

# The Chemical Age

VOL. LXXVI No. 1954

22 December 1956

## TITANIUM PRODUCTION

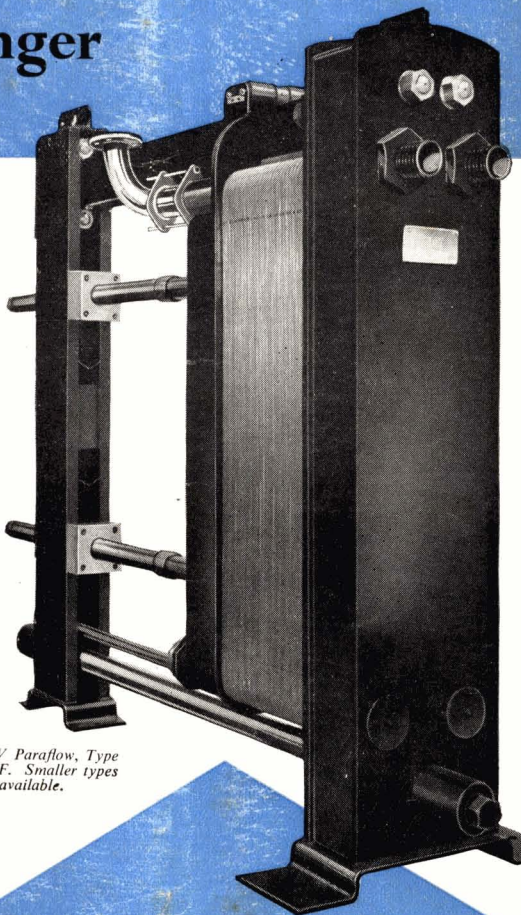
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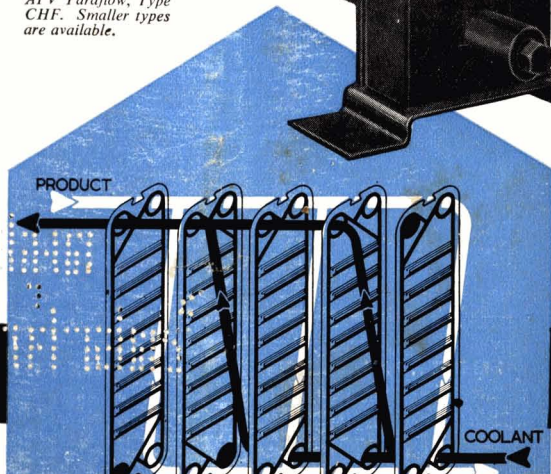
