs in petrol, so that there was still a long way to go. Dealing with the analyst's work in connection with food, Mr. Jamieson said that in 1914 the percentage adulteration in milk in Glasgow was round about 25 per cent. Now the figure had dropped to less than 3 per cent.

THE INSTITUTION OF THE RUBBER INDUSTRY has changed its address to 12 Whitehall, London, and its telephone number to Whitehall 5012.

EMPLOYEES of George MacLellan and Co., Ltd., Glasgow Rubber and Asbestos Works, have donated £100 to Glasgow charities.

THE NOMINAL CAPITAL of Masral Products, Ltd., has been increased by the addition of £500 beyond the registered capital of £6,000.

THE TREASURY has made an Order under Section 10 (5) of the Finance Act, 1926, continuing the exemption of oxalic acid from key industry duty till March 31, 1935.

MR. BLINDELL, replying to a question by Mr. Morgan Jones, in the House of Commons on December 20, stated that he knew of no agreement between Imperial Chemical Industries, Ltd., and Dupont de Nemours concerning the sale of munitions.

A MINER WAS KILLED in the anhydrite mine of the Billingham works of Imperial Chemical Industries, Ltd., on December 20. The man, Arthur Harpe, was riding on a set of wagons when he slipped and fell on to the track. Several wagons passed over him and he died soon afterwards.

AN INJUNCTION WAS GRANTED to the County Chemical Co., Bradford Street, Birmingham, on December 18, restraining Mr. W. Bigland from infringing its trade mark "Brylcreem." Mr. Bigland, trading as Biglands, of Southampton, had been using a hair preparation of his own manufacture, selling it in the plaintiff company's empty bottles as "Brylcreem" hair cream.

PRODUCERS of METHYLATED SPIRIT announce an all-round reduction of 1d. per gal. in the prices of spirit, to take effect from January 1. The new prices, which will remain in operation until June 30, are: 61 O.P. industrial, 1s. 5d. to 2s. per gal.; pyridinised industrial, 1s. 7d. to 2s. 2d.; mineralised, 2s. 6d. to 3s. Spirit 64 O.P. is 1d. more in all cases and the range of prices is according to quantities.

A LARGE NUMBER of certificates for exports of china clay to certain Continental destinations under trade regulations which came into force a month or two ago are being issued by the St. Austell Chamber of Commerce, Cornwall. China clay firms concerned in these exports were required by the foreign importers to have every shipment certified as 100 per cent. British and as coming from a British port. The Chamber of Commerce was recognised as the appropriate body to give these certificates.

MR. JULIAN PIGGOTT, of the British Steel Association, made a brief statement on December 20 on the agreement which Lord Barnby's mission to the Far East had reached regarding the supply of steel products to Japan and Manchukuo during 1935. The mission, he said had received certain definite assurances while in Japan and Manchukuo, and opportunities would be given for the export to those countries of quantities of British plain steel products.

MANY THOUSANDS OF POUNDS DAMAGES was caused by fire at the Tay Oil Cake Works, Stamergate, Dundee, last week. The works, which belong to the Northern Agricultural Co., Ltd., in association with the Aberdeen Lime Co., Ltd., are situated within a few yards of the main L.N.E.R. line between Dundee and Aberdeen and passenger traffic on the line had to be suspended. About 600 tons of cottonseed, large quantities of cattle feeding stuffs and extensive plant were destroyed. An oil refinery, power-house, stores and other buildings were saved.

A JOINT MEETING of the London Section and the Food Group of the Society of Chemical Industry will be held in the Rooms of the Chemical Society, Burlington House, Piccadilly, London, on Monday, January 7, at 8 p.m., when Professor T. P. Hilditch, B.Sc., F.I.C., will deliver his Jubilee Memorial Lecture, entitled "The Fats: New Lines in an Old Chapter of Organic Chemistry." Professor Hilditch will give a few illustrations of how the general build or composition of any fat can now be stated (at least broadly, sometimes within narrow limits) by reference to its botanical or zoological origin. Some of the factors in glyceride structure and fatty acid composition which determine the suitability of particular fats for different uses will be discussed, together with recent work on fish and other aquatic fats, and some slight similarities between the depot fats of some of the lower animals and those of fishes. The lecturer will also deal with recent developments in fat-hydrogenation such as the glyceride structure of hydrogenated fats and the reduction of fatty acids to higher fatty alcohols; modern work on the detergent, emulsifying and/or wetting value of ordinary soaps and "soapless detergents" which usually contain a long-chain alkyl group linked to a sulphate or sulphinate radical; and the curious similarity in type between a modern higher-alcohol-sulphate detergent and the natural lecithins or phosphatides.

# Metallurgical Section

July 7, 1934

## Microscopic Cracks in Hardened Steel

THE effect and elimination of microscopic cracks in hardened steel has recently been reported upon by Messrs. E. S. Davenport, E. L. Roff and E. C. Bain, of the research laboratory of the United States Steel Corporation, Kearney, N.J. ("Transactions of the American Society for Metals," April, 1934). This paper is an outgrowth of the authors' earlier work on the rates of transformation in steel, for in measuring the transformation velocity of steels at various constant temperatures it became apparent that the products of transformation at temperatures hitherto unused were unique and might possess unusual properties. The influence of grain size upon transformation rate had also been investigated, and the grain size study is here extended to include its effect upon the properties of steel as hardened at a constant elevated temperature.

It is reported that the brittleness of fully hardened and tempered steel is due, in part at least, to the presence of real or potential microscopic quenching cracks. The micro-cracks, or their causes, may not be wholly removed in tempering, once they are developed in the martensite; in one case, heating to 2,000° F. was required to eliminate them. Steel which has been treated to secure a moderately high hardness by means of direct austenite transformation at a constant temperature appears to be free from micro-cracks, and is very much tougher than the conventionally quenched and tempered steel of identical hardness, although it has been heated to the same quenching temperature and has the same austenite grain size. Many carbon steels, after severe quenching, were found to exhibit these intergranular micro-cracks across the original needles or plates of the martensite when etched. The severity of the micro-cracking, and the consequent lowering of ductility, increases with the grain size of the austenite from which the tempered martensite is developed under heat treatment.

Large austenitic grain size has an injurious effect upon the ductility of crack-free hardened steel, but the effect of the micro-cracks is large in comparison with the influence of grain size alone. The presence of a well-distributed soft constituent—such as fine pearlite resulting from a low quenching rate—eliminates the micro-cracks in the martensite, but at least 25 per cent. of the softer constituent seems to be necessary, and in the case of a coarse austenitic grain size cracks may form in spite of the presence of the softer constituent. These micro-cracks are believed to be the result of large dimensional changes, within an individual austenite grain, accompanying its step-wise transformation to martensite. The majority of the cracks run approximately at right angles to the principal axis of the

needles or plates of martensite; that is, in regions which are the first to transform. It is therefore inferred that the stress is one of tension in the axis of the needle, or, more probably, in one direction of the plane of the first plates.

## Welding Characteristics of Steel

WELDING costs can be lowered by carefully selecting the steels which are employed. According to the April issue of "Metals and Alloys," the most economical steel for welding is one which flows smoothly and cools without evolution of gases whilst solidifying, meanwhile retaining its desirable physical properties. Oxidation, non-metallic inclusions, change of structure, solvent power of metal for gases, hot shortness (low strength of metal at high temperature) and cold shortness, are the chief factors which determine the extent to which a particular steel welds better than others. For instance, oxidation may produce a gaseous oxide causing gas holes, it may also produce solid oxides which give rise to slag inclusions, or solid soluble oxides which give brittleness or low strength. Oxidation, by the removal of certain elements, may change the capacity of the steel for holding gases at its solidifying temperatures, and thereby give rise to porosity at the weld. Finely-divided non-metallic inclusions which melt at the high temperature of the arc form visible slag inclusions or slag holes. Hot shortness is commonly caused by a high sulphur content, and to a limited extent by non-metallic inclusions with a high silica content. A high phosphorus content is responsible for cold shortness.

Investigations which have been carried out at the research laboratories of the Lincoln Electric Co., Cleveland, Ohio, have shown that two batches of steel purchased under the same commercial inspection may have entirely different welding characteristics. Steel having a carbon content below 0.15 per cent. has a high gas absorption power; such a steel should be completely shielded during the welding operation. A silicon content of 0.1 per cent. makes welding unsatisfactory unless other alloying or de-oxidising elements are present. Aluminium in excess of 0.01 per cent. may cause a poor weld in the absence of other de-oxidising elements; nevertheless, a small addition of aluminium counteracts the ill-effects of silicon in producing porosity and in lowering the melting point of the products of de-oxidation. The effect of manganese is not so pronounced as that of silicon and aluminium, but a steel containing 1.5 per cent. of manganese will generally exhibit porosity unless vanadium is present. For some peculiar reason a manganese content of 0.25

per cent. definitely produces steel of poor welding quality; such a steel is capable of absorbing ten times as much nitrogen as a steel of the same carbon content with 0.45 per cent. manganese, or five times that of a similar steel with 0.1 per cent. manganese.

### Thermal Conductivity of Metals

NUMERICAL data on the heat conductivities of various metals and alloys have important theoretical and practical applications. The April issue of the "Journal of Research" of the United States Bureau of Standards describes apparatus for the accurate determination of thermal conductivities of metals and alloys up to a temperature of 600° C. The conductivity of the metal is compared, either directly or indirectly, with that of lead. Determinations are made by measuring the axial temperature gradients in two cylindrical bars soldered together end to end, one end of the system being heated and the other cooled, whilst the convex surfaces are protected from heat loss by a surrounding guard-tube. To determine the accuracy of the apparatus, experiments were made with lead, zinc and nickel. The conductivity of each metal was compared with that of each of the other two, and all results obtained were consistent within 2 per cent.

Most researches on the thermal conductivity of metals have been made in an attempt to correlate their thermal and electrical conductivities, and there have been surprisingly few determinations of the thermal conductivity of irons and steel. The thermal conductivity of structural materials over a range of temperature is, nevertheless, of considerable importance. The Bureau of Standards has therefore completed determinations of thermal conductivities of twenty miscellaneous irons and steels over the temperature range of 100° to 500° C. The materials were selected as typical commercial examples used for a variety of purposes and were expected to differ considerably in thermal conductivities. They included cast irons, carbon and low-alloy steels, high chromium and manganese alloy steels, and 18-8 stainless steel. The results of this investigation show that the differences in conductivity of irons and steels are much smaller at high temperatures than at room temperature. High-alloy steels have lower thermal conductivities than low-alloy steels, and an increase in the amount of alloy constituents in iron generally causes an increase in the temperature coefficient of thermal conductivity.

### Removal of Scale

CONVENTIONAL methods for the removal of scale have hitherto been in two directions. On the one hand, abrasion has been adopted, as in tumbling or sand-blasting; on the other hand, it has been possible to dissolve the scale, as in pickling or electrolytic methods. Writing in the March issue of "Metal Progress," Mr. N. Ransohoff describes a descaling plant which has adopted the idea of tumbling the work in an inhibited acid solution. The rough forgings are passed into a tumbling drum which is loaded with hardened stainless steel "stars" and steel grit, the weight of the tumbling material being in excess of the weight of the work to be treated, so that individual forgings "float" in the tumbling material (stars and grit) and seldom touch one another, in consequence of which sharp edges and corners remain undamaged. During the tumbling

operation a cold pickling liquor containing 2 per cent. of acid is kept flowing through the drum by means of a circulating pump, which is connected with a rubber-lined tank situated beneath the drum. The solvent action of the acid pickling liquor first loosens the scale, which is then completely removed by the abrasive action of the stars and grit. The detached scale is subsequently carried away by the stream of pickling liquor to the tank beneath the drum, where it settles out and is periodically removed. Scouring continues so long as the drum continues to rotate. Reversing the direction of rotation causes the contents of the drum to be discharged into a cone-shaped cylindrical screen, where the forgings are isolated from the stars and grit. When the drum is again rotated in its original direction the stars and grit are automatically returned to the tumbling drum ready to receive a fresh batch of work, and the descaled forgings pass on to a rotating washer where tumbling sludge is thoroughly removed by means of jets of hot cleaning liquor which neutralises every trace of acid. From this washer the forgings ultimately pass on to a draining compartment, where they dry by evaporation due to their elevated temperature. This plant which is in operation in the United States is said to remove scale in a very complete manner, and deep recesses are cleaned as thoroughly as an exterior surface.

### Age-Hardening of Nickel Bronze

THE practice of adding moderate amounts of nickel to bronze alloys has been established for many years, such additions being made for the purpose of increasing fluidity, refining the grain, improving the density, or hardening the alloy. An investigation of the age-hardening and mechanical properties of bronze alloys as influenced by nickel content has now appeared as Technical Publication No. 523 of the American Institute of Mining and Metallurgical Engineers. The alloys studied contain 0-12.5 per cent. tin, and 0-15 per cent. nickel, the majority containing both elements in varying proportions. In view of the fact that the major portion of the bronze used is employed in the form of sand-castings, attention was principally directed to study of the materials in the cast form. A review of the mechanical properties obtained in the various nickel bronzes indicated that the presence of nickel secures improvement in properties with decrease in cost, and that higher mechanical properties can be obtained for a constant metal cost by the partial substitution of nickel for tin. Taking the cost of the 88-10-2 copper-tin-zinc alloy as standard, it was found that maximum properties in sand-castings are obtained with tin 5 per cent. and nickel 3 per cent. A number of nickel-bronze sand-cast alloys, in the annealed and aged condition, showed tensile strengths in excess of 40 tons per sq. in. associated with an elongation of 15 per cent. Relationship between mechanical strength, limit of proportionality and elongation after various ageing treatments studied in the alloy with nickel 7.5 per cent. and tin 8 per cent.; the addition of small amounts of silicon, chromium, lead or iron, proved detrimental to the ageing characteristics of this alloy. Limits of manganese content were found to be highly critical. The high elastic properties of the age-hardening nickel-bronzes, their workability and their low metal cost, indicates a widened scope of application.

# The Development of Alloy Steels

By Professor SIR HAROLD CARPENTER, D.Sc., F.R.S.

AN alloy steel may be defined as one which contains one or more elements, other than carbon, purposely added to modify and improve substantially one or more of its useful properties. The qualification "in sufficient proportions" must be included, because elements may be added to straight carbon steels for other reasons, *e.g.*, to give the desired composition and to prevent the occurrence of some defect to which the final product might otherwise be liable. Manganese is present in all ordinary steels made by any of the large scale processes, and it is necessary to have it otherwise the steel is red short. For this purpose the amount required does not exceed 1.5 per cent. The product, however, is not an alloy steel but remains a straight carbon steel. The well-known "manganese steel" contains a relatively large amount, usually from 11 to 14 per cent., of this element which is needed to produce the characteristic and, up to the time of its discovery, novel properties of this type of steel. This is a true alloy steel. Something like thirty years elapsed between the patenting of the first alloy steel and the period during which they have become well established industrial materials. Since then, however, their influence on engineering practice in all its varieties has been quite remarkable, and it is no exaggeration to say that they have revolutionised the technique of its procedure.

## Principal Alloying Metals

It is somewhat remarkable to find that in spite of the large number of metals available, comparatively few have proved themselves suitable for the manufacture of alloy steels. It is not suggested that finality has been reached in this respect and there is little doubt that, with the progress of investigations, a greater number of metals will be used in the course of time. It is, however, significant that by far the greater number of alloy steels which have found industrial application are composed of one or more of only seven alloy metals. These are, in the order of their atomic weights and numbers, vanadium, chromium, manganese, cobalt, nickel, molybdenum and tungsten. All of them are Group A metals in the periodic system, with the exception of cobalt and nickel, which occur in the same group as iron itself. The first three occur just before iron in the system, and thus have the three immediately preceding atomic numbers, *viz.*, 23, 24 and 25, that of iron being 26. Cobalt and nickel, which follow, have the immediately succeeding atomic figures, 27 and 28. Five out of the seven metals, therefore, are the immediate companions of iron. The remaining two, molybdenum and tungsten, are homologues of chromium. All of them are high melting metals. Iron melts at about 1,530° C. and, with the exception of manganese, the remaining metals all melt above 1,450°, while two of them, molybdenum and tungsten, are among the highest melting metals known. Their crystal symmetry, with the possible exception of manganese, is also closely allied to that of iron. The capacity to form alloy steels has thus been found to be confined to a small number of metals adjacent to iron in the periodic system which are high melting and possess a very similar degree of crystal symmetry.

The simplest possible alloy steel is a ternary steel. It contains three elements—iron, carbon and the alloying element in question. Tungsten, chromium, manganese and nickel steels fall into this class. In many cases, however, two alloying elements are present and, in this case, the steel is known as a quaternary steel. Well-known steels of this type are the nickel-chromium, chromium-vanadium, chromium-tungsten, and silico-manganese steels. There still remain, however a number of steels which are even more complex, and contain three or four or even five alloying metals. These are generally classified under the head of complex steels.

Examples are found among high speed cutting tools which may contain chromium, tungsten, vanadium, cobalt and molybdenum, and heat-resisting steels which may contain chromium, nickel, tungsten and silicon. The first useful alloy steel made was Mushet's self-hardening tungsten tool steel, patented in 1868. Prior to Mushet's patent, the most efficient tool was made by quenching a carbon steel in water from above 750° C. Provided the chilling was properly carried out, the hardness of the steel could be increased about six times by this process. It is obvious that it depends for its success upon the capacity to withdraw heat from the steel at a sufficiently rapid rate. There is no difficulty about doing this for thin sections, *e.g.*, knives and tools of moderate size can be properly hardened in this way. It is, however, not possible to withdraw heat sufficiently rapidly at 750° C. from a thick mass

of steel to enable it to be hardened, and therefore the hardening of such a mass cannot be accomplished by quenching. It can, however, be effected in another way, *viz.*, by a change in composition produced by adding an alloying element—nearly always a metal. This was Mushet's discovery. As the name indicates, his steel did not require water quenching to enable it to be hardened. In virtue of the tungsten and manganese which it contained, it became hardened by merely cooling in air. This was the first case of deliberate hardening of steel simply by a change in the composition, and Mushet was the first man to prepare a steel-cutting tool which did not require quenching in a liquid. His air-hardened steel is generally considered to be a tungsten steel. It contained, indeed, 6 per cent. of this metal, but it also contained about 2 per cent. of manganese and 2 per cent. of carbon, and the manganese was required to give the steel the self-hardening property; actually, therefore, it was a quaternary steel.

## Tungsten Magnet Steels

Plain tungsten steels, however, have another use, in virtue of the magnetic qualities which they can be made to possess. They find application in permanent magnets for electric motors, in small dynamos and for hand use, and by far the greater part of the annual production consists of a steel containing about 6 per cent. of tungsten and from 0.6 to 0.75 per cent. of carbon. To obtain the best magnetic qualities such a steel must be heat-treated, and, therefore, all magnet steels are used in this condition. The process involves two operations: (1) the steels are hardened by heating and quenching, and (2) they are then magnetised, and if they are to be used for electric motors they are "seasoned" by a protracted heating at 100° C., so as to make their magnetism as nearly constant as possible. At the present time a great variety of alloy magnet steels is manufactured. The majority of these contain chromium as an essential constituent and either tungsten or cobalt or both. We are completely ignorant of how these alloying metals act in improving the magnetic qualities of carbon steels. The compositions quoted have been arrived at empirically by experiment. The explanation of their action must await proper scientific investigation.

## Ternary Chromium Steels

Plain chromium steels were some of the earliest alloy steels to be made. They were used for a variety of purposes, in all of which, however, the property of hardness was an important factor. These uses date from about the year 1880. The earliest application of such steels, which is still current, is for stamp shoes and dies, for crushing gold and silver ores. These contain from 0.8 to 0.9 per cent. of carbon and from 0.4 to 0.5 per cent. of chromium. Toughness as well as hardness is an essential property of these steels, and some

This historical survey of alloy steels is extracted from the first of a series of booklets which are being published by the Mond Nickel Co., Ltd., through its Bureau of Information on Nickel.



annealing is always required in their heat-treatment. Another important use is in the form of 5-ply plates for the manufacture of files.

Balls and rollers for bearings are generally made of such steels, containing about 1.1 per cent. of carbon and 1.4 per cent. of chromium. All sizes smaller than 0.5 inch in diameter are water-quenched from about 770° C., and then tempered at 190° C. for 30 minutes. Larger balls are quenched from about 800° C. By means of the tempering treatment the balls are toughened, internal stresses are lessened and there is less liability to crack. Mr. H. D. Hibbard ("Manufacture and Uses of Alloy Steels," John Wiley, Inc., New York, 1919) states, "the strength of a good well-treated ball is prodigious." A ball  $\frac{3}{4}$  in. diameter tested by the three-ball method sustained a load of 52,000 lb. On the small area of contact the intensity of the pressure amounted to over 1,000,000 lb. per sq. inch. It will be observed that in all these uses the quality of hardness is the outstanding property required.

### Rustless Steels

It is very remarkable that an entirely new use for chromium steels was discovered more than 30 years later, particularly as the literature of the intervening period contains many references to the preparation and properties of chromium steels. Mention may be made of the extensive researches of Sir Robert Hadfield, in this country, and Guillet and Portevin, in France, but none of these investigators, nor any others up to 1913, discovered that high chromium steels when suitably heat-treated were remarkably resistant to corrosion. This particular discovery was made by Mr. H. Brearley, in 1913. So rapid and important have been the manufacturing developments that followed on his discovery that the corrosion and stain-resisting properties of heat-treated chromium steels now constitute a very important application of this alloy.

Mr. Brearley's discovery of the corrosion-resisting properties of chromium steels was unpremeditated. He was engaged at the time in a research on the resistance to erosion of various steels for ordnance purposes. Among the steels examined were some containing large amounts of chromium in different conditions of heat-treatment. It was noticed that the high chromium steels were often not etched at all or could only be slightly attacked by the usual agents employed in the microscopic examination of steel sections and, moreover, that they did not rust in the atmosphere of the physical laboratory. Mr. Brearley's patent stated, "A typical composition for the untemperable steel embodied in my invention would be as follows:—carbon 0.24 per cent., manganese 0.30 per cent., chromium 13.0 per cent., iron 86.46 per cent. In producing such metal embodied in my invention I preferably use an electric arc-melting furnace. It forges easily into sheets or strips such as are required for knife blades for example, and can be hardened and tempered by ordinary commercial process." It is made clear in the patent specification that if this steel is to be resistant to corrosion it must not contain any microscopically distinguishable free carbides. Its corrosion-resisting properties, therefore, are dependent upon the whole of the carbon being dissolved.

### Stainless Steels

Not all stainless steels are plain chromium steels—some of the most important of them contain nickel as well, but all of them without exception contain chromium in considerable amounts, and the essential corrosion-resisting properties are due to this metal. The discovery of stainless steel is one of the major inventions of this century, and the great variety of its applications is only now beginning to be realised. The range of properties which can be obtained is such that it is no longer correct to regard stainless steel as a particular kind of steel, but rather as a modified form of it, in which most of the mechanical properties of ordinary steels may be obtained in addition to high resistance to corrosion.

Recent progress in the manufacture of corrosion-resisting alloys has depended on the use of nickel in addition to chromium, as by this means it has been possible to extend the range of resistance to corrosive conditions. The alloy manufactured in England under the name of "Staybrite" contains 18 per cent. of chromium and from 8–9 per cent. of nickel. Other varieties of this type are "Anka" and "V2A." Unlike ordinary stainless steel, which is a two-phase alloy, and has

to be heat-treated in order to provide optimum resistance to corrosion, these alloys in the natural state consist of a single phase, the iron being in the austenitic condition. The alloys are ductile and can be worked into almost any form. Staybrite can also be welded, but it cannot be hardened by quenching, although a certain amount of hardening can be produced by cold work.

### High-Speed Steels

The year 1900 witnessed a remarkable discovery by Taylor and White, in the United States, in respect of alloy steels containing tungsten and chromium, which produced a revolution in workshop machining practice. The Mueshet self-hardening tool was a tungsten-manganese steel. If the percentages of carbon and manganese are considerably reduced, and chromium, with or without vanadium, is added, the resulting alloy is also a tool steel, but it possesses even more remarkable properties than Mueshet steel. This was the Taylor-White discovery. They found that when such steels were subjected to a particular heat-treatment the resulting tools possessed properties entirely different from those of a carbon steel or Mueshet tool. The heat-treatment required departed from all previous practice and, indeed, violated all the best accepted canons of such treatment. Whereas great care has to be taken not to overheat a carbon steel tool and, to a rather less extent, a Mueshet tool in hardening, the tungsten-chromium tool steels introduced by Taylor and White required to be subjected to a heat-treatment in hardening so high that the "nose" of the tool was in a state of incipient fusion. These tools then required a second heat-treatment also at a much higher temperature than a carbon steel tool, *viz.*, from 550° to 600° C. After this they were in a condition to work and actually did work at temperatures far above those which would destroy the cutting power of any previously-used tool. Not only did the new tool machine the work at a *red heat*—as a result of which they were said to possess the property of red hardness—but they did not exhibit the best cutting properties unless they were used at these high temperatures. On account of this property a tool made of such high speed steel could be made to cut continuously at speeds from three to five times as great as that practicable with other tools, and when, as a result of the friction of the chip on the tool, the temperature may be 700° C. at the point of contact, and the chip itself heated to about 300° C. As a consequence of this, the chips machined from the work in this way are tempered a deep blue on the surface.

The Taylor-White process as worked out at the Bethlehem Steel Company was not a chance discovery. It was the final result of many experiments and years of hard work by Taylor, an engineer, and White, a metallurgist. The full story is told by Taylor in his presidential address to the American Society of Mechanical Engineers, 1906. Since the original patents were issued in 1901 others have been granted for almost every possible combination of elements which were in any way thought to be useful or valuable constituents of tool steel. Improvements in detail have been effected, but the Taylor-White process, which consisted partly of a new composition and partly, and even more importantly, of a new heat-treatment, remains essentially unaltered to-day.

### Manganese Steel

Returning to the early eighties, mention must be made of one of the most remarkable alloy steels ever made, *viz.*, the so-called manganese steel. The metal manganese occupies a position of peculiar importance in steel metallurgy. It is required in the manufacture of all ordinary steels and, in spite of a large amount of investigation, no satisfactory substitute has yet been found for it. Very large quantities are therefore used annually for this purpose. It is also the essential constituent of manganese steel. This alloy in the commercial meaning of the name is a variety of steel containing from 1.0 to 1.3 per cent. of carbon and from 11 to 14 per cent. of manganese. The original patents covered steels with from 7 to 30 per cent. of this metal, but alloys within the limits of the composition given have the greatest strength and ductility, and are therefore generally used. The discovery of this steel and the working out of its properties over a period of many years will always be associated with the name of Sir Robert Hadfield. It undoubtedly is one of the most important alloy steels ever discovered and certainly

it has become one of the most famous. It was the pioneer of steels of a new type, and the economies resulting from its use in various fields of service have been remarkable.

In the untreated state the properties of manganese steel are similar to those of untreated high carbon steels. The metal is very hard and its ductility is almost negligible. In order to develop the toughness which is so characteristic a property, special treatment is required. It consists in heating the article in question to 1,050° C. and then quenching it as rapidly as possible in cold water. Special care is needed to ensure that this operation is successful. The steel is a poor conductor of heat; a fact which does not make its heat-treatment easy, and tends to limit the thickness of the steel that may be profitably treated, which is generally about 4 inches. It also requires to be heated slowly. The hardness of the toughened steel is unique in that it was the first example obtained of an alloy steel which is both hard and tough. The disadvantage of the steel is that it possesses the combination of low elastic limit and high ductility, as a result of which it is prone to flow under stress. It does not possess high resistance to compression or to continually repeated blows of hard mineral which will gradually batter it out of shape, but probably no alloy steel has found so many uses as this one.

### Nickel Steels

Nickel steels have been found in use for upwards of thirty years. Chronologically what is usually termed nickel steel was the fourth alloy steel to find industrial application. The steels to which this element is added aggregate a large tonnage. Their most marked characteristic is the wide range of composition over which alloys of industrial usefulness have been found to exist. The compass is indeed greater than that of any other class of alloy steel. It ranges from 1 to 74 per cent., and embraces a great variety of structures. Speaking generally, nickel raises the tensile strength of an untreated carbon steel, and in a still greater proportion the elastic limit for a given content of carbon without sensibly diminishing the ductility. It is this property more than any other which determines the special application of nickel steel to practical uses.

Steels low in nickel all possess pearlitic structures. They are mixtures of nickeliferous ferrite and pearlite which have undergone the gamma to alpha change in iron and the iron carbide inversion at a rather lower temperature than any plain carbon steels. The lowering is probably something of the order of 70° C. The important mechanical properties of such steels are due to two reasons, (1) the increased strength of the nickeliferous ferrite as compared with pure ferrite, and (2) the refining of crystal structure produced by the addition of nickel. The majority of these steels contain between 2 and 4 per cent. of the alloy metal. One of their most important applications is in the construction of bridges, particularly those of great span, and this is interesting because bridge steels of this character constitute the principal exception to the almost universal practice of using alloy steels in the heat-treated condition. Locomotive boilers constitute another use of the steel in the normalised condition.

### Martensitic Structure

Steels of the "martensitic structure" type find only a limited application in practice, on account of the fact that they are very hard and possess almost no ductility. They are difficult to work either hot or cold, but can be rolled if proper care is taken. The most noteworthy of these, judged from a scientific standpoint, is the 13 per cent. nickel alloy containing 0.55 per cent. of carbon, discovered by Arnold and Read.

High nickel steels containing 25 per cent. or more of nickel and of low carbon content are austenitic in structure at the ordinary temperature. Actually their structure is very similar to that of manganese steel, the main difference being that the former show no signs of twinning, whereas the latter do. These steels are costly on account of their large nickel content, but in spite of this, they find certain applications on account of the variety of properties of the alloys. One of the most remarkable of them is "Invar," which contains 36 per cent. of nickel. This is used for making clock pendulums, rods for measuring instruments, and such parts, for which its very slight co-efficient of expansion and contraction

give it a particular value. The co-efficient of expansion, though small, is not negligible, and compensating devices must be used in "Invar" clock pendulums and the balance wheels of watches. The co-efficient of expansion corresponds to an increase in length of 0.05 of an inch per mile per °C.

### Nickel-Chromium Steels

Nickel-chromium steels are among the most important of all structural alloy steels. Their field of usefulness is continually being enlarged, not only by their application for new purposes, but also by their encroaching on the uses of other lower tensile alloy steels. They are also invariably used in the heat-treated condition, for they are eminently susceptible to proper heat-treatment. Their mechanical properties can be varied within very wide limits. They can be obtained in a condition possessing great resistance to alternating stresses, and are therefore very suitable for the moving parts of machinery, e.g., crank shafts, particularly where requirements are severe, as in aero engines. In the past these steels have suffered to some extent from a liability to what is called temper brittleness. This term was originally applied by Mr. Dickenson ("Journ. Inst. Automobile Engineers," 1917, 72, 342), to denote the condition induced in these steels by slow cooling from the tempering temperature which is revealed by the low absorption of energy in the single blow impact test of notched bars. Such steels, however, when rapidly cooled from the tempering temperatures are quite tough. These phenomena have been the subject of numerous investigations in recent years which have had for their object the desire to elucidate the cause, for not until this was known could a scientific remedy be applied. The work carried out by Dr. Greaves and Mr. Jones, under Dr. Moore's general direction, at the Research Department, Woolwich Arsenal, has been a most valuable investigation. To-day the problem hardly exists, since alloys can now be prepared which are essentially free from this drawback. A small quantity of molybdenum ensures freedom from temper brittleness.

By the widespread use of nickel-chromium steels great improvements have been effected in the productive of structures for various purposes, especially where saving of weight or increase of strength is important. The most conspicuous examples of these uses are in the automobile and aircraft industries. Heat-treated alloy steels with treble the strength of the simple steels they have replaced are now in regular use. They owe a part of their superiority to the presence of the alloying element, but still more to the care in heat-treatment which is given to them.

### Heat-Resisting Steels

Many of the requirements of modern engineering demand the use of metals and alloys which are capable of service above the atmospheric temperature and, indeed, at quite high temperatures. All alloys of iron are attacked more or less under such conditions, for the atmospheres concerned may consist of air, steam, carbon dioxide, sulphur dioxide and, speaking generally, of furnace gases, which may vary considerably in composition. The majority of the steels which have been found resistant under these conditions contain chromium, and some of them contain nickel as well. Silicon also appears to be a valuable constituent of such steels. It would seem, therefore, that the resistance to corrosion conferred by chromium on steel at the ordinary temperatures, which finds application in the manufacture of stainless steels, is also retained by this metal at high temperatures.

### Alloys of Low Expansion

ALLOYS having a small coefficient of thermal expansion consist of 50–60 per cent. of cobalt, 5–12 per cent. of chromium, and 28–45 per cent. of iron, with or without up to a total of 5 per cent. of one or more of the elements manganese, titanium, vanadium, aluminium, manganese, silicon, and boron, each less than 2 per cent., and are free or substantially free from carbon. The alloys after casting or forging are heated to about 1,100° C. for a long time and cooled either slowly or quickly, or are mechanically worked, with or without subsequent heating to about 100–500° C. followed by very slow cooling. (See Specification No. 405,607, of Kinkoku Zairyo Kenkyusho, Japan.)

# Stainless Surfaces for Chemical Plant

By J. HINDE, M.I.W.E.

WITHIN recent years the number of so-called stainless alloys has considerably increased. The majority of them find application in the chemical industry. No single alloy suitable for every purpose is yet available, but the choice is so wide that a satisfactory selection can usually be made. Where more than one material will serve the purpose, cost will be the deciding factor.

The word "stainless" is relative, and while one does not usually consider mild steel as possessing stainless properties, it is the metal most used in the chemical industry. Mild steel is used in the uncoated condition for storage and transport of strong sulphuric acid, alkalis, oils, etc. Copper-bearing steel, which is mild steel containing a little copper, is better in general corrosion-resistance than the ordinary steel. Where the steel surface is not satisfactory, the work may be galvanised, homogeneously lead-lined, metal sprayed, covered with rubber or glass enamel, depending upon the use to which it is to be put. Lead-lined plant is used for handling sulphuric acid of lower concentrations. Galvanised steel has many applications in the solvent industries and in those industries dealing with cellulose products, constructions which are too large to be galvanised, may be sprayed with zinc. Steel which has been sprayed or metallised with copper or aluminium is sometimes used for acetic acid plant; other sprayed coatings include tin and nickel. The use of a sprayed coating of an expensive metal which is known to be suitable for a particular purpose would appear to be a very economic proposition. Actually, the sprayed coating is only a few thousandths of an inch thick and any slight porosity or defect exposes the underlying steel and results in corrosion which may be dangerous, especially if the coating is cathodic with respect to iron.

## Stainless Steels and Weld Decay

Although stainless steels have not been on the market for many years they already have very extensive applications. The austenitic types are mostly used for chemical plant as they are quite resistant to a whole range of chemicals including nitric acid and most of the fatty acids. Against sulphuric and hydrochloric acids they are not particularly good. For acetic acid, steels containing molybdenum in addition to the usual chromium and nickel are more suitable. The application of these stainless steels was at one time limited, due to the defect called "weld-decay," which necessitated heat-treatment after welding or after any other fabricating process which involved heating to a temperature between 550° and 850° C. Fortunately, slight modification of the composition of the steels has resulted in this defect being overcome, and no heat-treatment after welding is now necessary. In Germany and the United States, but not in England, there has been developed a composite material known as stainless-clad steel, which consists of mild steel to one or both sides of which a layer of stainless steel has been rolled, giving a veneer of 10 per cent. or 20 per cent. of the total thickness. Actually, the difficulties of producing such steel make it more expensive than at first sight would appear necessary. There are also fabricating difficulties.

## Aluminium and Nickel

Aluminium is used in industries dealing with formaldehyde, solvents, fatty acids, fine chemicals, etc., but it is, of course, unsuitable for use with alkalis. The corrosion resistance of the surface may be improved by anodic oxidation which quickly produces a surface film of oxide similar to that acquired naturally. The inherent weakness of the metal is something of a disadvantage, but there are available exceptionally strong alloys of equal corrosion resistance. These alloys usually contain magnesium and manganese, and in some cases are quite as strong as mild steel; unlike Duralumin, heat-treatment is not necessary to develop these properties and they can therefore be satisfactorily welded. The lightness of aluminium and its alloys makes these materials very suitable for the construction of road transport tanks. In order to carry the maximum payload the weight of such a tank unit must be kept to a minimum.

Wherever caustic alkalis are used one is almost certain to find nickel employed in some part of the plant. Malleable nickel can be satisfactorily fabricated into the usual types of equipment and it has established applications in the soap, glue and other industries. Monel metal possesses very good corrosion resisting and engineering properties and it is used for a wide variety of plant; it is used also for descaling equipment in contact with sulphuric and hydrochloric acids, and in the dyeing, paper and varnish industries. Inconel, which is a high nickel alloy containing about 14 per cent. chromium, was primarily developed for the dairy industry, the metal being quite unaffected by and without effect upon milk and milk products; its general corrosion resistance, however, is so good that no doubt many uses might be found for it in the chemical industry.

Copper is still extensively used for acetic acid plant; recently the welding of the metal has been greatly developed, and all types of pressure vessels can be fabricated with the welded joint equal to the parent metal. Aluminium bronzes, special acid resisting bronzes and other copper alloys also have some considerable applications, especially to the smaller items of plant.

## Analysis of Metals and Alloys

### Spectroscopic and Microchemical Methods

THE spectroscopic and microchemical analysis of metals and alloys was the subject of a joint lecture to the London Section of the Institute of Metals, on March 15.

Dr. G. Barr showed how the rays characteristic of a particular "impurity" might be identified in a many-lined spectrum with the aid of comparison spectra or by making use of tables of *raies ultimes*. He emphasised the necessity of adopting standard conditions for the production of the spectra: in an arc discharge the diameter of the electrodes, the length of arc, the current and the circuit voltage were of importance, while in the oscillatory spark discharge the capacity and self-inductance in series with the spark, as well as the characteristics of the high-voltage feed, affected the relative intensity of different lines. In quantitative work the spark conditions were approximately fixed by a method, due to Gerlach, in which the self-inductance was adjusted until the intensities of a certain pair of lines were equal. The intensity of an "impurity" line increased with the concentration of the impurity, but the *raies ultimes* were not usually the most sensitive to changes in concentration. In view of the difficulty of obtaining, even from a given pair of electrodes, series of spectra that were of constant intensity, it was generally necessary to compare the intensities of the lines due to minor constituents by reference to suitable lines of the ground substance. For such comparisons the rotating logarithmic sector described by Tyman and Simeon had been found to be of value, and one of the slides exhibited showed how the lengths of certain lines due to iron, silicon and titanium contained in different samples of aluminium increased, relatively to the lengths of the aluminium lines, as the concentration of impurity increased.

Miss I. H. Hadfield then gave a brief account of the methods of qualitative chemical analysis when only small quantities of material were available. The development of chemical microscopy from the wet reactions used by the geologists was described. In this system the constituents are identified by observing under the microscope the shape and habit of the crystals formed when treated with selected reagents. The reagents used frequently differ from those used in macro-analysis. A number of slides were shown to illustrate this point and to indicate the apparatus which is required. More recently another system of analysis had been developed, namely, spot reactions, in which the crystal form is neglected and the constituents are identified by the colours produced with specific reagents.

# Metallurgical Section

August 4, 1934

## The Testing of Foundry Sands

INVESTIGATIONS carried out by the British Cast Iron Research Association continue to be published. Recommended methods for the testing and control of foundry moulding sands are given in Research Report No. 108, with particular attention to the details of sampling and milling, and to the determination of moisture content, ramming qualities, strength, permeability, expansion and contraction and refractoriness. It is pointed out that it is useless to test a new sand in its virgin state, since the mere addition of water leads to an alteration of properties. The sand should be milled for ten minutes, as repeated experiments have shown that a new sand does not develop its greatest strength until such a period of time has elapsed; if less time is taken in milling, subsequent manipulation of the sand may alter its properties. It is also stated that numerous methods of ascertaining strength have been advocated, but after a trial of most of the published methods, the Association has come to the conclusion that the breaking of the core under a compressive load is the simplest method and if properly carried out it gives the nearest and most consistent approximation to the strength of the sand as used under practical conditions.

The subject of refractoriness also receives careful consideration, for the resistance of moulding sand to the effects of high temperatures is important and determines to a very large extent the sphere of usefulness of that sand in the foundry. For example, moulding sand which softens and fuses at a temperature of 1,400° C. would be suitable for nearly all types of grey iron castings, but would be the cause of blemishes with steel castings, since steel is cast at a much higher temperature than 1,400° C. The determination of the refractoriness of a moulding sand, however, is not a simple matter, for such sands are mixtures of materials each having their own fusion point, with the result that the sand has a "fusion range" rather than a "fusion point." This range extends from the fusion point of the least refractory constituent to the temperature at which the whole mass is soft. Generally the bonding material of the clay is the least refractory constituent, so that a knowledge of the fusion point of this is important. Accordingly, it is suggested that refractoriness may best be studied by the determination of the fusion point of the sand particles, determination of the fusion point of the clay grade of the sand (as separated by elutriation), and behaviour of briquettes of sand when fired at definite temperatures for definite periods of time.

The moisture content of moulding sands can only be accurately determined by the use of a drying oven

and balance. For foundry control purposes, however, it is unfortunate that this drying method is lengthy and really needs conducting in a laboratory. Many attempts have therefore been made to obtain accurate moisture contents by other means. Electrical methods have been brought forward, in which the resistance, inductance, etc., of the moist sand are used as the basis of a comparative moisture calibration, but the presence of carbonaceous and metallic matter, as well as the varying properties of the waters used, lead to conditions which are not conducive to constant results. The experience of the Association in this connection shows that electrical methods give the best results with virgin sands and silica sands, but become more and more unsatisfactory under conditions where large amounts of used or floor sands are tested. The best method for rapid control purposes is that used in testers where the moisture content of the sand is calibrated against the pressure set up by acetylene gas when a calcium carbide mixture is well mixed with the moist sand in a sealed container. Various devices have also been incorporated into sand testing appliances for giving a mechanical estimation of moisture content. The Association carried out experimental work with a view to incorporating such a device on their compression machine with the object of determining moisture content by accurate measurement of the extent to which the compression core is depressed during the test, but it was found impracticable to measure moisture content in this way with the degree of accuracy required in sand testing.

### Surface Finish and Corrosion

OBSERVATIONS upon the effect of surface finish on the initial corrosion of steel under water have been reported to the American Society for Testing Materials. The authors, Mr. L. J. Waldron and Mr. E. G. Groesbeck, conducted their investigation at the Bureau of Standards. They used a low carbon steel finished with various abrasives and studied the initial corrosion rate in water flowing at velocities ranging from 5 to 225 ft. per minute. It is, of course, already well known that the surface of certain metals, such as iron, aluminium, zinc and copper, is coated with a film of oxide which is often very thin and closely adherent to the metal. Coatings of this kind would appear to protect the metal from further oxidation by preventing the diffusion of additional oxygen. On the other hand, if there are discontinuities in the oxide, a film of this nature may easily cause irregularities in the results of corrosion tests. "Breaks" due to the expansion and contraction of the metal, bending

and scratching, "weak spots" produced by inclusions at the surface of the metal, and the breakdown of portions of the film under the influence of chemical agencies in solution, are different factors which may give rise to discontinuities in the film.

### Exploitation of Nickel in Finland

THE Mond Nickel Co. has recently entered into an agreement with the Government of Finland, for the purpose of developing the nickel deposits which exist at Petsamo. Under this agreement the British company is to begin the work of prospecting and developing the extraction process before the end of May, 1935, and is to spend at least £10,000 annually on preliminary research during a period of from three to five years. If the fields prove workable, the company is to obtain a concession for forty years, subject to a royalty of 5 per cent. being paid to the Finnish Government, which estimates the probable amount of this royalty at 5,000,000 Finnish marks annually. The work is to be carried out by a Finnish subsidiary of the Mond Nickel Co., and workmen and employees are to be of Finnish nationality wherever possible.

Investigations on the nickel deposits of Petsamo, which cover an area of about 27 miles in length and two miles in width, have hitherto been controlled by the Government Geological Commission, which will gradually hand over its duties to the British company. It is reported that an annual output of 2,000 tons of nickel, and a corresponding quantity of copper, would have to be achieved before production became commercially profitable. To produce 2,000 tons of nickel annually, from 180,000 to 200,000 tons of ore would have to be worked. The total quantity of nickel-bearing ore in existence at Petsamo has been estimated at only 1,500,000 to 2,000,000 tons, with a probable nickel content of just under 1.4 per cent., but there is a possibility of further deposits being found. If these hopes are realised, from £800,000 to £1,100,000 is thought to be necessary for the installation of plant, although the refining processes will probably be carried out at Clydach, in South Wales.

### Empire Sources of Nickel

AT present Canada produces over 80 per cent. of the world's supply of nickel; most of it comes from the Sudbury district in the Province of Ontario, a very small quantity being recovered as a by-product in the treatment of the silver-cobalt-nickel ores of Cobalt in the same province. The Sudbury ore-bodies are irregular masses of mixed sulphides—iron, copper and nickel—found scattered at the edge of an elliptical area about 36 miles long and 16 miles wide. The known reserves of the district amount to well over 200,000,000 tons, with an average nickel content of nearly 3 per cent. Production in the Sudbury district is entirely in the hands of two concerns, the larger and older concern being the International Nickel Co. of Canada, which smelts its ore in the vicinity of the mines but refines its nickel at Port Colborne, Ontario, and Clydach, South Wales. The other company, Falconbridge Nickel Mines, smelts its ore on the mine, but sends the resulting nickel-copper matte to Christiansand, Norway, for separation and final treatment. Outside Ontario, deposits of nickeliferous pyrrhotite associated with chalcopyrite are known to occur in New Brunswick; in Manitoba, on the Maskwa

River in the south-eastern part of the province; in British Columbia, on Emory Creek in the Yale mining division; and in the Northwest Territories at Rankin Inlet, 300 miles north of Churchill on the west coast of Hudson Bay. In 1929, the year of maximum production, Canada produced 55,137 tons of nickel, but due to world-wide depression in industry only 15,164 tons in 1932. The only other important producer of nickel in the world in New Caledonia.

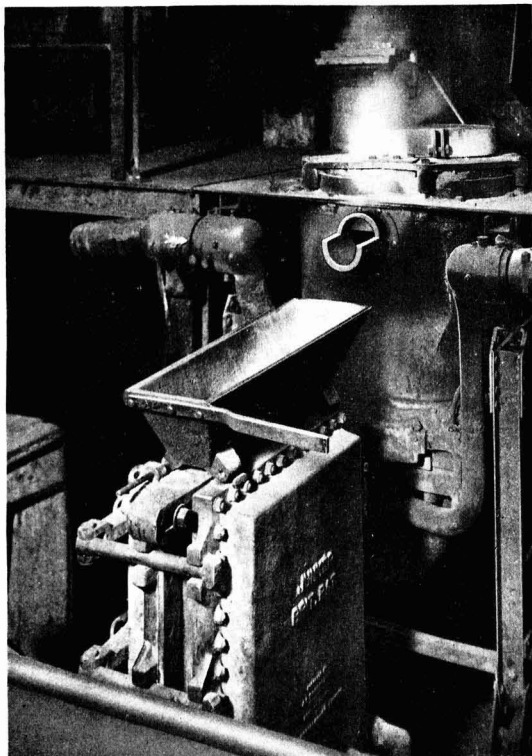
### The Casting of Brass Ingots

FOLLOWING the publication of research monographs which deal respectively with tin solders and metallurgical analysis by the spectograph, the British Non-Ferrous Metals Research Association has now issued a third research monograph, dealing with the casting of brass ingots. The casting of the ingot is a vital stage in the manufacture of wrought metals, for although improvements in quality and economies in working may be effected in later operations, perfection cannot be achieved if the original ingot is faulty. This fact has been fully realised by metal foundries, but the publication of information on the production of good ingots in non-ferrous metallurgy has been more or less neglected, although investigations on the subject have been in progress for a number of years. In this new monograph Dr. R. Genders and Mr. G. L. Bailey have summarised the results of researches which were primarily directed towards obtaining a full understanding of the factors involved in the casting process and the manner in which these factors influence the soundness and surface quality of the cast ingot. The bulk of the work described has been carried out on alloys of one type—ductile brasses used in the rolled condition; such materials extend over a range of composition and are produced in large quantities. Every factor involved in the production of the ingot is submitted to critical examination, the chief points being the composition of the liquid metal, the effect of casting temperature, speed of pouring and feeding, dimensions of the ingot, effect of mould coating and materials for the construction of ingot moulds. The results recorded are by no means limited to the alloys used, but have a much wider scientific and practical value in their relation to casting problems in general.

Those acquainted with the casting of steel will notice some striking comparisons and contrasts, in spite of the great difference in the scale of operations. The practical brass caster, on the other hand, will obtain a very good knowledge of the chief variables which arise in the casting of brass ingots, and of the advantages which are possible by the adoption of special casting methods.

It might, at one time, have been thought that the production of a cast brass slab free from all imperfections was a simple matter, but such disturbing factors as the rapid oxidation of the molten metal surface when exposed to the air and the consequent formation of a troublesome oxide skin, the turbulence of the stream of metal in the mould, the speed of crystallisation, and the contraction in volume on solidification unavoidably introduce defects in the ingot. Remedies for these troubles are not hard to find, but measures which may be taken to remove one difficulty are only too apt to give rise to others which may be even more objectionable.





Ajax Wyatt Electric Furnace, capacity 1000 lbs. brass, with Junker Copper Faced Water-Cooled Mould of similar capacity. (Reproduced by courtesy of I.C.I. Metals Ltd.)

# The Casting of Brass Ingots

A Notable Investigation  
by the  
British Non-Ferrous Metals  
Research Association

**T**HE practice of brass-making until recently has been carried out almost entirely in numerous small foundries, each separately dependent on traditionally trained foundry workers and in the majority of cases without the resources necessary for technical research. Under these conditions no radical changes of method occurred during the industrial development of the nineteenth century, and manufacture remained largely controlled by a technique built up entirely by empirical means.

The practical skill of the average brass caster and the care which he exercised in his work have been reflected in the general high standard of quality of brass sheet and strip produced. In the absence of scientific control, however, the causes of the common types of defect leading to difficulties in manufacture and the directions in which remedial measures might be sought could not be clearly ascertained. Establishment of the necessary knowledge of the process was essentially a matter requiring combined effort. A channel for such co-operation was provided by the inception of the British Non-Ferrous Metals Research Association in 1920, and the casting of strip ingots of brass was one of the first subjects selected for investigation.