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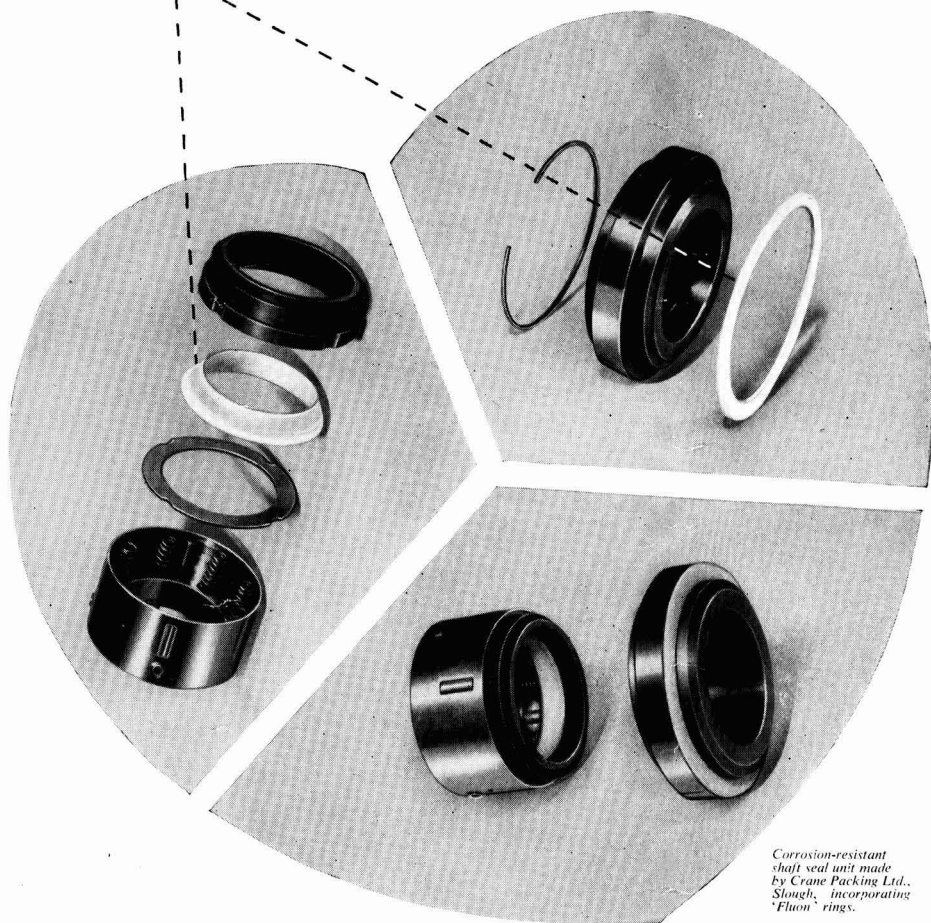
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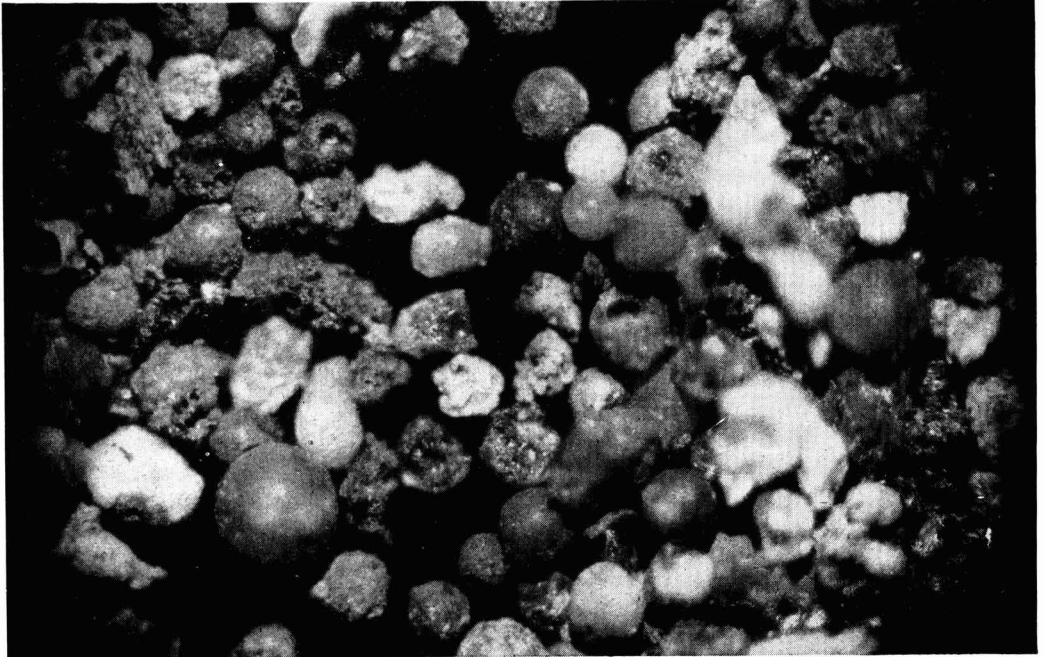
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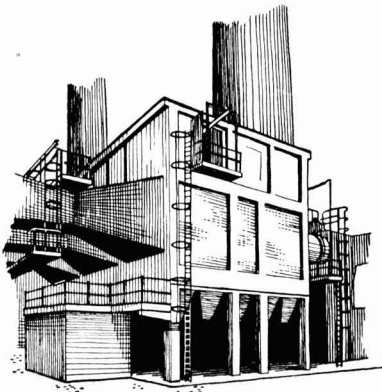


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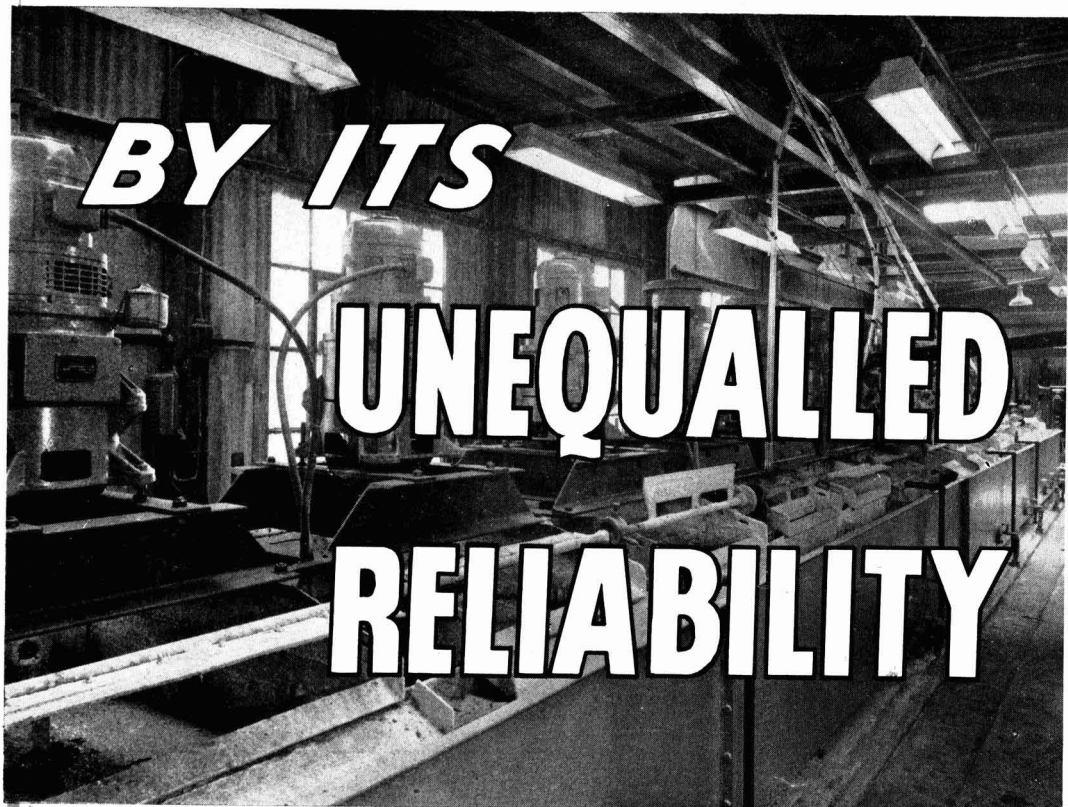
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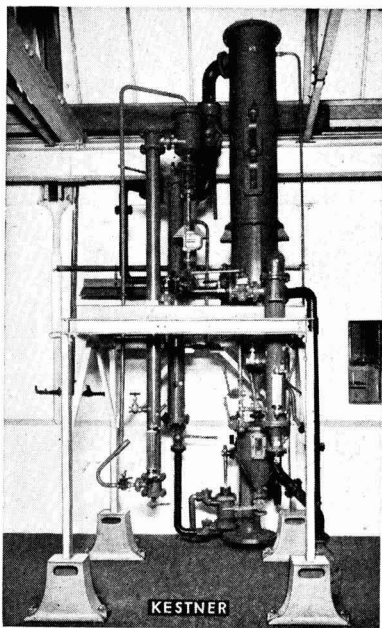
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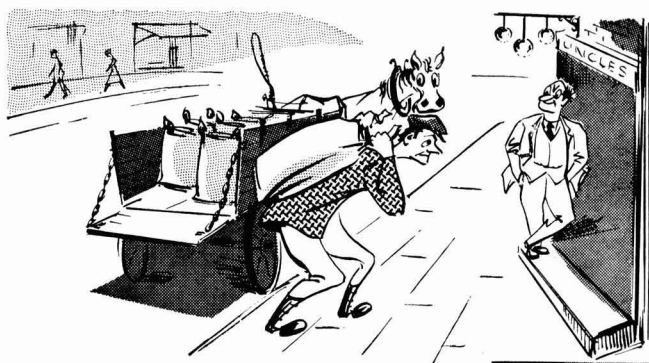
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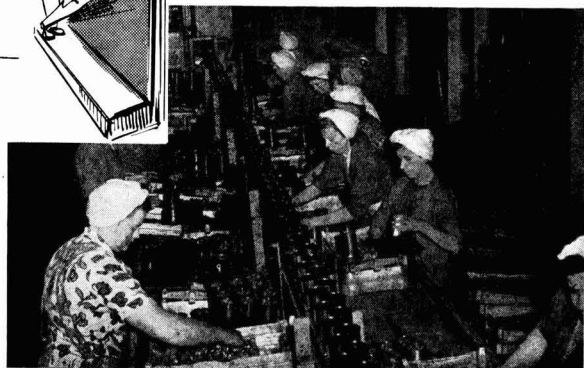
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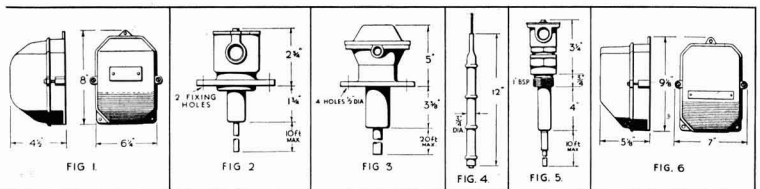
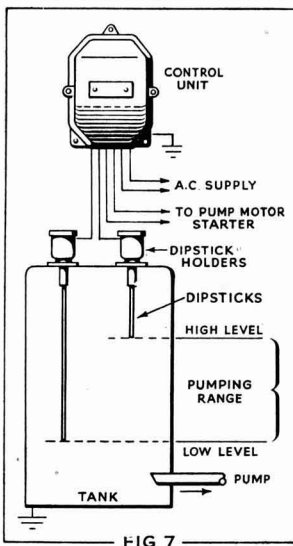