## Improvement of Quality

The object of the research was essentially the improvement of the quality of brass strip, to be reached mainly by the investigation of defects, the detailed study of the behaviour of brass in casting, and the development of improved methods. Although the cost of laboratory research is increased by working on a relatively large scale, it was considered that for a subject such as the casting of brass the disadvantages would be more than counterbalanced by the closer applicability of the results to manufacture. Since brass of 70/30 composition was not only the most important but also by general opinion one of the most difficult common alloys to manufacture in high quality strip, most of the experimental work was concerned with this composition. The

many factors affecting surface quality and soundness provided a sufficiently large field for the investigation which was carried out by Dr. R. Genders and Mr. G. L. Bailey and their colleagues at the Research Department, Woolwich.

The results of this research, and of a number of related investigations arising from it, were communicated to the industrial members of the Association from time to time and have now been published in the form of a special research monograph entitled "The Casting of Brass Ingots." This monograph presents the results of the research in summarised form, amplified by discussion of their practical bearing.

## Variable Factors of the Casting Progress

The chief variables which arise in the casting of brass ingots are casting temperature, speed of pouring, feeding of contraction, shape of ingot and position of cooling surfaces, thickness of ingot, mould temperature and thickness, mould coating, position of mould during casting, and gas content of the metal.

The range of temperature in which it is practicable to cast 70/30 brass is not more than 200° C. and the range in commercial practice is only about 50° to 100° C. The variation in rate of pouring which can be conveniently used in a 12 x 6 x 1 inch mould ranges from a rate of rise of metal in the mould of 1.5 inches per second (about the average speed used in works) to a very slow speed of about 0.3 inch per second. With a casting temperature of 1,060° C. or above, the unsoundness of the ingot decreases as the rate of pouring decreases. This is due to the plane of complete solidification across the ingot rising closer behind the fluid level with slower rates of pouring, the contraction at the centre being continuously fed by the fluid metal immediately above. A slow rate of pouring of about 0.3 to 0.4 inch rise of metal in the mould per second is necessary to secure internal soundness. With a very low casting temperature (1,000° C.) the ingot is unsound at both rapid and slow rates of pouring, and the unsoundness tends to increase with decrease of pour-

ing rate, owing to the formation of a type of defect differing from contraction cavities and produced by the entrapping of air bubbles by the rapidly solidifying metal.

Good surface quality is only secured by rapid pouring at a high temperature. As either casting temperature or rate of pouring is diminished the surface becomes more defective and under extreme conditions, *i.e.*, low casting temperature and slow pouring, defects of abnormal extent are produced if oxidation occurs. These are deep folds and semi-attached particles of splashed metal, and are due to the restriction of free flow by the crust of solid crystals and oxide formed continuously on the exposed liquid surfaces.

For cast-iron moulds such as are used in commercial brass casting, the mould temperature of about 100° C. normally used is satisfactory for general purposes. With mould temperatures up to about 600° C. there is little difference in the rate of solidification of the ingot. Beyond this temperature, however, further increase produces a considerable lengthening of the time of solidification, which at 750° C. is twice, and at about 850° C. five times that at 180° C. The solidification of a thin slab ingot of 70/30 brass cannot be delayed appreciably unless mould temperatures of the impracticable order of 700° to 800° C. are used.

#### Effect of Mould Coating

The general surface of a commercial brass ingot is always characterised by its slightly glazed "texture," due entirely to the effect of the mould coating or "dressing" of oil or resin-base material which is universally employed in brass casting. Such a surface could not be reproduced in ingots cast, for example, exposed to air in a mould lightly blackened or faced with graphite.

The importance of the mould coating or dressing as the main factor governing the surface quality of the ingot is fully appreciated by the practical caster. Methods differ in detail; the mixtures used, which are in practically all cases inflammable and based on either resin or oil, vary in composition and consistency, and either heavy or thin coatings may be favoured. As a rule, however, the bottom of the mould is coated more thickly than elsewhere, while it is customary to use the thinnest possible mould dressing with the higher copper brasses which are necessarily cast at relatively high temperatures. At the commencement of pouring, the dressing is ignited by the molten metal and a large smoky flame is produced at the mouth of the mould. The absence of oxide is due to isolation of the brass from the air by the cloud of gas evolved from the volatile coating. A second function of the mould dressing is the protection of the mould surface from undue heating by the molten metal, so avoiding the projection of gases from the cast iron into the surface of the ingot. This protection is afforded by many types of material and is not peculiar to the inflammable mould dressing.

#### Effect of Dissolved Gases

Most metals are capable of dissolving considerable quantities of gases, the solubility being, as a general rule, much greater in the liquid than in the solid state. Under ideal conditions of solidification the gases evolved would escape through the liquid metal, but under normal conditions particles become entrapped. In chill castings the unsoundness due to dissolved gases is not so common as in sand castings, and while this may be partially due to the more ready escape of the gas under conditions favouring columnar crystallisation, it is more probable that, owing to the rapid solidification, the gas is retained in solid solution in the metal. The gas most commonly responsible for this unsoundness is hydrogen, which is soluble to a greater or less extent in all molten metals.

In the case of aluminium and its alloys, which are very liable to show "pinholing" due to dissolved gases, it has been shown that hydrogen enters the metal largely as a product of reaction of the metal with steam. In the case of copper it is known that porosity is caused by a reaction between hydrogen and oxygen (both of which are soluble in the liquid metal), to form steam. Similarly, sulphur and oxygen together in molten copper cause unsoundness due to evolution on solidification of sulphur dioxide—an effect very noticeable in "blister copper." In the case of brasses containing 60 per cent. to 70 per cent. copper, however, it is

considered that gas cavities are attributable to causes other than the liberation of dissolved gases, for the atmosphere above the molten brass in the crucible consists of evolved zinc vapour, and the partial pressure of any soluble gases is accordingly lowered, so that they are automatically expelled from the metal. This conclusion is supported by the fact that the use of low casting temperatures in ordinary practice is followed by an increase in the number of spherical cavities.

With brasses of high copper content, however, the partial pressure of zinc at the casting temperature is considerably lower than with 70/30 brasses. For instance, 90/10 brass which is commonly cast at about 1,200° C., has at this temperature a vapour pressure of less than 100 mm. Hg., and the occasional occurrence of central gas cavities in this metal, giving rise to central blisters in rolled sheet, suggests that in such alloys the evolution of zinc vapour may not be always sufficiently rapid to preclude entirely the solution of other gases.

#### Material for Construction of Mould

Little scientific work has been done on the fundamental properties of ingot moulds. Cast-iron moulds have been accepted over many years without the fundamental properties requisite in a mould material having been adequately examined. For non-ferrous work the subject has received more attention as a result of the introduction of copper-faced water-cooled moulds for the casting of brass strip ingots. These moulds consist essentially of thin copper plates (0.25 to 0.75 inch in thickness) which form the working faces of the mould and are bolted on to a steel or cast-iron shell which permits cooling of the backs of the plates by circulating water. The mould stands vertically and the two faces are hinged at one side to open in book fashion, thus permitting easy handling; the circulating water is admitted through the lower hinge and allowed to escape through the upper hinge, and internal baffles are arranged to ensure an adequate circulation of the water over the surface of the copper plates.

#### Improved Casting Methods

As a result of the examination of the individual influences of the numerous factors involved in the casting process, the formation of the ingot during casting and solidification can be visualised and the temperature gradients and oxidation effects at any stage can be roughly predicted from the known conditions present in any particular case. Since the production of an ideal temperature gradient; that is, from bottom to top of the ingot, is impracticable with slab ingots of ordinary form, the only method of securing freedom from contraction cavities would consist in the use of an extremely low casting temperature. This, however, is not practicable in view of the inevitable introduction of surface defects and cavities due to entrapped gases.

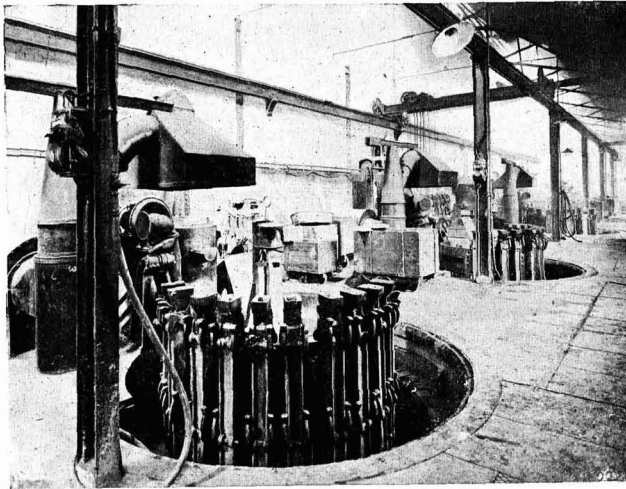
The conditions most favourable to the elimination of surface cavities and surface defects, such as folds in the ingot, in ordinary commercial casting methods are:—(a) A high casting temperature and a rapid rate of pouring, the main effects of which are to minimise the risk of entrapped gases and to avoid surface folds. (b) The use of the minimum amount of volatile mould coating necessary to give freedom from surface oxidation effects. (c) Casting with the mould placed vertically; the method of casting in an inclined mould is conducive to the entrapping of the gases from the mould atmosphere by injection, as well as of gases evolved directly from the mould coating. Any of these modifications singly or combined will have an influence in the direction of increasing the freedom of the ingot from defects which affect the quality of rolled strip.

#### Regulation of the Pouring Speed

Other refinements which have an important effect in improving the quality of the ingot by avoiding defects due to the mould and the inclusion of slag with the stream consist in the use of copper moulds, either solid or water-cooled, in place of cast iron in order to obviate possible blowing defects, and the employment of a bottom-pouring ladle which ensures a clean stream of metal with simple means of regulating the pouring speed.

Whilst these relatively simple measures outlined above are capable of improving ingot quality and reducing wastage to an important degree, it cannot be hoped that the ingot ideally

A Battery of Ajax-Wyatt Electric Furnaces engaged in the casting of Cartridge Brass. (Reproduced by courtesy of The Electric Furnace Co., Ltd.)



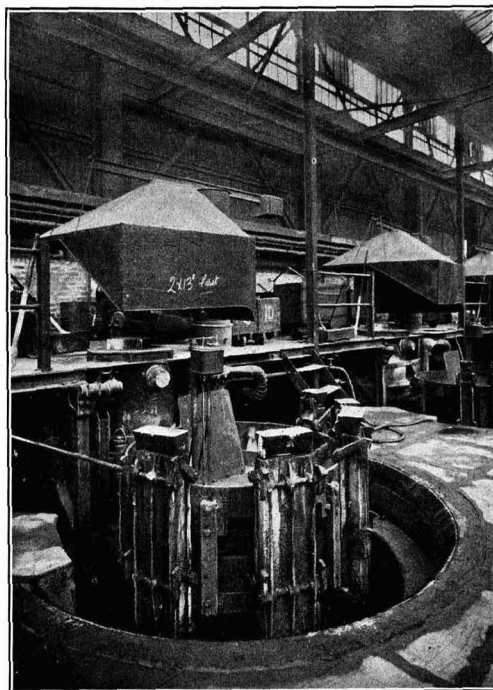
free from subsurface cavities will become a regular product unless steps are taken to avoid all means of gases becoming entrapped in the ingot. The most prolific source of entrapped gas cavities in commercial brass ingots is the commonly used volatile mould coating, the effects of which are intensified when the mould is filled in an inclined position. Some coating is necessary to protect the mould faces and to give a sufficient degree of heat insulation to avoid too severe an initial chill effect on the turbulent metal surface. Carbon (soot) deposited by an acetylene flame played on the mould surface is a satisfactory material and oxidation can be avoided by surrounding the stream of metal and the mouth of the mould with a luminous flame of coal gas. Tests of this method made by casting 70/30 brass in moulds of cast iron, malleable cast iron, steel and copper, and rolling the ingots to strip, show the material to be of high quality provided the ingot surface is not impaired by blowing. To obviate this danger, which is increased in the absence of a volatile coating, copper moulds are recommended. A number of ingots so produced and examined by X-rays showed no sub-surface unsoundness.

The addition of a small percentage of phosphorus (about 0.05 per cent.) has the effect of avoiding the formation of any coherent film of oxide on the surface of molten brass. Although the rate of vaporisation of zinc from phosphorus brass is actually greater than from pure brass, the oxide formed is incapable of being held in suspension or attached to the surface of the molten metal. Brass containing phosphorus can thus be poured with full exposure to air without the use of a volatile mould coating with no risk of the production of defects due to oxidation. A further characteristic of brass containing phosphorus is that, owing to the apparent enhanced fluidity of the metal, the ingot invariably shows a very clear impression of the mould.

#### The Durville Casting Process

The Durville process (British Patent 23,719 of 1913) was devised in order to avoid the surface defects to which alloys containing aluminium (such as aluminium-bronze) are liable when poured in the usual way. The alumina film has a tendency to form folds and splashes during pouring, and the essential feature of the Durville process is the avoidance of turbulence during pouring, which is the main cause of these effects. The apparatus used consists of an ingot mould connected by means of a trough to a ladle in such a manner that the lower edge of the ladle, the base of the trough and the lower edge of the mould are in a straight line. The ladle is filled with metal which is then transferred smoothly to the mould by inverting the apparatus.

The Durville process was originally used for the casting of aluminium-bronze containing 8 to 9 per cent. aluminium



Ajax-Wyatt Electric Furnace equipped with turn-table and moulds, as used for casting non-ferrous metals. (Reproduced by courtesy of The Electric Furnace Co., Ltd.)

for French coinage. In casting aluminium-bronze the metal is melted without the use of any flux and the temperature raised to 1,200° to 1,300° C. It is then poured into the ladle, which is attached to the casting apparatus. The metal is skimmed carefully and allowed to cool to about 1,100° to 1,150° C., the apparatus being then rotated. This operation is smooth and regular and occupies about 30 seconds. After detachment of the ladle, the ingots can be lifted out of the mould, the walls of which have a very slight taper outwards towards the top. Ingots of aluminium-bronze and of brasses containing aluminium, cast in this way are of high quality. The surfaces are smooth, showing only small ridges corresponding to the successive levels of the metal in the mould during rotation. On account of the low casting temperature used, contraction cavities are reduced to a minimum, and in general it can be said that such ingots are of high quality as regards surface and internal soundness. If pure 70/30 brass is cast in this way with the metal surface freely exposed to air, the skin of zinc oxide which forms is found to break and fold during the casting operations and give rise to surface defects. The presence of aluminium in the brass overcomes this difficulty by producing a strong surface film of alumina; as little as 0.1 per cent. aluminium in 70/30 brass is sufficient to enable the material to be cast with high surface quality, without the use of any mould coating.

### The Erial Casting Process

In the new type of water-cooled mould devised by Erichsen (British Patent No. 358,697 of 1931) the cooling plates or faces consist of a nickel-iron alloy of exceptionally low heat conductivity which possesses in addition a low coefficient of thermal expansion. It is claimed that as a consequence of this the rate of solidification of the ingot is slower than in an ordinary mould. During solidification the inner faces of the plates are at a higher temperature than the outer faces. The resulting unequal expansion causes the plates to bend so that the inner surface of the mould tends to become convex,

thereby exerting such a strong pressure on the ingot during solidification that liquid metal from the central portion is forced upwards. In thin ingots (up to 2 inches thick) the liquid metal is exuded in a sufficient quantity to compensate completely for shrinkage, and piping is avoided without any need for feeding; in some cases the ingot is actually increased in length by this effect. The pressure effect in the "Erial" mould is further claimed to give uniformly fine ingot structure, a favourable factor for subsequent working.

### Bottom Casting

The process of "bottom-casting" is well-known in the steel manufacturing industry, where it has become increasingly used. It has the advantages of giving ingots of good surface and subsurface soundness and of dealing conveniently with large volumes of steel. The moulds, a number of which are filled at the same time, are open at both ends and stand on a bottom plate in which are fire-clay tubes or runners leading from each mould to a central "trumpet" where the molten metal enters.

Application of the bottom-casting process to brass casting is of interest in view of the desirability of avoiding the turbulence which is characteristic of the ordinary top-casting method. When ingots of 70/30 brass are bottom-cast in air the formation of a surface oxide film on the metal rising in the mould results in defective regions on the ingot surface. By casting with the mould leaning at a slight angle the oxide film can be made to adhere to one face only, and an ingot is produced having one face almost perfectly smooth and free from defects, while the opposite face is uniformly covered with oxide skin folded along curves produced by the flow of the metal. With a volatile mould coating the ingots produced are similar in character to industrial top-poured ingots of the highest quality. Where the smoothest possible ingot surface is required, the bottom-casting process appears to have distinct advantages, especially in connection with the use of phosphorus as an addition to brass.

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## Tellurium-Lead

### Some Unique and Outstanding Properties

TELLURIUM-LEAD, which is a British discovery of vital importance to all users of sheet lead and lead pipes, is the result of prolonged investigations in the research laboratories of Goodlass Wall and Lead Industries, Ltd., supplemented by numerous large-scale trials. This new alloy has an extremely fine and uniform grain structure—many times finer than hitherto produced in lead or its alloys; this results in a smooth surface without coarse grain structure, mechanical resistance is thereby increased, and local corrosion lessened. Many cases of corrosion or mechanical failure of ordinary lead can be traced to coarse grain structure. The new alloy also has a unique and outstanding property in its capacity to toughen when strained, whereas ordinary lead has no latent strength and therefore does not toughen when strained. Tellurium-lead rolled sheets are toughened in manufacture, whilst in service the strains such as frost, vibration, bending, etc., normally responsible for the failure of ordinary lead pipes, actually strengthen tellurium-lead pipes.

Tellurium-lead has the unique capacity of developing latent strength when strained. This latent strength—not possible with lead or alloys of lead—gives tellurium-lead sheets twice the tensile strength of ordinary lead when fully toughened. The degree of such "work toughening" is under control and sheets can be produced in a soft or toughened condition.

Tellurium-lead pipes when strained have at least 60 per cent. greater strength or toughness than ordinary lead. They are strengthened by the causes normally responsible for the bulk of the failures of ordinary lead pipes. They have greater resistance to bursting by frost than any other lead alloy pipe and more than twice the resistance of an ordinary lead pipe. A tellurium-lead pipe subjected to one more

freezing than would burst an ordinary lead pipe withstood a greater hydraulic pressure than did an unfrozen ordinary lead pipe, showing that strain causes tellurium-lead to toughen and build up resistance to failure.

The vibration or fatigue resistance of tellurium-lead sheets and pipes as determined in a Haigh fatigue testing machine is three times that of ordinary lead. This is of vital importance when it is recognised that the vibration occurring in cables, on ships, viaducts, etc., and domestic supply pipes arising from water hammer and modern traffic is responsible for many of the failures of ordinary lead.

The metal has markedly increased resistance to the solvent action of drinking water as determined on waters obtained from many different parts of the country. It has greater resistance to corrosion than ordinary lead. The extremely fine and uniform grain structure gives a smooth surface with much superior appearance; mechanical resistance is greatly increased and local corrosion is reduced. Tellurium-lead pipes withstand more working and do not roughen when bent—their internal smoothness reduces friction losses and retards furring. Pipes are easily worked, and sheet can be supplied in a soft or toughened condition.

Tellurium-lead, it must be added, is virtually pure lead—its unique properties are obtained by the addition of a very small quantity of tellurium to existing grades of lead—the known valuable properties of these grades are maintained and new properties developed. It can be soldered or burnt in exactly the same way and as easily as ordinary lead.

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THE introduction of up-to-date machinery for the recovery of gold in Roumania is expected to entirely replace the existing primitive methods of gold mining.

# Metallurgical Section

September 1, 1934

## Production of Soft Cast Irons

FROM time to time the British Cast Iron Research Association has received inquiries as to means for regularly producing castings which can be easily machined at high speeds, softness and good machinability being essential qualities in many castings of a fairly light nature. Troubles have occasionally been traced to chilling, whereby the iron has become mottled or white in thin sections and would therefore demand the use of tools of the tungsten carbide type. In other cases the castings have proved to be uniformly grey, and in that condition they would present few machining difficulties were it not that present-day methods demand high machining speeds.

In his report on "Soft Cast Iron," which is published in the July issue of the Association's quarterly bulletin, Mr. L. W. Bolton discusses the production of castings which are as soft and machineable as possible in the "as-cast" condition, and also gives some useful information on the heat treatment which is necessary to impart maximum machinability and softness to such castings. He points out that four essential points have to be borne in mind with regard to composition. In the first place, where high phosphorus is not essential for fluidity, this element should not exceed 0.5 per cent., at which figure its hardening effect on the metal becomes pronounced. Secondly, the total carbon content should be high, and although the amount of carbon that the iron will hold is influenced by the amount of silicon and phosphorus which is present, a figure of at least 3.5 per cent. should be attained. The large amount of graphite in a high carbon iron breaks up the matrix and lessens the resistance of the metal to the tool, but coarse "kishy" flakes of graphite should be avoided; beneficial effects are only possible when the graphite is in a finely-divided form. Thirdly, the most suitable silicon content will depend on the section thickness of the casting, but should not exceed 3 per cent. Silicon, of course, is essential to ensure that the iron is uniformly grey, and it softens the metal up to a point by decomposing the pearlite and producing ferrite, which is a very soft constituent. In addition, it reduces the total amount of carbon that the iron will hold, and has a hardening and embrittling effect on the ferrite if present in excess of about 3 per cent. Lastly, the sulphur and manganese content should be in balance, but free manganese is preferable to free sulphur.

Extreme care should be taken to see that no chromium enters cast iron where softness is desirable. This warning is necessary in view of the increasing use of this element in some branches of the founding industry. The British Cast Iron Research Association

has found appreciable amounts present in some castings which have failed to give good machinability. In every case it has entered the mixture from purchased scrap, and if such scrap is used care should be taken to see that it contains no automobile engine castings, or any other castings which have been used for heat- or wear-resistance. Manganese in excess of that required to neutralise sulphur also has a similar effect in stabilising the carbide in cast iron, but in this respect is much less drastic than chromium.

In normal grey cast irons the combined carbon begins to break down at a temperature not greatly exceeding 400° C., but at such a low temperature the structural change would take a great many hours to be completed. The usual practice is to heat the metal to a temperature 10° to 20° (C.) above the critical temperature, maintaining this heat for, say, thirty minutes, and then cooling slowly to a dull red heat 500° C. The period at the maximum temperature is necessary to ensure that the whole of the carbide present goes into solution, but the major portion of the carbide is actually broken down during the cooling, which is an equally important part of the process. The slower the cooling the more opportunity is given for the whole of the carbide to decompose, and if possible the cooling from the annealing temperature to 500° C. should occupy at least one hour. The critical temperature of a pure iron carbon alloy on heating is 730° C., but in cast iron this temperature is higher because of the presence of silicon, critical temperature being raised roughly 30° (C.) for each 1 per cent. of silicon present. Manganese in solution in the iron also affects the critical temperature, but in the opposite direction to silicon, the reduction being approximately 25° (C.) for each 1 per cent. of manganese present.

### Gold Flotation Experiments

A RECENT report of investigations (No. 3226) of the United States Bureau of Mines gives the results of experimental work on the flotation of gold employing synthetic samples composed of definitely known constituents. By this method flotation data was obtained for various sizes of metallic gold particles, which should have practical application to the recovery of metallic gold by flotation from natural ores. For each test the particular sample used was prepared by mixing a definite quantity of natural metallic gold with clean sea-sand, previously ground to 60 mesh, and in one instance by adding a definite amount of barren pyrite to the gold-sand mixture. The gold was cleaned by careful pannings to remove non-metallic gangue, and then by prolonged treatment with dilute nitric acid to



dissolve the sulphides. In this way a product was obtained consisting of pure metal 850 to 900 fine in respect of gold, the remaining 10 to 15 per cent. being metallic silver. It was mixed with the ground sea-sand or ground sea-sand plus pyrites to form the flotation feed. After flotation the metallic gold in the concentrate and that remaining in the tailing was carefully removed from the gangue and cleaned as already described. It was then sized separately and weighed, thereby permitting the exact flotation recovery of each size of gold to be computed.

From the results obtained it has been concluded that clean metallic gold, under 60 mesh in size, can be floated successfully from a siliceous gangue or a siliceous gangue containing pyrites with the usual flotation reagents used for sulphide ores, but it does not float as readily or as rapidly as most of the sulphides. Gold which is coarser than 40 mesh is not recovered in the usual flotation concentrate, but almost complete recovery can be effected by flotation if the ore is ground to pass 60 mesh and no interfering slimes are present in the flotation feed. The addition of copper sulphate does not increase the percentage recovery, but certainly aids by increasing the rate of flotation of gold particles which are under 60 mesh in size.

### Anodic Oxidation of Aluminium

THE formation of an anodic film on aluminium is possible in a fairly large range of electrolytes, but the necessity for obtaining a film of adequate thickness to resist wear as well as corrosion limits the number of media which may be employed. Practical difficulties associated with the employment of an electrolyte which can only build the film at relatively high voltages, or at a slow rate, still further restrict the field of utilisable solutions so far as commercial purposes are concerned. According to Dr. S. Wernick, who read a paper on this subject before the Electro-Depositors Technical Society in London, last April, the best known electrolytes are chromates, sulphates, oxalates and phosphates. Different electrolytes have been developed in different countries, chromates being used in England at 40° C. and 40-50 volts D.C. to give an opaque light grey film, whilst sulphates find use in the United States and also in England at 15-30° C. and 10-20 volts D.C. for the production of a translucent silver-like film. Phosphates give a somewhat opaque bluish-grey film; oxalates, as developed in Japan and Germany, at 15° C. and 30-200 volts A.C., give an opaque yellowish-brown film. The different appearance of the various films should be noted. General physical properties of the film such as thickness, compactness, hardness and electrical insulating characteristics, are also greatly dependent on the method used.