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## VIEWPOINT

# Sources of Power

IT HAS been estimated that by 1957 the world's energy demands will have increased three and a half times over those of 1953; in the year 2000 eight times as much energy will be needed. But it is impossible to increase production of energy to this extent from current resources. In Britain, coal output, once so abundant compared with the country's needs, is now insufficient. Coal is also in short supply in many other countries.

Although supplies of coal within the earth are considered to be plentiful, for a variety of reasons they cannot be mined economically. The time is likely to come, therefore, when coal can be used only for the manufacture of chemical products. In the UK there are adequate reserves of poor quality coal and underground gasification of these might provide a source of power and chemicals. According to the National Coal Board a pilot scheme is expected to be operating by the end of 1958.

At least one company plans to develop methods of making synthesis gas for conversion into a number of products including oil fuels.

These remarks also apply to oil. At the present rate of usage it is doubtful whether oil will fill the gap for very many years, and recent events in the Middle East have shown the dangers of relying too rigidly on oil supplies.

One source of natural power which can be exploited still further is water. Europe's hydro-electric capacity is said to be almost fully exploited, yet hydro-electric power in Britain 15 or 20 years hence is estimated at just over twice present capacity.

Nuclear energy appears to be the most hopeful source of power, details of the latest schemes for the UK are given on page 474 of this issue.

From experience gained at Calder Hall, output of atomic power stations can now be increased considerably, and the whole nuclear power programme is significantly greater in size. By 1965, at least 15 stations will be built with a combined generating capacity of at least 4,000 megawatts.

Atomic power stations are under way in many countries. However, sources of power other than atomic are being examined. During the past three or four years there has been a marked interest in the possibilities of harnessing solar energy for industrial use. In 1954, an Association of Applied Solar Energy was formed in the US to sponsor this technology, and a year ago, a world symposium on solar energy was held in the US.

Many papers read at this symposium gave details of the engineering aspects of solar machines, solar stills, heat pumps, water heaters, high temperature furnaces and electricity from the sun.

Concentration of solar energy is possible with the high temperature furnace and as a source of power can compete with present electrical rates in some areas if development work is undertaken to reduce costs.

It is reported that scientists in Russia are endeavouring to exploit energy from the sun on a large scale.

Russia is said to be planning a large generator which, when completed in two or three years' time, may have a capacity of 1,200 kilowatts.

On the river Rance, in France, engineers are building the first tidal power plant to operate on an economical level. The motion of the tides will be utilised to provide, it is estimated, 342,000 kilowatts annually. Should this project be successful, a larger plant will be built. This, it is claimed, will increase France's electricity production by one-third.

Another French project now being carried out at Abidjan on the African Ivory Coast utilises the difference in temperature between water on the surface of the ocean and water from the depths—in this area a difference of 75°F. A turbine operating generator is placed over the steam current established between a vacuum evaporator in which the surface water boils spontaneously, and a heat exchanger in which steam condenses when entering in contact with the cold water brought up from the depths.

When completed this station will have a capacity of 3,500 watts.

*The staff of The Chemical Age wish all readers a very happy  
Christmas and prosperity in the New Year*



## INSECTICIDE SYNERGIST

### New US Process Developed

DEVELOPMENT of a process for manufacturing a new and superior synergist, Sesoxane, for pyrethrins and related insecticides has been announced by Shulton's Fine Chemicals Division, of Clifton, New Jersey, US. Sesoxane-pyrethrin formulations are stated to be of special interest to formulators and packagers of insecticides where a safer product is required for use on grains, fruits, and in household and animal sprays.

Sesoxane was discovered by Dr. Morton Beroza of the US Department of Agriculture in Beltsville, Maryland, and reported by him as the 2-(2-ethoxyethoxy) ethyl 3,4-methylenedioxyphenyl acetal of acetaldehyde. The chemical name, 2-(3,4-methylenedioxyphenyl)-3,6,9-trioxyundecane, has been adopted.

Tests have shown this chemical to be superior in synergistic activity to other commercial pyrethrin synergists and, because of its effectiveness, it is expected to cost less to use than other available synergist formulations for equal knockdown and kill. High kill can be obtained without the use of DDT or other toxic insecticides. Numerous tests have already been published by the US Department of Agriculture

showing the effectiveness of Sesoxane against flies and mosquitoes and its interesting possibilities as an effective insecticide synergist for cockroaches and other insects. A comprehensive report describing the effectiveness of Sesoxane was presented by Mr. J. H. Fales of the Entomology Research Branch, Agriculture Research Service, US Department of Agriculture, Beltsville, Maryland, at the 43rd annual meeting of the Chemical Specialties Manufacturers' Association.

Because of its solubility in kerosene, Freon 11, Freon 12 and other solvents, Sesoxane is easy to formulate in conventional equipment. It has a faint, pleasant odour and the low acute oral toxicity ( $LD_{50}$ ) of 2,000 mg. per kilogram in rats. Complete toxicological studies are in progress.

Basic chemical required for the manufacture of Sesoxane is piperonal (heliotropine). When the new synergist was first announced by the US Department of Agriculture, Shulton recognised in it an opportunity to expand its production of piperonal toward the manufacturing of Sesoxane and a superior and continuous process for manufacturing piperonal has been developed by the company.

## RECONSTRUCTION COMPLETED

RECONSTRUCTION has been completed of the sulphuric acid plant at the Flixborough, near Scunthorpe, Lincs, works of Nitrogen Fertilisers Ltd., which produces ammonium sulphate for use in the fertiliser industry. This sulphate is manufactured by the direct neutralisation of ammonia with sulphuric acid.

### New Roaster

A new roaster, of a unique rotary design developed by the company, has been installed in the sulphuric acid plant and will burn impure sulphur which results as the filter cake from the bright sulphur purification process.

A complete new converter unit has been supplied and erected by The Power-Gas Corporation Ltd., of Stockton-on-Tees, who are exclusive licensees in this country for sulphuric acid plant designed by Chemiebau Dr. A. Zieren GmbH.