The oxalic acid processes were originally developed with the primary object of providing high electrical-insulating coatings on aluminium. More recently the use of this electrolyte to provide protective and decorative coats has been explored, and it is believed that its application in this direction has now reached the commercial stage. So far as England is concerned, nearly all the anodic work which is produced commercially has been turned out in chromic acid electrolytes. Sulphuric acid processes, which have been introduced during the last two or three years, have varied greatly in the

degree of success which has been attained, but they possess the important advantage that comparatively low and constant voltages may be employed.

### Temper Brittleness in Nickel Steels

THE phenomenon of temper brittleness in nickel steels has been specially investigated on a series of steels selected from the current run of work at a Japanese steel-making plant. The materials were chosen with a view to the determination of the influence of composition on susceptibility to brittleness, and comprised electric-melted and crucible-melted samples of carbon, nickel, chromium, manganese, nickel-chromium, nickel-manganese, chromium-manganese and nickel-chromium-manganese steel. They were arranged in series designed to elucidate the influence of the individual elements. The April issue of the "Japan Nickel Review" gave a detailed report of this work, the theories of earlier workers in the field being discussed in relation to the findings of the present author.

Results are given of experiments on the inhibition of temper brittleness by the addition of certain alloy elements. The data, which is presented in the form of nearly 300 curves, leads the author to conclude that the temper brittleness of steels is related primarily to their composition. The effect was observed in chromium, manganese, phosphorus, silicon and nickel steels, but not in carbon, tungsten, molybdenum, vanadium and sulphur steels. Two types of temper brittleness were found. The first occurs in the tempering range 450°-525° C. and is unaffected by the rate of cooling after tempering; the second, occurring about 525° C., is caused by slow cooling after tempering. Both types are associated with the carbide constituent, the "first temper brittleness" being due to the precipitation of carbide from  $\alpha$ -iron and the "second temper brittleness" being the result of increase of solubility in  $\alpha$ -iron and due to variation in the amount of precipitated carbide present in rapidly and in slowly cooled samples. The carbide is, in most cases,  $Fe_3C$ , or its solid solution with the alloying element.

### Magnesium from Dolomite

THE Metallurgical Research Bureau at Washington has recently reported upon raw materials for the manufacture of magnesium metal and the processes which are available for the production of magnesium specifically from magnesite and dolomite. These processes may be divided into three groups. The first group comprises processes which involve the production of magnesium chloride from the carbonate and its subsequent electrolysis. In the second group are those processes involving the production of magnesium oxide, which is subsequently reduced to the metal. The third group is dependent on vaporisation and condensation of the magnesium metal after reduction in the electric furnace. Full details are given of the various processes concerned and an attempt is made to compare their advantages and disadvantages. Flow sheets are included, together with a selected bibliography and a list of patents. The final section of the report deals with leaching tests which have been carried out on magnesite and dolomite, but the results which are given merely represent preliminary work.

## Researches on Tin Promise Profitable Results

### International Tin Research and Development Council Publish First General Report

THE first general report of the International Tin Research and Development Council, now published, describes the organisation and progress of this body which was established by the governments and tin producers of the principal countries exporting the metal. One of the chief objects has been to plan a research and development programme to assist industries which use tin in their manufacturing processes and at the same time to conduct fundamental researches in order to develop new outlets for the metal. The Council have organised and are directing researches on basic problems relating to major applications of tin which they have delegated to university, government and industrial laboratories. Centralisation of research work has been avoided as far as possible in order to meet more adequately the special requirements of tin-consuming industries in the various countries. This work has so far been distributed in England, France, Germany, Holland and the United States.

Among the applications of tin covered by the researches are tinfoil, bearing metals, preparations of alloys from powdered metals, solders and other lead-tin alloys, bronzes, addition of other elements to tin, hot-tinning of steel and copper, electrodeposition of tin and tin alloys, anodic oxidation and colouring of tin, spraying of tin, pewter, tin foil, and block tin pipe. Progress has been made in all these researches, whilst in some important directions results have already been obtained which promise to have a definite influence in improving products containing tin. Several new uses for tin are mentioned in the report. A new dry electrical accumulator, in which tin forms one of the electrodes is described, and the application of tin compounds is indicated for a number of new industrial uses.

#### Corrosion of Tinfoil

Differences of opinion regarding the mechanism of corrosion of tinfoil exist among the research workers in the different groups. In connection with the Council's corrosion research it was therefore arranged that a careful analysis should be made of all the conclusions based on fact and the hypotheses advanced to explain the facts. The survey emphasises the importance of certain fundamental issues concerning the variation in the electric potential difference occurring between tin and steel in contact with the fluids present in foodstuffs. In contact with such solutions at discontinuities in the tin coating there are set up electric currents passing between the tin and the areas of exposed steel. Corrosion of one or other or both metals may occur, depending on a number of conditions, such as the acidity of the foodstuff, the presence of air, etc. As a result, a new hypothesis has been put forward, which gives a clearer picture of the whole mechanism, and a paper has been published embodying the results of this work.

From the practical point of view a clear understanding of the mechanism may lead to the development of methods of diminishing the attack on the tinfoil by adding a suitable corrosion inhibitor. Various attempts in this direction have already been made, but the results so far have been uncertain and inadequate.

The evaluation of the merits of the different kinds of steel used as regards corrosion of tinfoil is also of great importance. The work so far carried out in this direction tends to prove that the purer form of steel which can be utilised in the new cold-rolling process results in a decreased tendency to the formation of hydrogen swells. This improvement is not confined to the fewer breaks produced in the tin coating on deformation, but appears to involve a lower rate of attack of the exposed steel. The possibility of still further diminishing the corrosive attack by a slight modification of the composition and heat treatment of the steel suggests an important field for further investigation. The application of the new cold-rolling methods permits the use of steel of higher purity, particularly as regards the content of phosphorus, and eliminates some of the surface defects liable to occur in the heating

operations of hot-rolling, while the thickness of the steel sheet is controlled more uniformly owing to the greater ease of obtaining uniform pressure during cold-rolling. The structure of the steel is also finer and more uniform than that normally produced by hot-rolling.

#### Bearing Metals

For some years a considerable amount of research has been carried out on bearing metals containing little or no tin. Until recently the chief development that has resulted has been the introduction of lead alloys containing small proportions of alkali and alkaline-earth metals. The most important application of these alloys has been for the bearings of railway wagons and coaches, particularly in Germany. In the very important field of bearings for internal combustion engines, lead alloys containing tin or those without tin have so far found no important application. In view of their superior merits, particularly at the elevated temperatures reached in these engines, tin-base alloys have been almost exclusively used and have undoubtedly given excellent service.

Recent developments, particularly in the increasing use of relatively high-revolution engines giving high power per unit of weight, have imposed especially difficult conditions on the bearings with respect to pressure, temperature, and rapid changes in loading. These conditions have become more severe than in any other important class of engine or machine and have resulted in increasing difficulties in obtaining satisfactory service from the tin-base alloys normally used. A type of failure which is the most serious is the cracking of the white metal. It would appear that the conditions of service are advancing to a stage that calls for definite improvements in tin-base alloys, or, alternatively, for the substitution of some other type of alloy, such as lead bronze (copper-lead), which is now receiving attention, and certain cadmium alloys.

With regard to the failures of tin-base alloys, metallographic examination has been made to ascertain the manner in which cracking occurs. In the light of the data acquired an analysis has been made of the factors involved. It appears that there are certain directions whereby the trouble may be mitigated by modifications in design and in the nature of the liner used. A paper which incorporates suggestions along these lines has been published in the "Journal of the Institute of Metals." In addition, some preliminary results of investigations directed towards improvement of the fatigue-resisting properties of the tin-base alloys are incorporated in this paper, which indicate that improvement is possible without any marked diminution in the tin content of the white metal.

Investigations on the effect of varying casting conditions, both as regards the temperature of the metal and the temperature of the mould have been made, and the tensile properties used as a guide to the physical properties of the metal. The results, some of which have been published, are being correlated with the data obtained by pounding tests and fatigue tests.

#### Corrosion of Solders

In various specifications for solder amounts of antimony up to 3 per cent. are permitted. Since 1 per cent. of antimony replaces 2 per cent. of tin this means a diminution of the tin content of several important classes of solders by no less than 6 per cent. Further, the authorisation of relatively high antimony content encourages the use of a considerable amount of secondary or recovered tin in the manufacture of solder. It therefore appeared important to review the whole position regarding antimony.

The resistance of solders to corrosion is also important and in this connection a large number of technical inquiries have been received. In a number of cases, particularly where food products have been concerned, it has been possible to indicate definite advantages in the use of high-tin solders.

Inquiries have also been received as to the most suitable type of solders for rapid soldering operations, *e.g.*, in can making. Here the solder has to sweat into small crevices, and fluidity is likely to be the deciding factor in the soundness of the joint. Certain impurities, particularly liable to be introduced when secondary tin is used in the manufacture of the solder, may have a direct bearing on a number of anomalies in behaviour that have been observed. An adequate study involves consideration of surface tension and viscosity of the solder and the interfacial tensions between the solder, flux and metal surface to be soldered.

### Cable Sheathing and Water Pipes

In Great Britain there has been a definite development of the use of ternary lead alloys for cable sheathing in conditions involving vibration. The lead-tin-cadmium alloy has a somewhat lower resistance to fatigue than the lead-antimony-cadmium alloy, but evidence has been obtained that it has a higher resistance to soil corrosion. Recent work indicates that the tin content is the important factor as regards corrosion resistance, and alloys containing 2 per cent. and 3 per cent. of tin are used in conditions in which this is important.

The alloy containing 1.5 per cent. of tin and 0.25 per cent. of cadmium has found increasing application in certain districts for water pipes, owing to the advantages of light weight and improved resistance to attack by soft water. A recent competitor is lead containing a small amount of tellurium (about 0.05 to 0.06 per cent.). Another important direction in which tin may find an increased use in water pipes arises from the fact that certain waters have a solvent action on lead and all the lead alloys proposed for pipes. There are grounds, therefore, for anticipating that a tin-lined pipe, whether of lead or one of the forementioned alloys, would be so superior as to justify the increase in cost that would be involved. Although tin-lined lead pipes are at present being manufactured, it is considered that in their present form they are unsuitable to meet adequately the competition of pipes of other materials and that a lighter pipe is required, such as may be obtained by the use of a relatively strong lead alloy lined with tin or a tin alloy.

### Addition of Other Elements to Tin

Knowledge of the effect of additions to tin is more lacking than in the case of any other important metal. This is an important direction of research for increasing the possible applications of tin. Careful consideration was given to the initial direction of the work and it was decided that in the early stages it should cover four important aspects:—(1) A quantitative study of the effect of such metals as are almost invariably present in tin as a result of contamination during manufacture, *e.g.*, iron and copper during hot-tinning. (2) A quantitative study of the effect of such metals as are used to strengthen the alloy for use in bearing metals, *e.g.*, antimony, cadmium, etc. (3) The effect of adding metals which would not convert non-toxic tin into a toxic alloy, *e.g.*, nickel, cobalt, bismuth, silver, etc., chiefly in relation to improvements in tin coatings and tin alloys for use in contact with foodstuffs. (4) The effect of certain rare elements, such as have given remarkable results in other fields, *e.g.*, beryllium in copper and silver, and tellurium in lead.

Work on the addition to tin of copper, iron, cadmium, nickel, cobalt, silver and bismuth has made rapid progress and an important section of the work has been published in the *Journal of the Institute of Metals*. In the investigation of the effect of small amounts of germanium and beryllium on tin there have been experimental difficulties to overcome, but suitable alloying methods have been worked out, and the constitution and physical properties are being determined. At a later stage the corrosion resistance of these alloys will be examined. In this sphere of activity the Council is not only making a valuable contribution to general metallurgical knowledge of tin, but is also acquiring much information which will have a direct bearing on the future industrial development and applications of the metal.

The most important outlets for tin foil at the present time are for wrapping certain types of cheese and to a lesser extent, chocolates and tobacco. The troubles which occur owing to blackening of the cheese in contact with the foil

received much consideration by the Tin Research and Industrial Applications Committee. They have been largely eliminated by the practice of coating the tin foil with a transparent protective lacquer. The possibility of improving the quality of tin foil itself is now being considered in connection with the above and other applications. Contacts have been established with various tin foil manufacturers with respect to the problem of producing tin foil free from pinholes and of suitable physical qualities, *e.g.*, to withstand wrapping operations without perforations appearing at the folded edges.

### Tin in Steel

Copper was once regarded as a deleterious constituent in steel, but comparatively recent research has shown that it is beneficial from the point of view of corrosion resistance. As a result large tonnages of steels containing small amounts of copper are now being used for structural and other purposes. The quantity of tinplate scrap on the market is so large that the question of the effect of tin in steel has received considerable attention by steel metallurgists. At present it is excluded as an addition to the steel furnace. This stimulates the detinning of tinplate scrap which is one of the main sources of secondary tin. Moreover, the difficulty of detinning articles made from tinplate tends to become in certain cases a definite drawback to their use. It has been found that small amounts of tin in steel lead to difficulties in working, causing cracks during rolling or subsequent operations. Since such investigations as have been carried out have been confined to certain types of steel only, it was considered possible that by employing different steels, and varying the methods of working, more favourable results might be obtained.

Research has been carried out to ascertain the effect of tin on a number of steels, and it has been found possible to roll and forge satisfactorily steels containing up to 0.75 per cent. tin. The research is being continued in order to determine whether any advantage as regards resistance to corrosion is obtained by the presence of tin.

### A Promising New Application

A promising new use for tin seems likely to have been established as a result of investigations carried out to produce a satisfactory tin accumulator. It was known that attempts have been made from time to time to utilise certain valuable properties of tin as an electrode, but without any notable success. Some preliminary investigations on the merits of tin electrodes were in progress for the Council when a patent relating to improvements in this direction was published and communication was therefore established with the inventor.

Methods of collaboration with a view to stimulating interest in the accumulator were discussed with the inventor and with an important technical group which has already acquired an interest in it. As a result a number of accumulators have been sent to the Council and are being subjected to a wide range of tests. Meanwhile a brochure describing the accumulator and its possible advantages has been prepared and distributed as one of the Council's technical publications. The essential features of the cell are electrodes of tin and lead peroxide respectively, in an electrolyte consisting of dilute sulphuric acid. The whole of the cell is filled with porous acid-resisting ceramic material in which the acid is absorbed, so that it is in effect a dry cell. Since no gas is evolved during use, it may be sealed after charging, thus obviating leakage of acid, and may be used even in a horizontal or other position. A special feature is that the tin accumulator is lighter than a lead accumulator of similar capacity. Many possible applications of such a cell are already evident, and may be considerably extended as a result of further research.

SURFACES coated with oil-sludge are cleaned by phenol, cresol, or other coal-tar phenols, with or without diluents, *e.g.*, tar distillates or petroleum distillates. For example, oil heaters and oil-filled electric transformers are freed from sludge by filling the emptied vessel to a suitable height with commercial carbolic acid, injecting steam for about 2 hours to throw the acid over the whole of the surfaces, running off the acid, and rinsing the vessel with dilute alkali and with water. (See Specification No. 22,832 of A. Duckham and Co., Ltd., and S. E. Bowrey.)

# Alloy Steels To-day

By PROFESSOR D. HANSON, D.Sc.

This article has been abridged from the second of the series which is being specially published by the Bureau of Information on Nickel, Mond Nickel Co., Ltd. The first article, by Professor Sir Harold Carpenter, D.Sc., F.R.S., appeared in THE CHEMICAL AGE, July 7, 1934 (Metallurgical Section, page 3).

In order fully to understand the wide range of alloy steels now in use and the varied purposes to which they are put, it is necessary to have some understanding of what they are, and, in particular, of the rôles played by the various alloy elements used. Steel is essentially an alloy of iron and carbon, and its characteristic properties and their alteration by heat-treatment depend on the fact that the carbon may exist in different conditions in the material.

We have, firstly, the condition in which the carbon takes the form of small particles of carbide of iron embedded in crystals of the iron itself; these are the pearlitic steels, characteristic of slowly cooled carbon steels, which are relatively soft and ductile, and are so largely used for structural work and other parts in which a moderate degree of strength and toughness is required. Secondly, we have the condition in which the carbon is dissolved in the alpha variety of iron; these are the martensitic steels, obtained by the rapid cooling or quenching of carbon steel, and represent the alloy in its hardest form; by appropriate tempering, the degree of hardness can be regulated to any desired extent. The third condition is that in which the carbon is completely dissolved in the gamma variety of iron, the so-called austenitic steels, which are only found in the presence of appropriate amounts of certain alloying elements: they are harder and tougher than the pearlitic steels of the same composition, and possess a considerable capacity for cold work, but none of them has the intense hardness associated with the martensitic steels.

## Action of Alloying Elements

The influence of alloying elements on steel may be considered under two main headings: (a) their effects on the transformations of the steels, (b) specific effects characteristic of the alloying elements used. Influences coming under both these headings are important in relation to the use of alloy steels, but those falling under heading (a) are particularly vital, since they govern some of the most extensive uses of alloy steels in practice. Every engineer knows that, whereas it is quite an easy matter to harden a small piece of carbon tool steel and to obtain it in its hardest possible condition, it is a very different matter to secure the same result with larger articles such as die blocks or other complicated or massive tools. Difficulties arise not only in securing the requisite hardening effect with larger masses, but severe warping and cracking are likely to occur, especially with articles of complicated shape, due to the severe stresses set up by the water-quenching process which must be used.

These difficulties are due to the fact that the critical cooling velocity for carbon steels is very high; in other words, the steel must be cooled very rapidly through its critical point if the hard or martensitic condition is to be obtained, and unless this condition results, the whole process of heat-treatment fails. With ordinary carbon steels it is, in fact, difficult to obtain complete hardening throughout any piece which exceeds about an inch in thickness, and, if the manganese content be low, even this small size may be too large to enable proper hardening to be obtained.

Most of the alloying elements used in steels reduce the critical cooling velocity for hardening, that is, they make it possible to harden the material when slower rates of cooling are used, or, alternatively, to secure the fully hardened condition in large masses. Thus, for all except the smallest parts, the use of an alloy steel is essential if the advantages of heat-treatment are to be obtained. Even where the use of alloy steel is not made essential by the difficulty of securing complete hardening by quenching, it is often advantageous in reducing the risks of cracking and warping by allowing substitution of oil-quenching for the more drastic process of water-quenching.

Although the number of elements added to steel is relatively small, there being only 7 or 8 which find extensive application, nevertheless, when we take into account the wide range

of carbon contents, the varying amount of each alloying element and the many combinations of alloying elements used together, it is found that the total number of alloy steels now in use is very great, running into hundreds of different compositions.

The intensity of the hardening effect which can be produced in any steel depends more on the carbon content than on any other individual factor; in the absence of this element, the amount of hardening that can be obtained by quenching is small or absent, no matter what other constituent is added to the steel, and the intensity of the hardness obtainable increases with rise of carbon content. The ease with which hardening may be obtained differs enormously from steel to steel and depends primarily on those elements other than carbon which are alloyed with it. Manganese, chromium, molybdenum and nickel are all very effective in facilitating hardening, or, in other words, reducing the critical cooling velocity necessary for complete hardening to be obtained.

## Air-Hardening Steels

The air-hardening steels contain one or more of these elements in sufficient quantity to produce this effect; for example, a 0.4 per cent. carbon steel containing 5 to 10 per cent. of manganese becomes martensitic when cooled at ordinary rates without quenching; 8 to 20 per cent. of nickel produces a similar effect. In practice, for reasons associated with the specific influences of the individual metals themselves, it is more usual to secure air-hardening properties by the combined influence of two or more added elements; a steel containing 0.3 per cent. of carbon,  $\frac{3}{2}$  to 5 per cent. of nickel, and 1.0 to 1.5 per cent. of chromium is typical of air-hardening steels commonly in use, and, in this material, cooling in air is just as effective as quenching in oil in producing a hard product, while the dangers of cracking and warping through intense quenching stresses are largely eliminated.

Similar but less intense effects are produced by the use of vanadium, tungsten or cobalt. Silicon, on the other hand, acts in the opposite sense, making the suppression of the normal transformation more difficult by raising the temperature of the critical points into a region in which internal changes of this kind take place extremely rapidly. In practice, nickel, chromium and manganese are the most important alloying elements affecting ease of hardening; molybdenum is also effective, and it is employed to some extent, usually with nickel and chromium, but its use is limited by its high cost.

## Nickel and Manganese

The complete suppression of all transformations on cooling with the formation of austenite is specially favoured by manganese and nickel; in a 0.4 per cent. carbon steel, more than 10 per cent. of manganese or 20 per cent. of nickel is effective in this respect, while the influence of chromium is of the same order as that of nickel, although it varies more with the carbon content than does the effect of nickel. Nickel readily induces austenitic conditions when the carbon content is quite low, whereas this effect cannot be produced in low carbon steels by the influence of chromium alone, except when rapid cooling methods are employed. The influence of molybdenum, vanadium, tungsten and cobalt in producing austenitic steels is quite small within the limits employed in steel manufacture. Silicon acts in the opposite sense to nickel and manganese in rendering more difficult the production of austenite, and when present in sufficient amount, prevents this constituent forming under any circumstances. Nickel and manganese are therefore the principal alloying elements employed when the austenitic condition is desired.

Frequently a combination of nickel and chromium is employed in practice in the austenitic steels, some of the nickel being replaced by chromium in order to develop corrosion-

resisting properties to a high degree. The well known 18/8 alloy containing 18 per cent. of chromium and 8 per cent. of nickel is a case in point, but many other variations in composition, such as 13 per cent. of chromium and 13 per cent. of nickel, or 18 per cent. chromium and 14 per cent. nickel, are also used.

In addition to their effect on hardening power, alloying elements may act on the carbon in the steel producing carbides other than  $Fe_3C$ . The production of such constituents may influence the steel in a number of ways; as a rule, such carbides differ from iron carbides in their solubility in iron; tungsten carbide, for example, requires a much higher temperature for complete solution than does iron carbide, and a tungsten steel contains undissolved particles of carbide after the ordinary heat-treatment involving iron carbide has been applied to it. Particles of free carbide, as these are called, constitute hard spots in the steel, whose presence may or may not be desired. In structural steels, for example, in which the greatest possible homogeneity is normally required, the presence of such a constituent would be undesirable, but in tool steels, particles of free carbide frequently contribute to the cutting properties and are therefore advantageous. Tungsten, molybdenum, vanadium, manganese and chromium all facilitate the production of free carbides by combining with some of the carbon present in the steel.

### Carbide Forming Elements

Nickel and silicon do not form carbides; on the other hand, they tend to decompose iron carbide itself. This effect of nickel is not encountered in the main with steels, but in the case of silicon, the influence in this respect is particularly great, to such an extent that when used as an alloying element in steels required to be subjected to a hardening treatment, silicon must be counteracted by including a carbide-forming element such as manganese, as in silico-manganese spring steels, in which 2 per cent. of silicon is usually associated with 1 per cent. of manganese.

In addition to the above general effects, each alloying element has certain specific effects that are its own individual characteristics and cannot be classified under any general heading. Perhaps the most remarkable is the influence of chromium on the resistance to attack by chemical agencies, particularly oxidation and a wide range of acids. This is, without doubt, connected with the great capacity of this metal to form protective films under the influence of a wide variety of conditions. The stainless and rustless chromium-containing steels owe their properties to this quality in the material, and the specific nature of the effect in so far as chromium is concerned is well brought out by the similarity of the metal itself, in the form of chromium plate, with the stainless steels, the corrosion-resisting properties of the two being closely similar in their general characteristics. The film-forming qualities of chromium are also well maintained when the steel is subjected to oxidation at elevated temperatures, and the chromium-bearing steels are finding useful application by virtue of their resistance to scaling.

### Stability Under Heat

Tungsten and molybdenum have certain important specific effects which are so similar in character that they may be classified together. Both these elements confer on steel great stability under heat, particularly by maintaining high mechanical strength and hardness at high temperatures; this is no doubt largely due to their very high melting points and intrinsic hardnesses. This quality is particularly valuable in connection with cutting tools, since steels containing these elements in sufficient quantity retain great strength and hardness even at a red heat, and the development of high-speed cutting steels depends mainly on this characteristic.

Both tungsten and molybdenum greatly raise the normal tempering temperatures of steels, even when present in quite small quantities, and this makes possible the development of structural steels possessing high elastic limits which are stable over a wide range of temperatures.

Nickel is perhaps the most versatile of all the elements added to steel. It unites with iron or steel in all proportions, and useful alloys are obtained with practically every conceivable ratio of the two elements. Low percentages of nickel increase the strength, yield point and hardness without a corresponding loss in ductility, in both the annealed and heat-

treated steels; steels containing 20 to 30 per cent. of nickel are non-magnetic, after cooling at ordinary rates to atmospheric temperature, and steels within this range find many applications for which a non-magnetic material of high strength is required; between 30 and 40 per cent. of nickel, the coefficient of linear expansion is very low, and 35 to 36 per cent. of nickel produces an alloy which has practically a zero coefficient.

An alloy containing 50 per cent. of nickel has high permeability at low field strengths, and it and other combinations containing still more nickel are finding increasing application on account of their magnetic properties. The possibility of adding considerable amounts of nickel to iron without reducing ductility is no doubt due to the fact that iron and nickel combine in all proportions to give ductile workable alloys; nickel in the quantities added in the alloy steels dissolves in the iron without forming a carbide and furnishes a strong ductile basis very suitable for still further improvement by the addition of the elements of the carbide-forming group.

## Recent Metallurgical Patents

### Refining Iron

IN a process for refining iron in a blast or cupola furnace in the presence of refining agents carried into the furnace by the air blast, the slag is expelled continuously and rapidly by the air blast or gases generated in the furnace through an aperture or apertures which are permanently open. The apertures may be provided above the liquid level in the furnace or the tapping holes for the slag are left open. (See Specification No. 27,081 of W. Klepsch.)