A feature of the erection programme was the necessity to keep the existing plant in operation as long as possible so as to avoid a serious interruption in acid production. This was accomplished by erecting a new single shell 140-ton-monohydrate-per-day converter in the space occupied by one

of the two old 60-ton-per-day units.

The reconstructed plant is now operating and the converter unit is reported to have comfortably exceeded the guarantee of 140 tons output at 98 per cent conversion efficiency.

### New Rubber Chemical

A NEW RUBBER CHEMICAL has been introduced by Pennsalt Chemicals of Canada, Hamilton, Ontario. Diisopropyl benzothiazyl-2-sulphenamide (Dipac) is a delayed action accelerator formulated for modern high temperature processing where maximum freedom from scorch is essential.

### Change of Address

MOST departments at the head office of the Mullard organisation are now at Mullard House, Torrington Place, London WC1 (telephone: LANGham 6633). The remaining departments will be moving in shortly. All correspondence for all departments should be sent to the new address.

## Tetraethyl Lead

### New Process Discovered in US

DISCOVERY of a new process for making tetraethyl lead has been announced in the US by the Ethyl Corporation, a jointly owned subsidiary of the Standard Oil Co. (New Jersey) and the General Motors Corporation.

The process, described by Mr. E. L. Shea, chairman, as radically different from any used hitherto, involves a reaction between a metal alkyl and a lead compound.

While the development work was still in early stages at the company's research laboratories, the process showed considerable promise, said Mr. Shea. It eliminated certain intermediate materials and steps involved in present manufacturing operations and appeared superior to other processes known by the company. He added that further details of the process would be reported soon.

## Shale Oil Bid Fails

THE Minister of Fuel and Power, Mr. Aubrey Jones, has rejected an appeal by the Scottish Unionists Members' Committee that further aid should be given to the Scottish shale oil industry. In view of the uncertain future for the industry and decline in coal mining in the shale oil districts, the Government is, however, giving careful attention to the need for establishing alternative industries in the districts.

Mr. Jones points out in a letter to the committee that the preferential rate of duty on shale oil—half that on ordinary oil—cost the Exchequer about £1 million a year in lost revenue and represented about £6 a week for every person employed in the industry. The amount of oil produced represented only 0.25 per cent of the nation's supplies, and was so small a proportion as to rule out large-scale or expensive action to help the industry.

### Film on Locusts

THE RUTHLESS ONE, a new addition to the series of Shell films on insect pests, is designed to bring the desert locust problem to public notice. After showing the size and nature of the problem and studying the locust in all its stages, the film shows how information obtained from many countries is mapped and analysed by the Anti-Locust Research Centre in London, and how probable movements can be forecast so that the countries affected can be warned to prepare counter-measures in time.

# NOTE & COMMENT

## STAR ITEM

AS WE HAVE frequently criticised the industry for its publicity methods, it is only fitting that we should give praise when it is due. A recent booklet issued by Albright and Wilson and its associated company, Midland Silicones, on careers for graduates in both firms, is excellent. Had the two firms sought to produce a relatively compact booklet to tell the public about themselves and their products, they could hardly have done better. At the same time, though the booklet has only 28 pages including plenty of illustrations, the aim of rousing the interest of youthful intelligence and ambition has not been lost.

Why does this little publication read so pleasingly and convincingly? One answer is that it is not a piece of window-dressed propaganda. A good story exists and is told. The other is that this sound basic material has not been mishandled by over-dramatisation. It has been presented with a quiet and direct simplicity, and coloured diagrammatic or photographic illustrations have been used to provide the 'high spots.' Particularly attractive are the diagrams showing the main uses of phosphorus compounds and silicones, and the ordnance survey map reproductions showing the areas in which the two firms' principal works and offices are placed.

Perhaps there is a contemporary 'novelty' appeal in the expanding uses of phosphorus compounds and silicones, but the chemical industry as a whole has many background stories to tell which are as good and which lend themselves no less naturally to first-class presentation methods. Nevertheless, we still see far too little of this sort of prestige literature. A limited number of the very large chemical companies know the value of 'brochure publicity' and much of their output reaches an admirable standard. But beyond that?—well, unless many of the medium-sized chemical firms conceal their publicity from us, we do not see much that is likely to make a memorable impression. On this point, of course, we are not merely discussing booklet matter aimed at staff recruitment, but booklets having any desirable objective.