### Corrosion-Resistant Alloys

CORROSION resistant articles, particularly ship fittings which have to resist the action of sea water, and chemical plant, are made from aluminium alloys containing up to 10 per cent. of magnesium, with or without up to 5 per cent. of cadmium, up to 5 per cent. of antimony, up to 5 per cent. of bismuth, up to 3 per cent. of chromium, up to 5 per cent. of cobalt, or silicon up to such an amount as is required to form  $Mg_2Si$ . (See Specification No. 14,751 of K. Schmidt Ges.)

### Sintered Metal Compositions

A HARD metal sintered composition contains about 55-93 per cent. of tungsten carbide, 5-30 per cent. of tantalum carbide, and 2-20 per cent. of a binding metal of the iron group. The finely powdered mixture of the constituents is pressed, subjected to a preliminary sintering, preferably in a neutral or reducing atmosphere, shaped as desired, and then subjected to a final sintering. The mixture may be compacted during sintering. The preliminary sintering may be omitted. (See Specification No. 376,912 of Firth-Sterling Steel Co.)

### Extracting Gold

IN a process for recovering gold from colloidal materials, the material is formed into a slurry with aqueous cyanide solution, and during agitation of the mass there are added a depolarising agent, which may be aluminium sulphate, and an alkali, which may be lime. After settling, the clear solution is siphoned off and treated with zinc or carbon for the precipitation of the gold. The zinc may be arranged in a compartmented trough, that from the first compartment being periodically removed, roasted, fluxed, and smelted for the final recovery of the gold. (See Specification No. 21,040 of B. G. Nicholl.)

### Pickling Metals

RUST and oxide coatings are removed from iron, copper, brass, aluminium, tin, by a sulphonic acid of the benzene or naphthalene series which may be substituted in the nucleus by methyl, hydroxyl, a halogen, a nitro-group, or the like, but does not contain a side chain having more than 1 carbon atom. A little mineral acid may be present as impurity with little detriment. Acids specified are the sulphonic acids of benzene, toluene, phenol, and cresol; they may contain one or more sulphonic groups. Treatment with a 10 per cent. solution at 30-40° C. is suitable. (See Specification No. 29,691 of Chemische Fabrikpyros.)



# Metallurgical Section

October 6, 1934

## A Wider Use for Tin in Industry

THE researches which are in progress under the direction of the International Tin Research and Development Council have been planned to investigate basic problems in all the major applications of tin. There are apparently two important reasons why it has proved desirable to cover a wide range of subjects in the research programme. In the first place, work in one direction is usually found to help in a number of other directions, provided that all the activities are adequately co-ordinated. Secondly, modern industry has now been educated to expect technical assistance and advice from primary producers of the chief metals, as a result of the valuable services to industry which have been rendered by the research organisations they have established. The Council has already received numerous technical inquiries from the tin-consuming industries, and their first general report, recently published, indicates that this service has become an important branch of activity. Satisfactory answers to technical inquiries, it is pointed out, require intimate and extensive knowledge of a wide variety of subjects, usually greater than that which is already available in the industry, so that even if the research programme had been restricted to a few major lines the necessity of starting work on all the chief applications of tin would most certainly have arisen.

In planning the researches which are at present in hand the main policy which has been adopted is to serve the needs of the industries connected with the existing markets for products containing tin, and at the same time to conduct fundamental researches on the properties and reactions of tin in order to develop new outlets in the future. The need for retaining and strengthening the existing applications is stated to be urgent, because with practically all metals both loss and gain of market is taking place, due to new discoveries and technical developments.

### Researches on Tinplate

It was recognised in the initial planning of the programme that research on practically all aspects of tinplate is of vital importance, since tinplate forms by far the greatest single outlet for tin. Any important developments in favour of or against the use of this product would therefore have an immediate important effect upon the total consumption of tin. Even the best tinplate is not perfect and this tends to retard expansion and even to encourage contraction in certain important applications. Very great importance may therefore be attached to means for improving the tin coating on tinplate. Continuous efforts are made by the tinplate manufacturers to keep the percentage of

the visible defects as low as possible, but the proportion of prime plates is seldom greater than 75 per cent. and is often considerably less. The need for more exhaustive examination into the causes of defects has therefore been evident. Apart from visible defects, however, there are invisible defects which at the present time are not subjected to regular scrutiny. These defects consist chiefly of minute discontinuities in the coating, whereby a certain amount of steel is left uncovered by tin, which causes rapid rusting to occur when tinplate is exposed to moist atmospheric conditions, and the production of hydrogen gas when the tinplate is in contact with acid products. The latter effect is a matter of very great importance where tinplate is used in the canning industry.

Some of the large tinplate manufacturers have already carried out extensive researches on tinplate. The most important improvements that have taken place have been in the manufacture of the steel sheet for tinning. The actual tinning operation, however, has never been subjected to close metallurgical scrutiny, and what work has been done has led to very few published results and, therefore, is not available in the interests of the industry as a whole. The Council's chief researches on tinplate, which have been carried out after discussions with tinplate manufacturers, can makers and canning research establishments, comprise (1) researches on defects which are present in tinplate as manufactured; (2) researches on the further defects that are produced when tinplate is deformed, *e.g.*, during the process of can manufacture; and (3) researches on the behaviour of tinplate in contact with various corrosive media, particularly of the type met in canned foodstuffs.

### A Mirror-Like Nickel Coating

THERE has recently been evolved an improved procedure for obtaining a mirror finish on tinplate by suitable polishing, followed by the electrodeposition of nickel. A mirror-like coating of nickel requiring no further polishing is thereby formed on the tinplate surface. Since the polishing of steel to a mirror surface prior to nickeling is more expensive than the polishing of a tinned steel surface, the use of tinplate instead of steel sheet gives an economic advantage. The nickel deposit may be followed by a thin coating of chromium, so that brilliant chromium finishes as well as mirror-like nickel surfaces can be secured on tinplate for fabrication into fancy as well as utilitarian articles. Some troubles have arisen from the peeling of the nickel or nickel-chromium coating from the sheets, but the general view is held that this trouble will ultimately be overcome and

an increasingly important new outlet for tinplate will be secured. Researches are at present in progress on the factors involved in obtaining the requisite adhesion of nickel and nickel-chromium deposits to tin.

### Alloys from Powdered Metals

DURING the last few years considerable attention has been focussed on the sintering of metals mixed in powder form, at suitable temperatures, to produce special alloys. In this development tin powder is frequently used. For instance, it is possible to mix finely divided tin and copper in suitable proportions, and, by heating under reducing conditions, obtain a bronze of quite good mechanical properties. It is, however, also possible by this procedure to incorporate substances which cannot be satisfactorily incorporated by the usual methods of alloying. In the bronze alloys formed by this process, graphite can be incorporated so as to be uniformly distributed throughout the alloy. This is the basis of the oil-less type of bearing which has found an important application in the automobile and electrical industries, and which is eminently suitable for bearings not subjected to heavy loads but which (through inaccessibility or for other reasons) are required to function without attention to lubrication. The possibility of introducing various additions to tin which could not be normally introduced by the usual procedure of melting and casting may even have an important bearing upon the problem of obtaining a superior tin-base bearing metal. It has already been claimed that a small amount of finely-divided graphite dispersed through a lead-base bearing metal considerably reduces the coefficient of friction of the bearing. In view of such claims as this, the International Tin Research and Development Council is studying the effect of such additions on high tin-base alloys.

### Cyaniding Practice in Canada

THE commercial production of zinc dust in Canada has now increased to about 100 tons per year, and at this figure it represents about 40 per cent. of the apparent Canadian consumption in gold milling, where zinc dust is used as a precipitating agent in the cyanide extraction process. The total consumption in Canada in 1933 amounted to about 556,000 lb., as against 530,000 lb. in 1932. It varied from a minimum of 0.04 lb. to a maximum of 0.2 lb. per ton of ore milled. The dust varies in composition, but generally contains about 90 per cent. of metallic zinc and 10 per cent. of zinc oxide, the latter being present as a film upon the surface of the metallic particles. At most of the Canadian mines the Merrill-Crowe combination process, of deoxidising the solution and precipitating the gold by the addition of zinc dust, is now adopted for the reason that this process assures a fairly uniform product for refining. In comparison with earlier practice where zinc shavings were used, the use of zinc dust also reduces the actual consumption of zinc and cyanide, and gives a more complete precipitation of the gold. At some of the mines, small quantities of lead acetate or lead nitrate are added to the solution to intensify the precipitating action.

Zinc dust has also been used in Canada as a precipitant for silver, but has been discontinued because it was unsatisfactory in cases where the

cyanide solution contained appreciable amounts of arsenic and antimony; in addition, the zinc had a tendency to foul the solution and reduce its silver-dissolving power. As an alternative, in silver milling practice in Province Ontario, aluminium dust is employed and the silver is recovered as a clean high-grade precipitate, without any tendency for the cyanide solution to become fouled. Another mine adopts sodium sulphide as a precipitating agent, the resulting silver sulphide being desulphurised by contact with aluminium ingots in a caustic soda solution.

### Covering Capacity of Bronze Powder

THE covering capacity of aluminium bronze powder has been found to vary from 3,000 to 12,000 sq. cm. of surface per gram of powder, the low values applying to the so-called "varnish" grades and the high values to some of the "lining" powders. The density of aluminium bronze powder being about 2.5, the above-mentioned limits of covering capacity correspond to flake thicknesses ranging from 0.0013 to 0.0003 mm. According to "Industrial and Engineering Chemistry," May, 1934, the method adopted for the measurement of flake thickness is based on the assumption that if all the flakes in a given weight of powder could be separated out into a layer one flake thick and packed close enough to eliminate the interstices between adjoining flakes, the thickness of the flakes could be calculated from the area of the film, its weight and the density of the powder. A film which approximates to these conditions can be obtained by the proper manipulation of the powder on a clean surface of water. A shallow rectangular pan with a flat rim is used as a container for the water and two flat rigid strips of glass or metal which act as barriers are laid across the width of the pan to define the ends of the film of powder whose area is to be measured. A weighed amount of powder is carefully dusted upon the water surface which completely fills the pan, and the flat barriers, resting on the rim of the pan near each end, are said to perform the very important function of "coaxing" the powder into a film which is one flake thick.

### Ferrous Metals in Acid Soils

By comparing the average rates of pitting of ferrous metal specimens, which have been buried for 8 and 10 years, with values for the total acidity of the soils at the test sites, the United States Bureau of Standards has found that a definite relation exists between acidity and pitting. A similar relation was obtained between the loss in weight of specimens in a short-time laboratory test and the total acidity of the soils. The effect of soil acidity in inducing corrosiveness was attributed to its action in preventing the formation of protective layers of ferrous and ferric hydroxides on the metal surface. In neutral and slightly alkaline soils the hydroxide film tends to reduce the accessibility of oxygen at the cathode surface, thereby reducing the rate of corrosion. The effect on corrosion of the physical characteristics of soils which determine their permeability to air was studied by comparing data that measured permeability with an expression for the ratio of uncorroded to corroded area. The results indicate that the greater the permeability of the soils (exclusive of sands), the greater is the tendency for pitting.

# Progress in Non-Ferrous Metallurgy

## Autumn Meeting of the Institute of Metals

THE 26th annual autumn meeting of the Institute of Metals was held in Manchester, September 3-6, when a large number of papers on non-ferrous metallurgy were read and discussed. On this occasion the 13th Autumn Lecture was delivered by Dr. J. L. Haughton, his subject being "The Work of Walter Rosenhain."

General considerations on the improvement of white bearing metals for severe service formed the subject of a paper by Mr. D. J. Macnaughtan, director of research, International Tin Research and Development Council, London. He pointed out that development in the internal combustion engine is imposing increasingly severe conditions on the bearings. Consideration was given to the theoretical functions of an ideal white metal, and the manner in which the stresses produced in service tend to cause failure by cracking. Since the normal action of the stresses are compressive, special attention was also given to the tension stresses, which were shown to lower the fatigue range of the metal and to open up incipient cracks. Based on this analysis the mechanism of crack formation was discussed. Improvement in service behaviour may be secured by (1) diminishing the intensity of the stresses in the metal by modifications in (a) certain features of design; (b) the material used for the liner; (2) increasing the fatigue-resisting properties of the white bearing metal, in respect to which results obtained in preliminary investigations of the fatigue properties of high tin-antimony-copper alloys with and without addition of a further element were given.

### Corrosion of Tin Alloys

Some data on the corrosion of tin and its alloys.—Part 1.—The tin-rich tin-antimony-copper alloys, was reported by Mr. T. P. Hoar, M.A., Ph.D., University Metallurgical Laboratories, Cambridge. The tin-rich tin-antimony-copper alloys were examined with regard to their resistance to corrosion by dilute hydrochloric and citric acids and by various tap-waters. The straight 5 per cent. antimony alloy containing no copper was found to be usually somewhat more resistant to these types of corrosion than alloys containing copper. Soft water produces tarnishing; hard water gives no tarnish, but may give localised attack if there is much chalky deposit. This work indicates the desirability of incorporating about 5 per cent. antimony in tin from the corrosion-resisting standpoint, but suggests that the presence of 1-3 per cent. copper as well is deleterious. Tin and its alloys with small amounts of antimony and copper are corroded by dilute citric and hydrochloric acids in the presence of air. The addition of 5 per cent. antimony effects a marked reduction of the corrosion in hydrochloric acid, but is without much effect in citric acid.

Properties of heavily cold-worked nickel were discussed by Mr. H. Quinney, University Engineering Laboratory, Cambridge. The Curie point of commercial nickel of 99.62 per cent. purity has been found to be 330° C., which is much lower than that generally accepted for pure nickel. After the metal has been subjected to torsional overstrain the Curie point on heating is raised considerably, but returns to its original value on cooling. No such effect is observed with mild steel, since the strain is relieved before the metal reaches the relatively high Curie point.

### Influence of Pickling

The influence of pickling on the fatigue-strength of Duralumin was the subject of a joint paper by Mr. H. Sutton, M.Sc., and Mr. W. J. Taylor, both of the Royal Aircraft Establishment, South Farnborough. In order to assist in the detection of flaws, manufacturing defects, and fatigue cracks in aluminium alloy components it has been customary to pickle or etch the components in various solutions, the main etching usually being effected in an aqueous solution of caustic soda. Wöhler-type fatigue tests were made on test-pieces machined from Duralumin bar and the effect of various pickling treatments on the fatigue-strength, was investigated. The investigation has shown that pickling in caustic soda

solutions followed by cleaning in nitric-sulphuric acid solutions effects a substantial lowering of the Wöhler limit of Duralumin. The effect of the treatment is to produce a rough, serrated surface. When a layer 0.0025 in. thick has been machined off the surface, the pickled material shows normal properties. Pickling treatment in nitric-hydrofluoric acid solution effects much less reduction in fatigue range of Duralumin, and pickling treatment in sulphuric-hydrofluoric solution followed by 50 per cent. nitric, only very slight reduction. The latter treatment reveals the macrostructure of Duralumin parts and appears suitable for the examination of pieces at various stages of manufacture for defects such as forging cracks, laps, and discontinuities.

### Tarnishing of Highly Polished Metals

A reflectivity method for measuring the tarnishing of highly-polished metals was described by Mr. L. Kenworthy, M.Sc., A.R.C.S., British Non-Ferrous Metals Research Association, London, and Mr. J. M. Waldram, B.Sc., A.C.G.I., Research Laboratories of the General Electric Co., Ltd., Wembley. The method involves the separate determinations of the specular and diffuse components of reflection, and the use of an empirical formula combining these two properties. The application and validity of the method are illustrated by the results of periodical measurements, and observations made on specimens of pure tin and 2 tin alloys exposed to indoor and outdoor atmospheres.

Crystal re-orientation on heating drawn copper wires was discussed in a joint paper by Mr. G. S. Farnham, M.Sc., The University, Manchester, and Mr. Hugh O'Neill, M.Met., D.Sc., Department of Metallurgy, The University, Manchester. The behaviour of a silver-free copper wire reduced 50 per cent. by cold-drawing, was compared after "low-temperature treatment" (L.T.T.) with that of two silver-bearing wires reduced 50 per cent. and 40 per cent., respectively. L.T.T. hardening occurs in the first, but not in the last of these. X-ray spectroscopy makes it evident that preferred orientation is less developed in the silver-bearing wires. The general effect of L.T.T. at 130° C. is to reduce the amount of (111) preferment, but to cause an increase of (100) preferment. This change-over probably causes "orientation hardening."

### Crystal Densities of Brasses

Crystal densities of industrial brasses from X-ray data was dealt with by Professor E. A. Owen, D.Sc., and Mr. Llewelyn Pickup, M.Sc. (Lond.), Ph.D. (Wales), both of the University College of North Wales, Bangor. Crystal density values have been obtained from X-ray data of copper-zinc alloys in thermal equilibrium, for compositions in the  $\alpha$ -, ( $\alpha + \beta$ ), and  $\beta$ -regions. Whilst it is shown that the degree of porosity, cold-work, and grain-size have no effect on the results obtained, it is essential to take into account the heat-treatment given, when interpreting the values of alloys with duplex structures. The relation between the composition of the pure  $\alpha$ -phase and its density is not strictly linear, but both that of alloys in the pure  $\beta$ -region and that in the ( $\alpha + \beta$ ) region can be taken as linear to a high degree of accuracy. At the phase boundaries ( $\alpha$ )-(  $\alpha + \beta$ ) and ( $\alpha + \beta$ )- $\beta$ ) there are discontinuities in this relation. Densities derived from X-ray data are considered to be nearer the true values than those obtained by any of the other methods hitherto employed, and hence, for copper-zinc alloys in true equilibrium, the values obtained are the most reliable standards available for comparison purposes.

Properties of tin containing small amounts of silver, iron, nickel, or copper were discussed in a joint paper by Professor D. Hanson, D.Sc., Mr. E. J. Sandford, B.Sc., and Mr. H. Stevens, M.Sc., all of the University of Birmingham. The tin-rich ends of the silver-tin, nickel-tin, and copper-tin equilibrium diagrams were investigated by the authors. With the first, the eutectic occurs at 3.5 per cent. of silver, at 221.3° C.; with the second, at 0.18 per cent. of nickel at a temperature which does not vary appreciably from the melt-

ing point of pure tin; with the last, between 0.70 and 0.75 per cent. of copper at 226.9° C. The solid solubility of silver in tin is shown to be approximately 0.02 per cent. at room temperature, increasing to 0.06 per cent. at 210° C. The solid solubility of nickel is less than 0.005 per cent., and that of copper less than 0.01 per cent. at 226° C. The method of making additions to tin was discussed, and it was shown that no particular difficulties are met with in the case of silver, iron, copper, and nickel. The influence of additions of these metals on the tensile strength of tin was also discussed. A great increase produced by quenching silver-tin alloys is not permanent at room temperature, whilst with the other three alloys quenching has no effect. Additions of iron above 0.4 per cent. are without effect, although up to this percentage an increase of 40 per cent. in the tensile strength is found. Nickel up to 0.3 per cent. produces an increase up to 2.1 tons/in.<sup>2</sup>, but further additions have no influence. Copper up to 2 per cent. greatly increases the tensile strength after all heat-treatments investigated. Silver is shown to refine the grain of tin, but not to prevent grain-growth at high temperatures. The addition of iron above 0.05 per cent. or of nickel above 0.06 per cent. prevents such grain-growth.

The deformation lines in alpha brass were dealt with by Mr. Carl H. Samans, Ch.E., M.S., Ph.D., Chase Brass and Copper Co., Waterbury, Conn., United States. A microscopic study of 70:30 brass single crystals of two different orientations which had been reduced 50 per cent. in thickness by cold-rolling revealed the presence of many of the so-called "lines of deformation." X-ray determinations, by the Davey-Wilson method, of the orientations in the rolling plane showed conclusively that the markings were mechanical twins parallel to octahedral planes.

In his paper on the spectrographic detection and estimation of minute quantities of impurities in copper, Mr. M. Milbourn, B.Sc., I.C.I. Metals, Ltd., Birmingham, described an accurate and convenient method for the detection and estimation of small quantities of bismuth, arsenic, lead, iron, nickel, silver, antimony, and tin. Details of technique, sensitivity, and line intensity comparisons were given.

The spectrographic analysis of some alloys of aluminium was dealt with by Mr. Ernest H. S. van Someren, B.Sc. He described the technique of analysing aluminium alloys by means of their spark spectra in the ultra-violet, using the method of internal standards. Tables were given for the estimation of copper, zinc, iron, silicon, manganese, magnesium, nickel, tin, and cadmium; and also for the detection of lead, antimony, chromium, titanium, and bismuth.

A "synthetic spectrum" method of analysis and its application to the quantitative estimation of small quantities of bismuth in copper was the subject of a paper by Mr. D. M. Smith, A.R.C.S., B.Sc., D.I.C., British Non-Ferrous Metals Research Association. The author said a method has been devised for the production of "synthetic" spectra as standards for comparison, the spectrum of a standard alloy being exactly superimposed on that of the pure metal which forms the main constituent. The total time of the two exposures is equal to the normal time of exposure of a sample which is being analysed, and a series of spectra is obtained in which the impurity lines show a systematic increase in intensity. While the method was originally applied to the checking of the reliability of standard samples, it can be used for the quantitative determination of impurities in metals, once the standard calibration curve has been obtained. The application to the determination of 0.0001 to 0.004 per cent. of bismuth in copper was described.

## Precious Metals from Jewellers' Waste

### Technique of a Simple Recovery Process

THE use of arsenic for collecting small amounts of precious metals contained in jewellers' waste has been known for many years, but it is only of recent date that the process has been so modified that it may be carried out by the jeweller himself.

If jewellers' waste is melted with lead and arsenic, the gold and silver and any small percentages of platinum and palladium passes into the lead, whilst minute proportions of the others, namely, iridium, osmium, rhodium, and ruthenium is collected by the arsenic. That is, if jewellers' waste is melted on lead containing arsenic, by skimming off the crusts which form on the surface of the molten lead, the four of the rare metals are completely recovered.

The working of the process calls for no previous skill or experience. The material is first melted up with metallic lead. As the impurities present invariably contain a little sulphur, which would form a layer on top of the lead (known as "matte") it is customary deliberately to add some iron pyrites, and cover the lot by adding a little slag. That is, three layers are formed, namely, the lead at the bottom, the "matte" in the middle of the mass, and the slag on top. By this means, the surface of the lead containing precious metals is protected by the "matte" where any small amount of metal (lost to the lead) may be reclaimed, whilst the slag may be skimmed off and thrown out as waste.

Lead containing arsenic is first charged into a crucible, or preferably a fat-shaped receptacle which is most suitable for absorption purposes. Iron pyrites are next added, and then a covering of slag, and the whole kept molten. An accumulation consisting of all classes of jewellers' waste is then added, and the mass kept molten for some time. In order to ensure a perfect division between the lead and "matte," a little scrap iron is added which makes the "matte" lighter; this iron is added after the mass has been well melted. The slag is then skimmed off and thrown out, and the lead is run off from the bottom, if possible, as the appearance when pouring assists in making an easy separation from the "matte." The "matte" which is separated may contain some silver, and may be accumulated and treated by itself.

The lead is skimmed by means of perforated ladles, which remove all arsenical skimmings containing the iridium, osmium, etc., but in so doing an excess of lead is also removed, which, however, is preferable as a safety measure. This excess lead has to be reduced, and so the lead skimmings are returned to the melting pot, and again melted with about one-third of their bulk of iron pyrites, and a covering of slag.

On pouring, a separation is made as before. In this case, however, the lead does not necessarily require to be skimmed, as the more concentrated arsenic compounds solidify readily on top of the lead when it is poured into a mould, and it may, therefore, be separated from the harder product by lifting off in a cake of whitish brittle material. This is known in the trade as "white-metal," and is further reduced in bulk in the usual way by sulphurising, using the well-known "top and bottom" process employed in nickel works.

The "white-metal" is powdered and mixed with its own weight of powdered coal and three times its weight of salt-cake, and fused at a dull red heat. The mass at first swells up, and then settles down to a quiescent thin fluid, which is poured into an iron mould. The "white-metal" will now be reduced to about one-third of its bulk, but to ensure a still further concentration, the resulting metal is again ground to powder, and the above process repeated twice more. Any gold which may have gone into the original skimmings (instead of into the lead) is now to be found in the thin fluid mass which solidifies on top of the metal. The latter is now termed "speiss" and finds a ready market with the smelters of precious metals without further treatment.

When the "speiss" reaches the precious metal firms, it is dissolved in nitric acid, and other acids, and a series of separations made, so as to recover the different constituents individually. Where insoluble matter has to be dealt with, it is fused with barium peroxide and barium nitrate. The lead remaining after the arsenical skimmings have been removed is cupelled and parted in the usual way to recover silver, gold, and platinum.

## Some Recent Metallurgical Patents

### Iron Alloys

IRON alloys consist of substantially pure iron, up to 1 per cent. of copper, and an amount of silicon, or titanium or both which is not more than about two-fifths of the copper present, the material during manufacture being deoxidised by such an amount of titanium, or of silicon and titanium, as to ensure complete deoxidation and to leave the required proportion of silicon or titanium or both in the product, and the copper being added preferably at the end of the refining process. (See Specification No. 409,380, of American Rolling Mill Co.)

### Iron and Steel

IN a process for the production of iron and steel which do not increase in brittleness appreciably on ageing, in standard Siemens-Martin furnaces without additional deoxidation, a portion of the manganese of the charge is maintained in the molten metal and a part of the manganese which has already passed into the slag is recovered for the metal by adding silica at the end of the melting down period for reducing the basicity of the slag. The added silica may be glass, sand, refractory material, etc. (See Specification No. 410,256, of Vereinigte Stahlwerke A.-G., and J. Peters.)

### Purifying Steel

In a process for dephosphorising steel in an induction furnace, the molten steel is treated therein with a molten and very fluid basic slag which covers and protects the surface of the metal against oxidation by the air substantially from the beginning of the operation, an intense agitation of the metal being produced by the electric field. Slags comprising silica, lime iron oxide and a flux such as fluorspar, sodium or potassium salts, etc., are referred to and in an example a slag comprises 3 per cent. of silica, 60 of lime, 35 of iron oxide and 2 of fluorspar. (See Specification No. 405,633 of Société D'Electro-Chimie, D'Electro-Metallurgie et Des Acieries Electrique D'Ugine.)

### Treating Metallic Surfaces

Colouring or corrosion-inhibiting substances are deposited in oxide coatings which have been produced chemically or electrolytically on aluminium or aluminium alloy surfaces and the coatings are then treated with water at above 80° C. as described in the parent Specification. Alternatively, in the case of corrosion-inhibiting substances such as chromic acid, chromates, dichromates, or silicates, the treatments may be combined by using an aqueous solution of the corrosion-inhibiting substance maintained at a temperature of above 80° C. The colouring substance used may be an organic dye or a precipitate of an inorganic pigment, and the duration of the hot water treatment is preferably 5-40 minutes. (See Specification No. 32,130 of Aluminium Colors, Inc.)

### Ore Concentration by Flotation

Ores containing sulphide minerals of copper and nickel are concentrated by flotation by treating the ore pulp with a cyanide prior to the flotation so as to inhibit the flotation of the nickel mineral. The pulp is preferably made alkaline and zinc sulphate may be added to improve the recovery of the copper. After the removal of the copper, the pulp may be again subjected to flotation, preferably after addition of copper sulphate, to recover the nickel. The concentration of an ore containing copper sulphide (chalcocopyrites), nickel sulphide (pentlandite), and iron sulphide (pyrrhotite) is described, the one being first ground to free the minerals. The pulp is made alkaline by the addition of lime and a collecting agent such as amyl xanthate is also added, these additions being preferably made during the grinding stage. A cyanide of an alkali or alkaline earth metal, zinc sulphate, and pine oil are then mixed with the pulp and a copper concentrate separated by flotation, this concentrate being cleaned with the addition of lime. Copper sulphate is then added to the reject from the first flotation and the nickel is recovered by a further flotation using a collecting agent such as sodium ethyl xanthate. (See Specification No. 240,929 of International Nickel Co., Canada.)

### Concentrating Ores

PHOSPHATES in phosphate rocks are concentrated by agitating a pulp of the rock with a naphthenic acid material and separating the concentrate either by aerating to form a froth or by passing the pulp over a vibrating table. The pulp is preferably alkaline, and insoluble and unsaponifiable oil, such as fuel oil, is preferably added. The naphthenic acid may be saponified before addition to the pulp by treating the naphthenic acid material with an excess of caustic soda. (See Specification No. 409,514, of Phosphate Recovery Corporation.)

### Aluminium Alloys

Aluminium alloys contain 2-12 per cent. of copper and 0.005-0.1 per cent. of tin, with or without a total of 0.02-3 per cent. of one or more of the hardening elements manganese, chromium, boron, molybdenum, zirconium, beryllium, and titanium, and with or without a total of 0.1-14 per cent. of one or more of the elements zinc, nickel, and silicon, the proportion of nickel not exceeding 7 per cent. The cast or wrought alloys are artificially aged at about 100-200° C., and this treatment may be preceded by heat-treatment at over 400° C. (See Specification Nos. 3,569 and 3,570 of Aluminium, Ltd.)

### Carburising

IN the method of applying a heat-stable and acid-resisting coating to the surface of an article as described in the parent specification, the surface layer of the article to be carburised contains one or more carbide-forming elements other than or in addition to those of the chromium group. The elements referred to are iron, silicon, boron, cerium, titanium, zirconium, vanadium and tantalum. The coating may be applied mechanically, by chemical or electrolytic deposition, by diffusion or by sintering or fusing and the article to be coated may be composed of metal, porcelain, ceramic material, sintered alloys or porous masses, such as artificial stone material and artificial resin. (See Specification No. 400,719, of Deutsche Edelstahlwerke A.-G., and E. F. Kruppa.)

### Cemented Carbides

A process for making hard cemented carbide material includes forming a hard carbide containing free carbon, mixing the carbide with a metal or metals of the group iron, cobalt, and nickel, and a metal capable of forming a hard carbide, or an oxide of such a metal, and sintering the mixture to decarbonise it in respect of free carbon. Hard carbides containing free carbon are formed by carburising metals or their oxides. Mixed carbides may be formed by reducing or oxidising mixed salt crystals or double salts to form alloys or oxides which are subsequently carburised. The metal added for decarbonising may be the same as or different from the metal of the carbide to which it is added. The mixture may be hot pressed, or cold pressed and sintered twice at different temperatures. (See Specification No. 32,606 of A. H. Stevens.)

### Coherent Masses from Powdered Metals

Iron sponge, preferably mixed with additional substances, is pressed to form bodies which are then extruded. The specific gravity after the initial pressing may lie between 3.5 and 8, and the body may then have ferrochromium, metal powders, phosphorus, or other materials diffused into its surface, by inserting it in an excess of the material and heating to red heat. By suitable choice of pressures, speeds, and temperatures in the extrusion operation products containing 0.24, 0.11, or 0.045 per cent. of carbon may be obtained from a preliminary block containing 1.7 per cent. of carbon. The materials mixed with the sponge may control the working properties, the strength, the resistance to corrosion, or the stability on heating. Chromium, ferrochrome, chrome alloys, phosphorus, ferrophosphorus, copper phosphide, or copper may be used alone or in combination. Chromium, nickel, and molybdenum jointly, or their compounds, may also be



used. To obtain a product containing 0.2-2 per cent. of phosphorus, 1-10 per cent. of 20 per cent. ferro-phosphorus may be added, while for one containing 0.2-0.65 per cent. of sulphur 0.7-2 per cent. of sulphur-iron may be added. To improve the properties of stress and hardness, molybdenum, vanadium, cobalt, or other metals, or non-metals such as carbon and silicon may be added. Basic or acid Siemens Martin, Thomas, Bessemer, blast furnace, or electro-furnace slag may be added to produce a product resembling wrought iron. (See Specification No. 494 of H. Esser.)

#### Treating Iron-Silicon Alloys

In a method of treating iron-silicon alloys, especially silicon steels containing 1-6 per cent. of silicon, to improve their electrical properties, the alloys are hot rolled, annealed, cold rolled or worked to produce an elongation of at least 1 per cent. and/or to introduce strain into the alloy and then reannealed at 1,600° F. or higher. The initial stage of the hot rolling is carried out at approximately 1,500° F. and the metal is subjected to a number of passes through hot rolls, the last pass being made at a temperature just below the critical temperature. The cold working or rolling is also carried to such an extent that the magnetic permeability of the alloy is increased preferably to at least 10 per cent., the watt loss being also decreased at least 15 per cent. The silicon steel sheets may be used for the manufacture of transformer and armature cores. (See Specification No. 10,555 of E. M. Freeland.)

## Electroplating Industry

### New British Standard Specifications

THE first two of a new series of British Standard Specifications for the plating industry have been published by the British Standards Institution. They are B.S.S. No. 558 (1934), Nickel Anodes for Electroplating, and B.S.S. No. 564 (1934), Nickel Ammonium Sulphate and Nickel Sulphate for Electroplating. These specifications have been prepared at the request of the Electrodepositors' Technical Society, who, amongst other interested organisations, have assisted in their preparation.

In the electroplating industry the purity of materials employed is of the highest importance if good work is to be produced. It is well known that frequently plating jobs are spoiled through defects in the solution which defy all attempts of the experienced plater to discover. In such cases the failure is usually attributed to the impurity of one or other of the chemicals used or possibly to some excess of impurity in the anode. These British Standard Specifications have been prepared to reduce to a minimum the possibilities of failure; they require that the material shall be up to a high standard of quality and that any impurity must not be present in excess of the maximum specified. By specifying "British Standard" the risk of a spoiled plating bath is considerably reduced. It is hoped, therefore, that full use of these new British Standards will be made by purchasers as well as by suppliers of electroplating materials.

Copies of these specifications may be obtained from the Publications Department, British Standards Institution, 28 Victoria Street, London, S.W.1, price 2s. 2d. each, post free.

### Thickness Tests for Tin Coatings

THE International Tin Research and Development Council has issued as Technical Publication, Series A, No. 12, a reprint of a paper by Dr. S. G. Clarke, published in "The Analyst," which describes a new rapid method of determining the thickness of a tin coating on steel. The procedure is simple: the tin is dissolved from a known area by a cold solution of hydrochloric acid and antimony chloride, and its amount is determined by the loss of weight of the specimen. The basis metal is unattacked, but a small correction has to be applied to allow for the layer of tin-iron alloy. In addition to brevity, the method has the advantage that the thickness of tin on a given portion of the surface can be determined by protecting the remainder with a coating of cellulose varnish. Copies of this paper may be obtained on application to the International Tin Research and Development Council, Manfield House, 378 Strand, London, W.C.2.

## Electrodeposition of Nickel

### A Review of Recent Developments

THE theory and practice of electrodeposition have made enormous strides during the past decade. New processes have been developed, additional metals have been brought within their scope, and the whole industry has been placed upon a surer, more scientific foundation.

The problems involved in the electrodeposition of nickel have received a great deal of attention, and a large amount of information resulting from scientific investigations has been placed at the disposal of the industry. Much of this information is so widely scattered, however, that many readers of the Metallurgical Section of THE CHEMICAL AGE will be interested in a review of recent developments which has been prepared by Mr. A. W. Hethersall, and now published by the Bureau of Information on Nickel (Publication L5).

Dealing first with the importance of the preliminary cleaning processes, Mr. Hethersall notes that it is only recently that the mechanism of these processes has become clearly understood, and that the necessity for this has been largely stimulated by the fact that subsequent chromium plating calls for a high degree of adhesion of the underlying electrodeposited nickel. This knowledge, coupled with developments in the use of solvent vapour cleaning and cathodic alkaline cleaning, has enabled considerable improvements to be made in the removal of grease and other foreign matter. Etching of the surface of brass and copper has been found to give very beneficial results, and highly adherent deposits have been obtained after anodic treatment in suitable solutions. Vastly improved adhesion has also been obtained on steel by anodic etching, and this method is tending to displace the older methods of dipping in dilute sulphuric or hydrochloric acid solutions.

Difficulties encountered in nickel plating zinc or zinc-base die castings, due to the tendency of the zinc to dissolve in the nickel-plating solution, are now being overcome in different ways, while in the case of aluminium, attempts to secure adherent deposits have been chiefly directed to devising satisfactory means of producing an adherent immersion deposit on the surface to provide a sound foundation for the subsequently applied deposit. To this end a number of different dipping solutions have been suggested.

Research into the causes of defects in nickel deposits, particularly of pitting, has thrown new light on the mechanism of nickel deposition and has indicated methods for the control of the properties of nickel deposits. It has been established, for example, that precipitated basic matter in the solution adjacent to the cathode is chiefly responsible for controlling the hardness and general characteristics of nickel deposits, and that the amount of basic material precipitated is markedly influenced by the solution pH. From the work carried out on pitting, the conclusions are drawn that, if suitable precautions are taken to avoid contact of the solution with organic material, freedom from pitting may be secured by operation of low pH, and in solutions used at higher pH, the tendency for pitting may be reduced by the introduction of sodium or potassium salts.

Investigations have been carried out recently on the effect of pH on the throwing power of nickel solutions and also on the mechanical properties of electrodeposited nickel, while the influence on nickel deposition of chromic acid in the solution has been studied, and means suggested for its satisfactory removal.

THE Copper Development Association, whose offices are at Thames House, Millbank, London, S.W.1, was formed in 1933 by the principal members of the British copper industry and includes among its activities the collection and dissemination of all kinds of information relating to copper and to materials containing copper. With this object in view it is proposed to issue from time to time publications dealing with the technical and other aspects of the use of copper and its alloys in their many industrial and scientific applications. A short historical review entitled "Copper Through the Ages" has been prepared as the forerunner of these publications.

# Metallurgical Section

November 3, 1934

## The Blistering of Iron Oxide Scales

IN works practice, the blistering of the scale during the re-heating of steel for rolling into sheets may have a deleterious effect on the final product. Beneath the blisters oxidation continues at a reduced rate, resulting in differences of level for the steel surface, and also in the formation of a secondary scale deposit. Variations in the thickness and tenacity of this scale may result in an irregular surface in the finished sheet. Where the sheet metal is used for special purposes an excessive amount of rubbing down may be required, and in the case of tinplate the material will be of inferior quality unless the tin coating be heavy. Mr. R. Griffiths, of the Department of Metallurgy, University College, Swansea, gave attention to this matter in an investigation reported to the Iron and Steel Institute at its September meeting. The experimental work described was carried out in order to throw further light on the cause and prevention of scale blistering, and also to ascertain conditions for the formation of a non-adherent scale.

Mr. Griffiths put forward an explanation to account for the blistering of the oxide in air and the absence of blistering in oxygen. He pointed out that there is a volume change of the order of 100 per cent. when iron is oxidised, and stresses are set up which tend to disrupt the scale and loosen it from the underlying metal. The tendency for cavities to form, due to the lifting of the scale by the stresses, would be assisted by the presence of a non-oxidising gas in the atmosphere, which might diffuse through the scale and collect in the cavity, thereby reducing the stress necessary to continue the expansion of the cavity. The stresses exerted by scale in its formation cannot be very great, in that particles of foreign matter placed on the surface of the scale during oxidation are not forced to remain at the surface but become buried. If a blister is formed during oxidation in oxygen, the exposed surface of the steel beneath the blister will be rapidly attacked by any oxygen present in the blister, so creating a vacuum, and the blister will immediately collapse. It thus appears to be necessary for an inert or non-oxidising gas to be present to support the blister after its formation.

Experimental work indicated that a number of factors were involved when a non-adherent scale was formed. It appears necessary to have a rigid, compact type of scale, such as is produced when oxidation is carried out in air to which steam has been added. The composition of the base material also has an influence. The relative coefficients of expansion of the scale and steel are important, in that the steel, on cooling down, would tend to contract more than the scale, and, given the

optimum rate of cooling, the steel would detach itself from the scale envelope. In those cases where the oxide has penetrated around the grain boundaries, with steels containing silicon or phosphorus, the strong hold of the scale on the steel would prevent detachment, but the presence of a high percentage of manganese appears to overcome the latter effect by assisting in the formation of a compact and stronger scale. Rates of heating and cooling also have an influence on the formation of a non-adherent scale, for when these rates are low a tenacious scale is gradually formed on heating, which on cooling slowly can accommodate itself to the dimensional changes.

Blistering of the scale may occur at temperatures of oxidation between 850° C. and 1,050° C. on steels which oxidise appreciably, regardless of the composition of the steel, but the presence of an inert gas is necessary for the blistering to take place to any extent. The presence of water vapour in sufficient amounts in the oxidising atmosphere prevents blistering. By the addition of steam to oxygen or air, the surface of the steel, after removal of the scale, is found to be clean and free from oxide. The composition of the base material, the condition of the oxide envelope and the rates of heating and cooling, however, govern the conditions for the formation of a non-adherent scale.

### Desulphurisation of Pig Iron

THE desulphurisation of pig iron is discussed in Report of Investigations No. 3240, recently published by the United States Bureau of Mines, which is of special interest in view of the fact that the elimination of sulphur from pig iron is an important feature of present-day blast furnace practice. In one direction, attention has been directed to the effect of temperature on the desulphurising action of manganese, independent of slag reactions. Laboratory tests show that metal containing several tenths of 1 per cent. of sulphur can be desulphurised by allowing the metal to cool, provided the residual manganese approximates 2 per cent. The product, per cent. Mn  $\times$  per cent. S, approaches a constant value at a given temperature; normal variations in silicon and manganese have little effect upon the constancy of this product. From the data obtained the sulphur content of the metal between 1250° C. and 1500° C. can be predicted for any manganese content. Mechanical contamination of cast metal can be prevented by removing the solid layer of manganese sulphide and kish, and skimming the metal during the pouring operations. The possibilities of depending less upon slag-metal reactions for desulphurisation, and more upon the use of high-manganese

iron in plants using high-sulphur coke are outlined briefly. Small-scale tests indicate that low-sulphur metal and a roasted product, suitable for producing ferro-manganese, can be produced by treating high-phosphorus spiegel with iron sulphide.

### Steel Industry Developments

THE Workington Iron and Steel Co., which is a branch of the United Steel Companies, Ltd., has now completed the construction of its new acid Bessemer steel plant, which is the only plant of its kind in Great Britain. Acid Bessemer steel, as manufactured at Workington, will be made entirely from molten pig iron produced from Cumberland raw materials by mechanically-charged blast furnaces. At these works three blast furnaces will be producing 7,500 tons of iron per week, one of the furnaces operating at the high rate of 3,000 tons per week. The new plant, which is controlled by electricity throughout, consists of two 25-ton acid Bessemer converters, and will have a capacity of 7,000 tons of steel ingots per week. This development in the manufacture of high quality acid Bessemer steel should do much to improve the position of the British steel industry.

Another outstanding event of the month concerns the production of stainless steel. Under the arrangement which was entered into some months ago for the amalgamation of stainless steel interests of Thos. Firth and John Brown, Ltd., at Sheffield, with those of Vickers, Ltd., a new company is being registered under the name of Firth-Vickers Stainless-Steels, Ltd. This new company will operate plants which have been considerably extended since the fusion was decided upon. It is understood that facilities for the production of stainless steels, with subsequent manipulation into sheets and bars, has already been increased at the two individual works by about 50 per cent.

### British Blast Furnaces

IN its first report to the Iron and Steel Institute, the Blast Furnace Practice Sub-Committee, which is a sub-committee of the Blast Furnace Committee of the Iron and Steel Industrial Research Council, lays emphasis upon the low output per furnace in Great Britain as compared with that in the Dominions. It states that the average production capacity per furnace is actually lower in this country than in any of the other important iron producing countries of the world, and points to four reasons which must be taken into consideration.

In the first place Great Britain was a pioneer iron producer, and consequently a number of the older and smaller furnaces are still in operation. Secondly, the blast furnaces in Great Britain serve a widely varying market and flexibility has therefore been important. In the third place, it should be noted that the furnaces in Great Britain which have the lowest production capacities are those using low grade iron-bearing materials; fourthly, British manufacturers have not given very much attention to the necessity for grading materials, which has been a matter of importance in American and Continental blast furnace practice. Nevertheless, the output per furnace has steadily risen during recent years, due partly to the shutting-down of some of the older plants, and partly to the gradual increase of productive capacity of those in operation.

Financial limitations alone have retarded the rate of progress in this direction, as any substantial increase of output at some of the older works would necessitate expenditure on auxiliary equipment as well as on the blast furnace itself, and this has been almost impossible under the depressed conditions in the industry.

The difficulties of maintaining the output of large producing units during the past three or four years appear to provide a justification of the British policy of smaller blast furnaces and greater flexibility. In addition, with unsized and ungraded ore of low iron content, a prolonged time of contact in the furnace is essential, and this has been a matter of importance in limiting furnace output. With improved ores which are graded and (if possible) concentrated, a considerable increase in furnace output may be anticipated. Future progress, it is stated, will largely depend upon the necessary capital becoming available for the modernisation of the older plants of the country, and the technical solution of the problems of the treatment of materials—particularly ores and coke—involved in the furnace reactions.

### Renewed Interest in Molybdenum

ABOUT 60 patents dealing with molybdenum as a component of alloys were granted in the United States during 1932. In most of them, the molybdenum content is comparatively small, and in others molybdenum seems to have been introduced with the idea of "not missing anything" rather than with a definite idea of its having a specific function, for it is given as one of a half-a-dozen or more elements that "may be added" in very small quantity.

According to J. L. Gregg ("The Alloys of Iron and Molybdenum," 1932), molybdenum, when used alone, seems to have little effect on "un-heat-treated" steel. In heat-treated wrought or cast steels with more than 0.3 per cent. carbon, less than 1 per cent. of molybdenum substantially increases the tensile strength; this effect is reduced with less than 0.3 per cent. carbon. Several tenths of 1 per cent. of molybdenum increase the strength of low-carbon steel at high temperatures, but may increase susceptibility to chemical attack. Used to the extent of 0.5 per cent., it increases the tensile and transverse strength, endurance limit, and hardness, improves the high-temperature properties of grey cast iron, and apparently does not lower machinability. Steels containing 0.15 to 0.25 per cent. of molybdenum and 0.5 to 1.1 per cent. of chromium, with or without 0.4 to 0.7 per cent. of manganese, are now used extensively in the automobile industry; steels containing 2 to 3 per cent. of nickel in place of the manganese are also used, but in smaller quantity. Molybdenum (0.2 to 1 per cent.) is used generally in the so-called nitriding steels, as it makes the extremely hard skin of iron nitride less brittle. The development of the use of molybdenum in the United States was largely due to Wills and Phillipson. After the companies with which Phillipson was connected had put large sums of money into a molybdenum mine and mill, the demand was too small to pay for the investment. Following the earlier work of Wills, however, Phillipson developed and advertised the value of molybdenum in quantities of 0.2 or 0.3 per cent. in machine steels and ultimately his company's plant had to be greatly enlarged.

# Electrodeposition of Nickel

THE more important advances which have been made in the technique of nickel plating during the last few years are discussed by Mr. A. W. Hotherhall in a booklet published by the Bureau of Information on Nickel, Mond Nickel Co., Ltd. The subject is dealt with under the headings of preliminary cleaning procedures; etching treatments for brass, copper and steel; nickel plating of zinc and aluminium; mechanism of nickel deposition; causes and prevention of pitting in nickel deposits; mechanical properties of nickel deposits (tensile strength and hardness, effect of heat-treatment, hardness and polishing); effects of nickel deposits on resistance to fatigue; and throwing power of nickel solutions. The information has been arranged to give real assistance to the practical plater.

## Mechanism of Nickel Deposition

Recent investigations have considerably advanced the fundamental knowledge of the mechanism of nickel deposition and have thus laid the foundations for further developments in the control and applications of nickel deposition. For some time it has been known that the hardness and general characteristics of nickel deposits could be varied over a wide range by altering the conditions of deposition, and it was previously supposed that these changes were largely controlled by the direct effect of co-deposited hydrogen. It has now been established ("Trans. Faraday Soc.," 1933, 29, 729) that the factor chiefly responsible for controlling the properties of nickel deposits is the presence of precipitated basic matter in the solution adjacent to the cathode. In nickel sulphate solutions containing boric acid, basic nickel compounds are visibly precipitated when the solution pH exceeds about 6.0 (quinhydrone) and at a higher value in the absence of boric acid or when the solution contains ammonium salts.

During electrolysis the migration and discharge of hydrogen ions from the solution immediately adjacent to the cathode raises its pH. This rise tends to be counteracted, especially in the outer layers of the film, by convection currents or by mechanical agitation of the solution. If, however, the rate of rise in pH is excessive, due to high current density, for example, visible evidence of precipitated nickel compounds is obtained in the form of green coloured matter on the cathode. Before this condition is reached, the deposit passes through successive changes in appearance, first increasing in brightness, then becoming cracked and possibly exfoliated, and finally dark and powdery or "burnt."

As might be expected, the solution pH exerts a predominant influence on the amount of basic material which is precipitated at the cathode face. In the investigation ("Trans. Faraday Soc.," 1933, 29, 729) which led to this conclusion it was found that, at high pH values, all solutions gave hard deposits and with a reduction in the pH the hardness fell in all solutions to a minimum figure—in general, for the conditions used, at about pH 4.7-5.0 (quinhydrone). Below this pH value, the behaviour of the various solutions investigated differed according to their composition, the greatest difference being produced by the presence or absence of chloride. However, in solutions of the compositions ordinarily used for the deposition of nickel (single or double salt solutions containing chlorides), little further change took place on continued reduction in pH (Fig. 1). The results described refer to deposition in highly purified solutions without agitation at a temperature of 35° C. and a current density of 11 amps per sq. ft. At higher current densities it is anticipated that the minimum hardness would be found at a lower pH value, and in solutions operated on a commercial scale, a modifying influence might be introduced by the presence of impurities, especially of organic matter.

## Pitting in Nickel Deposits

Observation of the appearance of a large number of nickel deposits prepared under controlled conditions in highly purified solutions ("Trans. Faraday Soc.," 1933, 29, 729) led to the conclusion that pitting was associated with the presence of flocculated basic matter at the cathode face. This material, accumulating around the base of a bubble, would increase its rigidity and adhesion to the cathode face, and the bubbles

## Precipitated Basic Matter and Pitting Troubles

of hydrogen constantly being formed at the cathode would therefore tend to adhere and thus cause pitting. As indicated previously, the physical condition of the precipitated basic material is influenced to a marked degree by the composition of the solution, whilst its amount at a given current density is also controlled to a considerable extent by the solution pH. Thus, at 11 amps per sq. ft. below pH 3.0 (quinhydrone) no pitting was experienced in solutions of the Watts single salt type, and below pH 2.6, in none of the solutions investigated. At higher pH values in solutions of the Watts type, the prevalence of pitting appeared to depend chiefly on the absence of alkali metals from the solution. In a solution free from potassium or sodium (the chloride being introduced as nickel chloride) pitting was very much more severe than when sodium or potassium were used in preparing the solution. This was attributed to the known stabilising effect of alkali

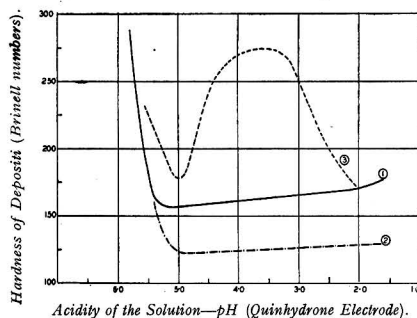


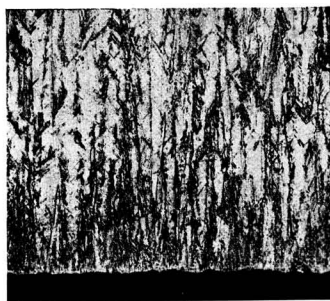
Fig. 1. Effect of pH on the hardness of deposits.

Curve 1.—Solution contained nickel sulphate, boric acid and potassium chloride. Curve 2.—Same as 1, but with nickel chloride instead of potassium chloride. Curve 3.—Same as 1, but without potassium chloride.

metal ions in preventing flocculation of the basic colloids precipitated at the cathode face.

The immediate practical conclusions which may be drawn from this work are therefore that, if suitable precautions are taken to avoid contact of the solution with organic material, (a) freedom from pitting may be secured by operation at low pH, and (b) the tendency for pitting may be reduced in solutions used at higher pH by the introduction of sodium or potassium salts.

Hydrogen peroxide, added as such or produced in the solution by the addition of sodium perborate, has now become of recognised value in commercial nickel-plating as a means of reducing or eliminating pitting. A proposed alternative ("Trans. Electrochem. Soc.," 1932, 62, 91) is the addition of nitric acid or nickel nitrate, the solution being operated at pH 4.8-5.7 (colorimetric equivalent to 4.3-5.7 quinhydrone) with an addition of 0.2 g. nickel nitrate  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  per litre (3 1/5 ounces per 100 gallons). Unlike hydrogen peroxide, nitric acid or nickel nitrate do not undergo spontaneous decomposition, and, as too high a concentration of these materials promotes the formation of bright, cracked deposits and reduces cathode efficiency and throwing power, care must be taken to avoid excessive additions. In a nickel-plating bath, excess of hydrogen peroxide is removed by spontaneous decomposition—slowly at low pH and rapidly at high pH. In contrast, however, to the above oxidising agents, the presence of small concentrations of chromic acid in nickel solutions derived, for example, from chromium-plating baths has been found ("Trans. Faraday Soc.," 1930, 26, 481) to exert a very detrimental influence on nickel deposition. Deposition was entirely prevented (hydrogen only being discharged) by concentrations of 0.22 and 0.05 g. (33 and 4/5 ozs. per 100 gallons)  $\text{CrO}_3$  per litre in the single and



× 100

As deposited.  
Brinell No.  
125.

× 100

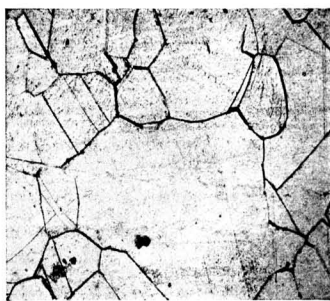
After annealing.  
Brinell No.  
71.5.

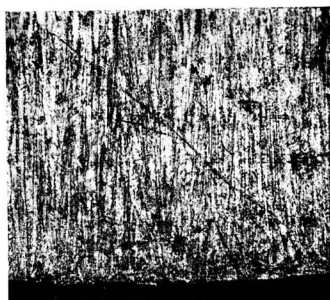
Fig. 2. Solution—  
nickel sulphate,  
boric acid, nickel  
chloride—pH 4.9.

double salt solutions respectively. Lower concentrations produced a marked reduction in cathode efficiency with the formation of bright and cracked or exfoliated deposits.

Removal of impurities from contaminated solutions has been found to effect a reduction or elimination of pitting. One process developed for this purpose ("Ind. Eng. Chem.," 1928, 20, 1,049) is that in which the solution is treated with chlorine, followed by the addition of finely divided Kieseluhr (afterwards removed by filtration). Another suggestion ("Monthly Review, Amer. Electroplaters' Soc.," Dec., 1933) based on the well-known adsorptive power of freshly precipitated ferric compounds for colloidal material, involves the addition of ferrous sulphate to high pH solutions, followed

This was found to be due to the presence of segregated oxide in the deposits, and a striking confirmation of the theory of the causes of variations in properties of nickel deposits was thus obtained ("Trans. Faraday Soc.," 1933, 29, 755), previous attempts to determine by direct methods the relative amounts of oxygen in hard and soft deposits having failed ("Trans. Faraday Soc.," 1930, 26, 89).

The greatest increase in ductility on annealing was found in the case of the softest deposit produced in the single salt solution free from alkali metals (Fig. 2). The more refined and less definitely oriented structure of the harder deposits formed in the double salt solution was found to result in the formation of small equiaxed grains on annealing (Fig. 4).



× 100

As deposited.  
Brinell No.  
156.

× 100

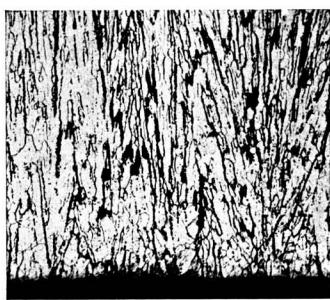
After annealing.  
Brinell No.  
69.9.

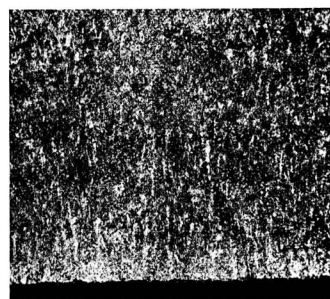
Fig. 3. Solution—  
nickel sulphate,  
boric acid, potas-  
sium chloride—  
pH 5.7.

by hydrogen peroxide to oxidise the iron and effect its precipitation. A third process ("Trans. Faraday Soc.," 1930, 26, 163) involves the oxidation of organic impurities by treatment with potassium permanganate (0.1-0.2 grams per litre) (1 3/5-3 1/5 oz. per 100 gallons).

In the case of applications where the deposited metal is to be subjected to high temperatures, conditions of deposition may be selected which avoid the considerable embrittlement produced on annealing certain types of deposit. Thus, in the Watts single salt solution (free from alkali metals) and in the double salt solution, considerable increase in ductility takes place on annealing over a wide range of deposition pH, whereas improvement is only secured in a narrow range of pH in the single salt solution containing alkali metals.

A greater amount of non-metallic material was therefore required to cause brittleness on annealing than in the case of the single salt deposits, in which there was a greater tendency for the non-metallic material to segregate into bands extending through the deposit at right angles to its surface (Fig. 3). In all cases the hardest deposits produced at high pH values became very brittle on annealing.

It has been found ("J. Electrodep. Tech. Soc.," 1930, 5, 63) that, when deposited on a smooth surface, the harder deposits are easier to polish because of their greater freedom from roughness. Surface roughness appears to be frequently a controlling factor in determining ease of polishing; a fact which has given rise to misconceptions as to the relative hardness of deposits, when judged by their ease of polishing.



× 100

As deposited.  
Brinell No.  
223.

× 100

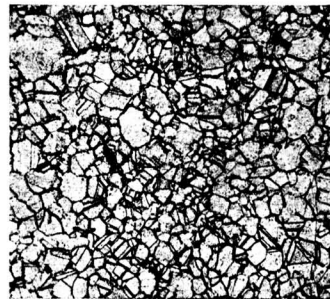
After annealing  
Brinell No.  
70.5.

Fig. 4. Solution—  
nickel sulphate,  
ammonium sul-  
phate, potassium  
chloride—pH 5.2



## Improved Qualities for Cast Irons

### Annual Report of the British Cast Iron Research Association

THE annual report of the British Cast Iron Research Association, for the year ended June 30, 1934, records the gratifying fact that the total income received for the year under review was the highest in the history of the Association.

The most important developments during the year refer to a means of achieving a greater degree of graphite refinement in cast iron, and are likely to apply to both pig and cast irons. While the immediate application is to grey irons, the principles involved will apply to the white, chilled, and malleable irons. The marked effect of size and distribution of graphite on the properties of cast iron is generally recognised. Much of the advance made in the past few years on strength of cast iron has been made by reducing graphite size, and this has been rendered possible mainly by the reduction of the total carbon content. The lowering of the total carbon, however, brings certain founding difficulties in its train. The metal is more liable to be mottled or white on pouring, has greater shrinkage in the foundry, and requires melting at higher temperatures, which may increase the difficulties with respect to refractoriness of moulding sands. The range of temperatures over which the metal is fluid enough to pour into sound castings is shorter.

#### Control of Melting Conditions

A few years ago the Association's senior metallurgist was able to demonstrate that by suitably controlling conditions of melting it was possible to secure irons, both not only with graphite in the ordinary flaky condition but also in an extremely finely divided condition known as "supercooled." Subsequently, the effect of various gases in the metal was studied and it was found possible to change the structure from one type to the other by treatment with suitable gases. The matter was carried further and it has been possible to evolve a process in which, by a suitable addition to the metal and a comparatively simple after-treatment while molten, it is practicable on a laboratory scale to secure graphite in the extremely divided supercooled form, regardless of the original total carbon content. Previous attempts to secure this finely divided graphite throughout a heavy section have been only partially successful. Patent protection for the process has been applied for in the interests of members, and commercial trials are being undertaken in members' foundries. By virtue of this new treatment it has been possible, on laboratory melts of 100 lb. or 200 lb., to increase mechanical properties of an ordinary No. 3 pig iron by 50 per cent., without alteration of composition.

#### Alloy Cast Irons

Further work has been done and several reports circulated on the heat-resisting cast irons Silal and Nicrosilal. About a dozen members of the Association are licensed to produce these alloys and their application in service presents a number of problems of considerable variety and interest. The original work carried out in the laboratories of the Association has now been confirmed in papers presented to scientific societies in or published in the technical Press of Germany, France, and Belgium, the latest paper being that by Professor Thyssen, of Liège, to the Iron and Steel Institute, on the occasion of its recent visit to Luxembourg.

A beginning has been made on the study of the effect of molybdenum, an effect which is generally acknowledged to be of a remarkable character for the size of the addition made. Further experiments have been made during the year in conjunction with members on the production of high quality alloy cast irons made by the inoculation process and members have been satisfied that the production of such irons is commercially quite practicable. One such iron, made by a member and containing about 1 per cent. nickel, was reported in a paper to the spring meeting of the Iron and Steel Institute as having a tensile strength of nearly 20 tons per square inch. This iron is a low carbon iron containing 1 per cent. nickel. There are definite commercial possibilities in high quality cast irons having strengths of this order.

During the year a report on Recommended Methods for the

Testing and Control of Foundry Moulding Sands has been issued and widely circulated, and it is now possible for any foundry to embark on the testing of moulding sand with access to all the necessary information on apparatus and technique. Full details of apparatus required and methods of procedure are given in the report and a large part of this apparatus has been designed and evolved by the Association. An appendix to the report gives information on the choice of apparatus, with manufacturers and prices, and a series of figures is also incorporated giving the results of tests on the recommended apparatus using sands from different parts of the country. A further important report circulated exclusively to members has been prepared on binders for core sands, a large number of binders—proprietary and otherwise—having been tested with various sands.

#### Melting Practice

The balanced blast cupola makes satisfactory progress. In the last report of the Association the number of cupolas installed or under construction was forty-one, having a total hourly tonnage of 275 tons. At present the number of cupolas installed or under construction is sixty-one, having a total hourly tonnage of 386 tons. Very satisfactory reports continue to come from users. One of these furnaces has been erected at the foundry of Preston and Bishop, Ltd., Clement Street, Birmingham. This furnace was erected in the main through the generosity of Mr. H. James Yates, Vice-President of the Association. The cupola has an internal diameter of 24 inches and a rated output of 2.35 tons per hour. It forms an important addition to the Association's experimental resources, not only for making experimental melts, but also for tests on fuels and refractories. Gratifying reports on the balanced blast cupola come from Australia, where the first one installed at the works of T. Main and Sons Pty., Ltd., of Melbourne. This firm has undertaken to manufacture and supply or convert suitable cupolas in Australia and a good deal of interest has been aroused by the successful operation of the first furnace which was operated solely on the Association's written instructions. Several other furnaces are under construction.

Some interesting figures were published during the year by the Scottish Coke Research Committee of tests on coke in various cupolas, and the consumption of charge coke was lower in the balanced blast cupola than in any of four other furnaces used. The charge coke consumption for the balanced blast cupola was 5.45 per cent. and ranged for the other furnaces up to 8.5 per cent. The results of an important series of tests by the staff of the Association have also been made available to members, giving figures for sulphur pick-up, carbon pick-up, metal loss, and production of slag in two different types of cupolas. The variation of composition in scrap normally makes these figures difficult to obtain, but, in the tests referred to, careful estimations were made of the whole of the material both entering and leaving the furnaces.

#### Development Department

The Development Department of the Association deals with technical inquiries from members and with application to members' works of the discoveries made in the laboratories. During the year over 1,100 inquiries were received from members and reported upon, and a large number of visits were paid to members in connection therewith. Work of an important and interesting character frequently arises from problems and inquiries submitted by members. Last year, following difficulties which were evident in the process of vitreous enamelling cast iron, two meetings of members were called to discuss the situation and a general report was subsequently circulated. This year the matter has been followed up experimentally with the aid of samples from members showing the difficulties complained of and a report has been prepared on the way the defects arise. Another report has been prepared on the causes of phosphide extrusions on the surface of castings, which have caused considerable difficulty from time to time in the industry.

During the year twenty-five research reports were issued or made available to members. Fifteen bureau reports and eight translations were also issued during the year. A complete list of research reports, bureau reports, and translations issued during the year is given as an appendix to the report.

The most important development during the year has been the scheme proposed by the Association for the establishment of a British foundry high school, completing the superstructure of the scheme of national foundry education referred to above. The proposals were approved by the Education Committee and later by the Council, and various bodies concerned have been invited to support the scheme.

It is proposed to establish a course in founding lasting one academic year and providing instruction of the highest type, open only to those who are already in the industry and who have demonstrated their capacity for exercising higher responsibilities in it. In the majority of cases candidates will have had suitable preliminary education or will have taken advantage of part-time facilities in the evening technical schools and colleges, now available throughout the country. The number of students will not be allowed to exceed a suitable maximum and it is hoped to arrange for the specialised teaching to be in the hands of nationally-known and recognised experts in the industry. It is proposed to pay visits each week to foundries and to have once a year a visit to other parts of the country. Such a course should appeal to those men who are, or who hope to become, foundry managers, foundry engineers, foundry chemists, foundry metallurgists.

During the year the University of Sheffield announced its willingness to establish a course in metallurgy suited to founders leading to a degree. There is no overlapping between the two schemes, for the Sheffield scheme will apply essentially to undergraduates who have had no experience of the trade, while the Association's proposals concern those already in the industry.

#### Finance

The accounts circulated to members with this year's report show a total income of £10,632, and a credit balance on the year's working of £258. Until the end of the year 1930-31 the Association was in receipt from H.M. Government of a grant of £ for £ on industrial subscriptions. The increase in industrial subscriptions from 1923 was of such a character that the grant gradually increased on this basis from £2,000 per annum to nearly £5,000 per annum. In 1931, however, a change took place in the conditions of grant and the Association was required to increase its industrial support to qualify for a diminished grant. In the succeeding three years, the

last of which is, the year now under review, the Association earned grant of £3,000 by receiving in industrial subscriptions £6,250, £6,500, and £7,000 respectively. The income from industrial sources plus the grant yielded, for the year ending June 30, 1934, an income in excess of that of any previous year, the very material drop in the grant having during this period been gradually offset.

As a result of negotiations with the Advisory Council of the D.S.I.R., a new grant offer was received from them on July 31, 1934. The terms of this offer actually apply to the current financial year, which began on July 1, and to the four years following. These terms are of a generous character and should stimulate the industry to qualify for the grant which the Government has undertaken to pay. Briefly, if the industry will increase its subscription income to the Association by £1,000 per annum, the Government will increase its grant by £2,000 per annum. If the industry will still further increase its support, the Government is prepared to pay £ for £ up to an additional limit of £3,500 during the five years for which the offer operates. The grant is thus increased in the first place from £3,000 to £5,000 and in communicating the offer the Advisory Council make it clear that the additional sum is designed to encourage long-range investigation which it is difficult for an individual firm to carry on, and difficult even for a research association to conduct without adequate funds and staff. By taking full advantage of the extended offer the Association can increase its total income to £20,000 per annum, £8,500 from grant, and £11,500 from the industry.

#### Support from Firms in the Industry

The Association must look in the main for this increased sum to those firms in the industry who, although eligible for membership, do not yet support it, and arrangements are being made to endeavour to secure their interest and adhesion. The offer necessitates raising not only £1,000 for this year but an additional £875 per annum for each of the four succeeding years.

At the close of the year the total number of ordinary and trade members was 308 and of associate members 61. Affiliated associations of manufacturers have a membership of 39, the total subscribing membership thus being 408.

Following his serious illness, Mr. J. E. Fletcher, M.I.Mech.E., who has acted as Consultant to the Association since 1924, was compelled to relinquish some of his active work, and the Council made arrangements for Mr. F. J. Cook, M.I.Mech.E., to advise on matters in which his great experience of the industry would be helpful.

## Some Recent Metallurgical Patents

### Refining Metals

MOLTEN metals and alloys are purified or refined by the addition of compounds of phosphorus or boron or both in admixture with ferrocyanides, particularly alkali ferrocyanides, with or without the addition of slag-forming materials such as silica, silicates, or alkalis. The treatment of copper, nickel, brass, bronze and German silver is referred to. (See Specification No. 413,881 of Deutsche Gold- und Silber-Scheideanstalt Vorm. Roessler.)

### Austentic Steel

AUSTENTIC steel, resistant to corrosion at about 500° C., containing 12-30 per cent. of chromium, 6-30 per cent. of nickel, and less than 0.3 per cent. of carbon, contains also niobium and tantalum, the proportion of niobium being greater than that of tantalum. The sum of the niobium and tantalum may be up to about 10 per cent., and is preferably at least 10 times the carbon content. The alloys are suitable for making fusion-welded articles, for example, oxyacetylene welded articles, and welding rods for making such welds. A number of nickel-chromium steels each containing a specified proportion of one of the metals vanadium, titanium, tungsten, uranium, zirconium, and tantalum, are also described by way of comparison. (See Specification No. 414,211 of F. M. Becket, and R. Franks.)

### Corrosion-Resisting Iron Alloys

A CORROSION-RESISTING iron alloy contains less than 0.05 per cent. of carbon, 0.2-0.6 per cent. of manganese, 0.2-0.6 per cent. of silicon, less than 0.02 per cent. of phosphorus, less than 0.015 per cent. of sulphur, 0.3-0.6 per cent. of copper, and 0.01-0.15 per cent. of tungsten. The alloy may be made, for example, in an open hearth or electric furnace, by refining under a very calcareous slag to obtain a very soft iron, removing the slag and forming a fresh lime slag, and adding tungsten and manganese, copper being added at any stage in the process. (See Specification No. 413,895 of Soc. Anon. des Hauts-Fourneaux Forges et Acieries de Pompey.)

### Magnesium and Manganese

MAGNESIUM, magnesium waste, and magnesium alloys are protected against oxidation during operations carried out at elevated temperatures, including melting and casting, by wetting the material with hydrocarbon oils, such as petroleum, or mineral oil, or distillation products thereof, which become vaporised at the temperature of treatment. Magnesium obtained by the processes described in Specifications 362,835 and 381,115, may be freed, in the solid state, from magnesium oxide and carbon by washing with hydrocarbon oil. (See Specification No. 414,577 of Oesterreichisch Amerikanische Magnesit Akt.-Ges.)

# Metallurgical Section

December 1, 1934

## Zinc Smelting in Great Britain

THE zinc industry, notwithstanding the expansion in output, reduced cost and improved efficiencies, has not up till now given the Imperial Smelting Corporation anything like a fair return on its capital expenditure. This was the view expressed by Sir Robert Horne, when the Corporation held its annual meeting on November 12. He was of the opinion that nothing else could be expected when there was a decline to the extent of 25 per cent. in the price of zinc in October as compared with the average of the last six months of 1933, which was by no means high.

In the past few years the Imperial Smelting Corporation has almost completely reorganised the zinc industry of this country. In 1932 production of zinc was under 24,000 tons; for the first six months of this year it was at the rate of 50,000 tons; and it is now nearly 70,000 tons per annum. With fair prices it might soon reach 75,000 tons. There is, however, an import duty of 10 per cent. on foreign zinc, and this import duty, if it had been made fully operative, would have given some help to all concerned. In October, 1932, however, when the imposition of such a duty was under consideration at Ottawa, an error was made which had the effect of weakening the position of the zinc producer in the United Kingdom. Under the Ottawa agreements the Empire producers—that is, those outside the United Kingdom—are required to sell their metal in Great Britain at what is said to be the "world price," which is defined as the London Metal Exchange official quotation. This quotation is on the basis that the duty is "for buyers' account," but any article on which duty is still to be calculated cannot (in an official market) have the same value as an article quoted "duty free," and any effort to secure price-equality between the two must therefore have a continuous deflationary influence on the price of the duty-free article.

### Zinc Die-Casting Alloys

IT was the discovery of the remarkable influence of aluminium on the behaviour of zinc-base die-casting alloys which raised these alloys to the status of engineering materials. Much important work in this connection is of American origin, but in Great Britain such alloys are now manufactured by National Alloys, Ltd., which is controlled by the Imperial Smelting Corporation. A recent article by A. Burkhardt ("Metallwirtschaft," August 31) deals with these alloys and reviews the influence of different components and impurities. It is pointed out that the first zinc die-casting alloys had rather poor properties, as they contained zinc of low purity, and corrosion and ageing phenomena gave rise

to marked deterioration of the mechanical properties and dimensional instability.

The introduction of purer zinc (0.05 per cent. lead) was the first step towards improvement, but still more important progress was made with the addition of magnesium, when the impurities detrimental to corrosion were rendered almost completely impotent. The transition to a more highly pure zinc (99.99 per cent.) gave slight but definite dimensional instability over a long period and a marked decrease in density, but the abolition of copper was the final stage in the improvement of these alloys.

All zinc die-casting alloys contain aluminium, for zinc exerts a strong disaffinity for iron and no element other than aluminium is known to diminish this disaffinity. Intercrystalline corrosion, however, is accelerated by the addition of aluminium, but is slowed down by copper. The ageing phenomena brought about by copper segregation, however, have such an unfavourable influence on the tenacity and dimensional stability of the aluminium-zinc alloys that any advantages gained by the inhibition of intercrystalline corrosion by copper are outweighed. The lead content is a determinative factor in the corrosion-chemical sense, all such alloys being liable to intercrystalline oxidation if they contain lead, and with the inevitable lead content of the virgin zinc it became important to find an addition which would compensate for the corrosion provocation of the lead. In this connection magnesium has proved to be of great service, the chief disadvantage being an increase in the hot shortness of the alloy. For a magnesium content above 0.05 per cent. the alloy is less easily injected into the mould, and the casting exhibits hot cracks at areas of transition from thick to thin walls. Of all other impurities, tin has the most detrimental effect, which is similar to that of lead but wider. Even a tin content of 0.001 per cent. is liable to give rise to intercrystalline corrosion.

### Iron-Aluminium Alloys

THE addition of varying quantities of aluminium to certain base metals has already been found to be of immense value. For instance, aluminium brass, which was developed by the British Non-Ferrous Metals Research Association and used principally for condenser tubes in marine service, owes its sea-water resistance to the formation of a very hard and self-forming film of alumina. In another direction there are nitriding steels which owe their high abrasion resistance to a film of aluminium nitride produced by treatment with ammonia.

The results of fundamental research into the physical

properties of iron-aluminium alloys were given in a paper which C. Sykes and J. W. Bampfyld read before the Iron and Steel Institute, at the Brussels meeting, in September. The alloys were made in electric furnaces using either high-grade fireclay crucibles or a furnace which was lined with magnesite, the high-grade Swedish iron being melted first, and the aluminium being added after the removal of any slag which was present. The carbon content in the final alloy was about 0.06 per cent. Melting losses varied with the composition of the alloy and also with the state of the iron before the additions were made, but were quite small with alloys of 10 per cent. aluminium and upwards. Production of ingots and sand castings was rather more difficult than in the case of ordinary steel, especially when the aluminium content was high. In sand castings a high pouring temperature and a good head sufficed to give a smooth finish to the casting.

Alloys containing up to 16 per cent. of aluminium could be readily forged and rolled hot. Those containing up to 5 per cent. were ductile, could be cold-worked and were similar in properties to pure iron. On the other hand, those containing from 5 to 16 per cent. were quite brittle in ingot form when cold and needed to be worked hot, but even then they required careful handling. That the aluminium content necessary to prevent oxidation increases with the temperature was found to be quite definite. For service at temperatures below 1,100° C., however, there is very little point in adopting alloys with an aluminium content in excess of 10 per cent., particularly as the ease of manufacture is increased by decreasing the aluminium content. At higher temperatures the maximum aluminium content possible with ability to fabricate, namely, 15 per cent., is desirable in order to allow a considerable life before breakdown occurs.

### Vanadium in Alloy Steels

THE influence of additions of vanadium to carbon steel, and to steels containing nickel and chromium, was described in a paper which H. H. Abram also presented at the same meeting of the Iron and Steel Institute. It was pointed out that the addition of vanadium causes an increase in the hardness of quenched carbon steels and steels of low alloy content, such as 0.65 per cent. chromium steels. It does not affect the maximum hardness of water-quenched alloy steels of higher alloy content, but it has a marked influence on the hardness of these steels when they are cooled at some slower rate or when they are tempered within a certain range of tempering temperature.

When steels containing vanadium are rapidly cooled from a high temperature and are then re-heated at 500° to 700° C., temper-hardening is induced to an extent which varies with the composition and previous treatment. This temper-hardening is shown by low-carbon and medium-carbon steels and, to varying degrees, by all the alloy steels which were examined, being most pronounced in the nickel and nickel-chromium steels containing about 2.6 per cent. of nickel and being favoured by cooling from a high initial temperature. In alloy steels containing vanadium, a high initial temperature does not increase the hardness of the quenched or the very slowly-cooled specimens, but it influences the hardness of specimens

cooled at intermediate rates. In some alloys, the air-cooled specimens are harder when cooled from a high initial temperature: in others, the hardening effect of high initial temperature is only observable when a slower rate of cooling is employed. The hardening is not due to persistence in the formation of martensite at these slow rates of cooling, but to the partial breakdown of a solid solution which is preserved in the super-saturated condition by faster rates of cooling. This solid solution is present in the more rapidly cooled (water-quenched or oil-hardened) material, and can be caused to precipitate its dissolved constituent by suitable re-heating treatments.

In order to obtain the full effect of temper-hardening it was not necessary to exceed 0.35 or 0.4 per cent. of vanadium in any of the alloys examined. A further increase in vanadium had no advantageous effect on the properties of the steel. In actual practice, less than 0.3 per cent. of vanadium is usually employed, but the extent to which the properties of commercial vanadium steels are affected by temper-hardening, and the conditions of initial temperature, rate of cooling and time and temperature of tempering which are required to reveal the effect, have not been investigated. It is clear, however, that temper-hardening may occur in steels with as little as 0.15 or 0.2 per cent. of vanadium.

### Selenium and Tellurium

WITHIN the last few years technical interest in selenium and tellurium has increased. The companies that hitherto found no market for these two elements are now working the dumps previously set aside as useless material. Apart from the large consumption of selenium in the manufacture of red glass, the more important application of selenium is in the new photo-electric cells. Consumption of tellurium has increased more than three-fold within the last few years, due to the discovery, on the part of U. C. Tainton, that small additions of tellurium to the zinc electrolyte facilitate the elimination of the very objectionable cobalt impurity. In addition, Professor F. C. Mathers, of the University of Indiana, has successfully demonstrated the comparative ease with which tellurium can be plated out from fluoride solutions. The deposit thus obtained is extremely resistant to corrosion, even in acid and in strongly basic atmospheres. Both selenium and tellurium can be extracted from the slimes which are produced at electrolytic copper refineries, but it has been said that the copper refineries of the United States and England could produce little more than 200 tons of selenium and 100 tons of tellurium annually.

### New Uses for Copper

THE development of new uses for copper also offers many opportunities. Up to the present, progress is chiefly due to newly discovered properties of the metal or its alloys, or merely the substitution of copper for some other metal which has hitherto applied itself to a particular industrial use. Examples of progress falling into the second category include the use of copper or brass water pipe, and the use of copper as a base for various types of plated goods. In the first category can be placed the recent introduction of very thin copper sheets, backed with tarred fabric for roofing purposes. The alloying of copper with beryllium to pro-

duce a material which may be precipitation-hardened by heat treatment at low temperature, is another direction in which progress has been made. This heat treatment alloy is suitable for springs and similar uses where resilience and resistance to fatigue are required. A small percentage of copper in steel permits the application of low temperature heat treatment to increase the hardness of the alloy without quenching, and should prove to become valuable where the size or shape do not allow quenching.

### Case-Hardening by Cyanide

VISITORS to the Machine Tool and Engineering Exhibition which was held in London, November 8-24, were invited to bring their own samples for case-hardening treatment at the stand occupied by Imperial Chemical Industries, where a small case-hardening furnace was turning out 100 lb. of work per hour, heat-treated or carburised to a depth of 0.005 inch.

The Cassel method of controlled case-hardening exemplified by this furnace differs from pack-hardening in that instead of employing a compound in the solid state as the means of introducing carbon it uses a case-hardening agent in the molten state. The bath consists of sodium cyanide and sodium carbonate, and the melt is kept at a temperature of 700°-950° C. Some idea

of capability of this method may be gained from the fact that with a bath of 50 per cent. cyanide strength at 950° C. a case depth of 60 thousandths of an inch can be conferred on any mild steel parts after six hours' treatment. The depth of "case" can be judged accurately according to the period of immersion and the temperature of the bath. The temperature itself can be accurately controlled by a thermo-couple which is immersed in the liquid alongside the work, and the actual temperature of the work is thus shown on the pyrometer. There is no decarbonisation, and sealing is also avoided, as oxidation of the parts during transfer from the furnace to the quenching tanks is obviated by a film of salt covering all surfaces.

This method of case-hardening ensures a tough core and perfect finish, with absolute uniformity in the finished work. The even heating which is obtained with a molten bath reduces warping and distortion to a minimum, but where distortion is absolutely unavoidable a new deep cementation compound, produced by the Cassel Cyanide Co., can be used in the ordinary case-hardening bath. The "case" depths thus obtained are deeper than those conferred by a cyanide bath, and have the advantage of being glass-hard for over half of their depth as compared with a third of their depth for metal treated in a cyanide bath.

## Modern Cast Irons in Chemical Engineering

### Marked Improvement in Resistance to Corrosion and Heat

ANY consideration of modern cast irons in chemical engineering should strictly be prefaced by a definition of cast iron and also by a definition of the chemical industry, and neither can be put into clearly-marked categories, said Mr. J. G. Pearce, M.Sc., F.Inst.P., Director of the British Cast Iron Research Association, at the outset of a paper on "Modern Cast Irons in Chemical Engineering," read at a meeting of the Chemical Engineering Group, which was held in London on November 9.

Metallurgically, an iron-carbon alloy is regarded as a cast iron when its carbon content is above about 1.8 per cent., and as a steel when the carbon is below. The presence of other elements, however, alters this figure, and some materials, such as the high-chromium cast irons, are true cast irons although their carbon content is very small. Cast irons usually contain free graphite, but this is not necessarily true of the white cast irons, and graphite is occasionally found in the steels. A convenient definition avoids reference to metallurgical considerations and considers cast irons as irons capable of being cast with the ordinary resources of an iron foundry.

#### Uniformity in Structure

Modern cast irons are much more uniform in structure than their predecessors. Iron will normally take up carbon to the eutectic value of 4.3 per cent. This figure, said Mr. Pearce, is lowered by the presence of other elements normally found in cast iron, notably silicon and phosphorus. Thus, an iron containing 2 per cent. silicon and 1 per cent. phosphorus will have a eutectic carbon value of 3.4 per cent. With the aid of silicon, which breaks down or graphitises the iron carbide which this carbon normally forms, the bulk of the carbide can be converted to iron and graphite, a process that is also facilitated by slow cooling. The founder uses silicon, suited to the section to be cast, to produce a grey and machinable casting from what would otherwise be white iron. The carbon remaining in combination is in the form of iron carbide (cementite), existing with iron (ferrite) as the finely-laminated duplex-structure pearlite.

An iron or steel is usually fully pearlitic with 0.9 per cent.

carbon in the combined form. Combined carbon in greater quantity exists as massive iron carbide or cementite, which is normally unmachinable, and arises, unless deliberately wanted, from too low silicon or too rapid cooling. Conversely, with too high silicon or too slow cooling, the pearlite carbide breaks down to graphite and iron (ferrite). Castings formerly tended to be mixed structures of graphite, pearlite, and ferrite. In recent years, however, by closer metallurgical control, a more uniform structure can be obtained, consisting of pearlite and graphite.

The most striking advances in the production of strong irons have come about by reduction of the size and quantity of graphite. To maintain a pearlitic structure, the graphite content can at present be best attained by using a low total carbon. The precise figure required is governed by the silicon content for a given section of casting. Several methods of achieving this are available, but essentially the principle is to cast an iron so that it would be white when poured and then to make it grey by a ladle addition of a graphitising material. The graphite produced in this way is smaller than that normally attained. One such process, patented by the International Nickel Co., Ltd., makes use of nickel and ferro-silicon as a ladle addition.

Broadly speaking, anything which contributes to the strength, soundness and homogeneity of cast iron also improves its resistance to corrosion and heat, but there are sufficient exceptions to this general rule to make it impossible to treat these properties in one group. Some measure of repetition is therefore unavoidable.

#### Resistance to Corrosion

All ferrous materials are liable to corrode, but discussion of this point often obscures the fact that cast iron normally offers excellent resistance compared with other ferrous materials not specifically manufactured to resist corrosion. In general, where cast iron is not at least as good, it offers better resistance than steel, as, for instance, to dilute and strong acids and alkalis.

Soundness and homogeneity of the casting are of great



importance in increasing resistance to both corrosive and thermal attack, as the graphite cavities present channels for the admission of the corrosive and heating media. The extremely fine form of graphite seems, at any rate, in some media, more liable to corrosion than flake graphite, although extremely coarse graphite is not recommended.

The influence of elements of composition cannot be simply stated, because a number of elements exert more than one effect. Thus, silicon softens the metal by virtue of its effect in breaking down cementite to ferrite and graphite, so that increasing silicon is accompanied usually by increasing graphite. Intrinsically, however, silicon hardens and embrittles the metal, as is seen most completely in the case of the high silicon irons. Silicon also appears to diminish corrosion-resistance to alkali, which needs a low carbon, low silicon, low phosphorus material, yet grey and free from carbides. Manganese and sulphur do not have a very great effect on corrosion of cast iron in the amounts normally present. The increased use of scrap has tended to raise the amount of sulphur normally present, and by the use of soda ash it has been found possible to reduce the amount of sulphur by, roughly, 50 per cent. Manganese and sulphur exist mainly as an inclusion of manganese sulphide. Phosphorus does not materially affect the corrosion-resistance towards acids, but the resistance is lowered in the presence of alkalis, and hence a hematite iron is preferred for caustic pots.

#### Addition of Nickel and Chromium

Marked improvement in corrosion-resistance has been found by using low-alloy additions to cast iron, especially nickel. Used under proper conditions of a balanced composition, the silicon being modified in ratios now well understood by metallurgists, the improvement due to nickel is doubtless brought about in part by increased soundness and homogeneity. Chromium does not of itself improve corrosion-resistance to  $H_2SO_4$ ; in fact, it makes it worse. Nickel and chromium together, however, can be employed in such proportions as to render unnecessary any alteration to the silicon content, and under these conditions there is no objection to the use of chromium. The metal is fully pearlitic.

The acid-resisting high silicon irons have been used for 30 years in the chemical industry. This material is extraordinarily resistant to many acids, alkalis and salts, being perhaps the best all-round acid-resisting ferrous material, but suffers from brittleness, and it is a very difficult material to cast sound. For these reasons many attempts have been made, especially in Germany and the United States, to improve it, but without marked success. The material, in fact, has not undergone substantial change. At the same time, improvements in foundry technique have been made, and the metal is now made more sound than formerly. It is well known that the resistance of the high silicon alloys increases slowly to about 12 per cent. silicon, when a sudden increase takes place and there does not appear to be any advantage in increasing this figure over about 16 per cent.

#### Niresist and Nicrosilal

The latest contribution to the corrosion problem is that of the nickel-bearing austenitic cast irons, of which two are commercially available, Niresist, developed by the International Nickel Co., Ltd., and Nicrosilal, developed and patented (British Patent 378,508) by the British Cast Iron Research Association. They are roughly 2 to 200 times as resistant as ordinary cast iron to corrosion, according to the medium, and compare in resistance with phosphor bronze. They are also considerably more resistant to erosion than ordinary cast irons. Niresist contains approximately 14 per cent. nickel, 6 per cent. chromium, and 5 per cent. copper. Nicrosilal is a development of the iron Silal referred to below and contains about 6 per cent. silicon, 18 per cent. nickel and 2 per cent. chromium. The carbon content of these alloys varies between 1.8 per cent. and 3 per cent.

All irons and steels are liable to oxidation or scaling at elevated temperatures. Cast irons containing no graphite, such as the white irons, behave like the steels, but grey irons, containing graphite, are liable, in addition to growth, to an actual increase in dimensions. The growth usually takes place in two stages: first, the iron carbide breaks down to

iron and graphite, which may involve a 2 per cent. increase in volume. Secondly, the iron itself oxidises, and this causes a still larger volume increase. During breakdown, oxidising gases penetrate the graphite cavities and oxidise the iron in the neighbourhood. The volume increase sets up a stress resulting sooner or later in a fine crack, which opens a further channel to attack. The process continues until the casting is honeycombed with cracks, which appear, for example, on the working face of an ingot mould or retort as crocodile markings.

Silicon in solution in cast iron intrinsically confers resistance to growth, and this fact was made use of by the B.C.I.R.A. in developing Silal heat-resisting cast iron (British Patent 323,076). This iron usually contains 4 per cent. to 6 per cent. silicon, and hence contains no carbide to break down. The graphite can be made in a very fine state of division, and the silicon actually makes the body of the metal more resistant to oxidation, and hence growth; the silicon also raises the critical point of the metal about that of the ordinary cast irons. All pearlitic and ferritic cast irons pass through a critical change at about  $760^\circ C.$ , at which graphite dissolves, followed by a contraction in volume. The metal then becomes austenitic. This change fixes an upper limit of temperature for the use of grey irons. By silicon additions the change can be raised to  $1,000^\circ C.$  Nickel, chromium and copper can be used to depress this temperature to atmospheric and hence irons austenitic in the cold suffer no change of this kind.

#### Points from the Discussion

Mr. A. HOARE said he had tested Nicrosilal iron for super-heater supports which had hitherto been made of austenitic steel. Nicrosilal iron, however, was very much cheaper, but unfortunately the Admiralty were not yet to be weened from the use of austenitic steel and still insisted on using the much more expensive material.

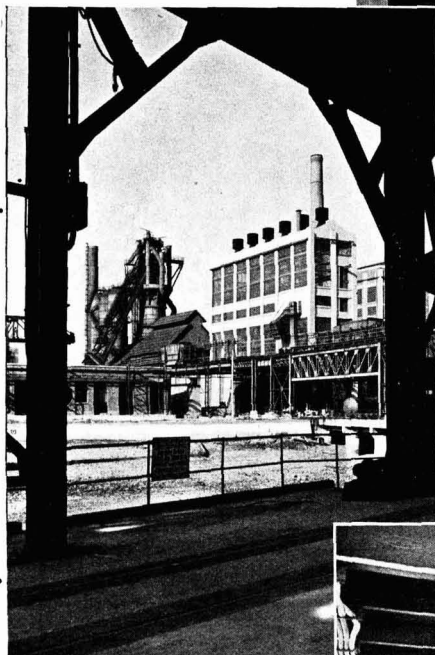
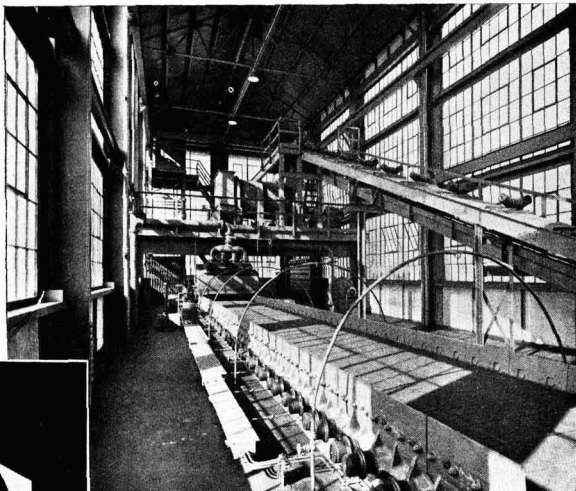
Dr. A. B. EVEREST said he did not think that Mr. Pearce had done justice to these new irons from the point of view of abrasion. There were many cases in which fluids were used containing solid particles which caused erosion of pumps, valves and pipes, and some of the new hard irons definitely gave the erosion resistance which was required. The author had recommended up to 12 per cent. of nickel for caustic pots and it would be interesting to know if Mr. Pearce had had experience of that sort of material, because he would not expect more than 5 or 6 per cent. to be really useful.

Mr. W. RUSSELL, speaking of corrosion testing, emphasised the absolute necessity of working with works solutions instead of synthetic solutions. Small quantities of impurities were often to be found in works solutions which played the devil with the results, and therefore it was necessary to use these solutions if troubles were to be avoided. Again, the loss of weight alone was not always a sufficient test, for it was known that there had been a great loss in strength in certain cases, and yet not a corresponding loss in weight. The effect of erosion as well as corrosion had been mentioned. In that connection he recalled some chemical plant which he knew would stand up to the particular acid quite well, but unfortunately in addition to the acid there were some mineral constituents which had an abrasive effect and the acid-resisting film was readily worn off, so that instead of getting the length of life that was expected the plant corroded away very much more rapidly.

#### Avoiding Fire Bar Troubles

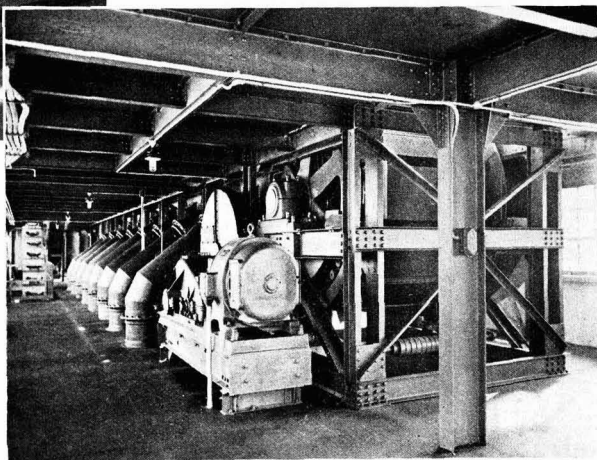
Mr. PEARCE, replying to the discussion, said it was to be regretted that the Admiralty insisted in using the more expensive material. Difficulties with fire bars were often due to certain British coals which had such a high calorific value that, especially now they were so frequently washed they tended to melt the surface of the metal, but that was a fact which must be put up with. The best plan was to use a common iron or unwashed coal. There were certain types of coal which carried an ash content which had a peculiar slagging effect in metal and in that way seemed to erode it readily. Nicrosilal cost something of the order of 1s. to 1s. 3d. per lb. in the finished form, but Niresist with slightly less nickel was definitely cheaper.

Right: The sintering plant consists of a horizontal, endless travelling chain of metal carriages or pallets, each having a floor which is formed of firebars. These pallets, which are 6 ft. wide, carry a 7 to 10 in. layer of a mixture of the finely divided ore and coke breeze having the correct proportion of moisture. At the commencement of travel, ignition is started by an overhead combustion chamber or ignition muffle, which is fired by blast furnace or coke oven gas. The sintering floor is 71 ft. long.



Above: A general view of the sintering plant taken from the jetty in the River Thames, showing the blast furnace on the left. The total power consumption is about 15 Kwh. per ton of sinter, and the total operating costs are less than 2s. per ton.

Below: The pallets travel slowly over a series of suction wind-boxes, so that air can be pulled through the layer of ore and coke breeze by a powerful fan connected to the trunking seen in this view. The burning of the coke breeze gives the necessary temperature for sintering (partial melting and agglutination) to take place. The sintered product is discharged over an inclined stationary screen, and any "smalls" are returned for re-treatment. The effective grate area provided for the sintering process is 310 sq. ft.



### Sintering at a Modern Blast Furnace Plant

THE latest principles of sintering iron ore smalls for the blast furnace are well represented in Great Britain by the "Dwight-Lloyd" plant which is installed at the Ford Motor Works, Dagenham. This plant deals with 700 tons of sintered material per 24 hours, the blast furnace having a capacity of 500 tons of pig iron per 24 hours.

## Correspondence

### Research on Electrodeposition of Metals

SIR,—Research work of national importance on the electrodeposition of metals is threatened with curtailment and we appeal to industry for co-operation in maintaining this essential work.

We have been privileged to serve on the committee appointed by the Department of Scientific and Industrial Research to direct the progress of researches on electrodeposition which have been carried out at the Research Department, Woolwich, the University of Sheffield and the Royal Aircraft Establishment, Farnborough, during the past eight years.

The work includes electrodeposition of nickel and chromium, the protection from corrosion of aluminium and its alloys by the electrodeposition of zinc, cadmium, etc., and fundamental questions, such as the occurrence of stress in deposits and the mechanism of adhesion. Many papers have been published and the work accomplished has substantially increased our knowledge of the theory and practice of electrodeposition and led to important advances. It is, however, by no means completed, for many important problems in electrodeposition are still unsolved. The steady continuation of research on a substantial scale is necessary to ensure that technical progress in the electroplating industries in this country does not lag behind developments abroad.

The cost of this work has been very considerable and has up to this time been borne almost exclusively by the Exchequer. The Department now take the view that research of this kind, carried out for the benefit of industry, should be maintained by the branches of industry concerned. Consequently, the work can continue only if it is adequately supported by industry. We should greatly regret its suspension or curtailment, since we believe it to have reached a

stage at which further important practical results may be expected. Closing down would entail the dispersal of experienced staff having valuable specialised knowledge of the subject.

It is now proposed that this work should be taken over entirely by the British Non-Ferrous Metals Research Association. In this way, Government support of the research would not cease but would be dependent on the funds forthcoming from industry. The scheme in view has the full approval of the Department of Scientific and Industrial Research.

The minimum expenditure necessary to continue this work is £1,000 per annum and it is hoped that sufficient funds will be provided for this purpose by those sections of the industry concerned with electrodeposition. The Association has made arrangements whereby the firms who subscribe to this work shall have preferential access to the results as they become available, together with other privileges of membership, and all those who are interested are asked to write to the Secretary, British Non-Ferrous Metals Research Association, Regnart Buildings, Euston Street, London, N.W.1, for further particulars.

We hope that this appeal will meet with a ready response and that the work which has begun so well will be continued.

W. R. BARCLAY,  
Consulting Metallurgist to the Mond  
Nickel Co., Ltd.

R. S. HUTTON,  
Goldsmiths Professor of Metallurgy, Cambridge.

H. MOORE,  
Director of the British Non-Ferrous  
Metals Research Association.

## Some Recent Metallurgical Patents

### Metals and Alloys

IN A process for producing metals and alloys very low in carbon, for example, chromium, manganese, and their iron and steel alloys, by the direct reduction of the ore with a silicon-containing reducing agent in a molten main metal both low in carbon, the charge used comprises ore, limestone, and reducing agent, which have been preliminarily heated at 1,000–1,100° C., in a strongly oxidising flame until the combustion of carbon is complete and the limestone is converted into caustic lime. The charge after grinding and thorough mixing is loaded in a layer over a continuously rotating heating platform. The treated charge is discharged into closed iron boxes for cooling and storage. If the combustion gases are to be circulated for further use, they are purified in respect of carbon dioxide, and sulphur, for example, by caustic potash. (See Specification No. 413,570 of G. Andersen.)

### Refining Copper

COPPER oxide and other oxidised impurities are removed from copper by violently intermixing the molten metal and a previously molten very fluid slag having a high dissolving power for the oxidised impurities, the reaction being completed in such a short time that no external heating is necessary. The slag may comprise silica, titanium oxide, alumina, lime, magnesia, alkali or alkaline earth salts, oxides of iron or other metals not reducible by copper, and may be melted down in a water jacketed furnace so as to produce an autoligning. After exhaustion, the slag may be regenerated for further use by reduction, for example by carbon. Violent intermixing is produced by pouring the copper in a thick jet on to the slag in a ladle, by mechanical agitation as by rocking a ladle containing the metal and slag, or by means of a gas. (See Specification No. 409,911, of Soc. D'Electro-Chimie, D'Electro-Metallurgie, et des Acieries Electriques D'Ugine.)

### Fluxes

A VEHICLE for use with fluxing salts such as zinc chloride, ammonium chloride or mixtures of such salts, particularly in the soldering of aluminium and aluminium alloys, comprises a mixture containing chlorinated diphenyl and chlorinated naphthalene. A solid, viscous, or liquid chlorinated diphenyl may be mixed with a solid, viscous, or liquid chlorinated naphthalene to give a solid, viscous, or liquid vehicle, the fluidity of which may be adjusted if desired by the addition of an organic solvent such as benzol, toluol, xylol, or other aromatic hydrocarbon, carbon tetrachloride, cyclo-hexanol, or a mineral oil. A liquid vehicle may be obtained by mixing together solid chlorinated diphenyl, solid chlorinated naphthalene, and the solid paradichlorobenzene, in certain specified proportions, the fluidity being adjusted if desired by the addition of an organic solvent. (See Specification No. 413,519 of Aluminium, Ltd.)

### Coating with Metals

AN adherent coating of a metal, an alloy, a metal oxide, or a metallic compound such as a glass, glaze, or enamel, is formed on the surface of a vitreous body such as glass or a vitrified body such as vitrified porcelain by projecting the metal, etc., e.g., by the Schoop process, on to the body, the body having previously been tempered by rapid cooling from a temperature adjacent its softening point and then re-heated, preferably to between 200 and 400° C. Alternatively the deposition of the coating may be effected during the cooling, after the body has been brought to a temperature below the lower limit of relaxation of the internal stresses. The coating of rough, polished, or moulded glass with metals such as silver, aluminium, zinc, copper, or brass is referred to. (See Specification No. 413,900 of Soc. Anon. des Manufactures des Glaces et Produits Chimiques de St.-Gobain, Chauny, et Cirey.)

# The Chemical Age

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