Sometimes it is suggested that the industry should collectively impress upon the public its place (and pride of place) in the national economy; the American industry has certainly become more and more active in this direction. But the collective method has two weaknesses—its very generality stands in the way of effective emphasis, and as a rule projects that are run and funded by many interests lose much of their initial vigour along the wayside of committee discussions. The best way for the chemical industry to obtain better public understanding is for all reputable firms to tell their own stories simply and attractively.

## A CANADIAN MARKET

SO MUCH has been said about Canada's industrial expansion in the past decade that it tends to be forgotten that she is still a great agricultural country. At present the value of Canada's farm output is about a tenth of that of the US. Canada can produce much of her fertiliser requirements; indeed, she is an exporter of certain fertilisers. However, her home production of pesticides is very small, much below requirements—and there is every sign that demand is rapidly rising. It might be too readily assumed that these deficiencies can be remedied by purchases from the US. America herself has a large pesticide industry and her closeness to Canada gives her the advantage that is all too familiar in so many classes of Canadian import trade. Nevertheless, Europe is selling Canada pesticide materials, and Canada's readiness to buy from Europe was recently commented on in a US publication (*Journal of Agricultural & Food Chemistry*, 1956, 4, 905.)

Pesticide sales (including weedkillers) in Canada for 1955 totalled \$22.8 million. But it has been predicted that sales will double by 1960 or 1961. In the US, the British-originated selective weedkiller for cereals and grass, MCPA, is much less popular than 2,4-D, but this preference is not nearly as pronounced in Canada. Use of MCPA is rising and this is being sold to Canada by the UK. Germany is selling lindane or gamma-BHC to Canada despite the large US production of this insecticide. That an expanding dollar-earning market is there to be won cannot be disputed.

## DEVELOPMENT OPPORTUNITIES

A NUMBER of the major US producers have associated Canadian companies—some are little more than selling branches of the US parent firms, others formulate for Canadian use basic materials sent from America. This set-up might seem difficult to compete with, but it has weaknesses as well as strengths. One weakness is that private research—a most vital influence in this field—is being carried out on a low scale in Canada. In most cases, research is carried out by parent US companies and results are passed on. This stands out in contrast to the high level of Canadian government research on pesticides. How far product-usage research is inadequate must be a matter of speculation, but pesticide effectiveness is often greatly influenced by climatic conditions. Field research under Canadian conditions cannot be imported. There would seem to be development opportunities here well worth British examination—opportunities that should lead to the steady building-up of export sales.

# MEETING POWER DEMANDS

## Contracts Awarded for Nuclear Generating Stations

CONTRACTS for the first three UK nuclear power stations to be built under commercial tender have been awarded by the Central Electricity Authority and the South of Scotland Electricity Board. Negotiations for a fourth nuclear power station, in Somerset, are understood to be taking place. These stations are part of the Government's long-term programme visualising 12 or more power stations before 1965.

The largest of these power stations, which will have a generating capacity of 360 megawatts, is to be built by the General Electric Co. Ltd. for the South of Scotland Electricity Board. It is claimed that this will be the largest atomic power station in the world. Negotiations are taking place for it to be built at Hunterston, about 30 miles from Glasgow.

A 300 megawatt power station is to be built at Bradwell, Essex, by the Nuclear Power Plant Co. At this plant, Head Wrightson & Co. Ltd., a member of the Nuclear Power Co., will undertake the design, supply and construction of the heat exchangers, the installation of the graphite around the reactors and the supply of shield cooling, blowdown and evacuating systems.

### Third Station

The third nuclear power station, which will have a generating capacity of 275 megawatts, will be built at Berkeley, Glos, by the AEI-John Thompson Nuclear Energy Co., in collaboration with Balfour Beatty & Co. and John Laing & Son.

The GEC power station has been designed by the GEC-Simon-Carves Atomic Energy Group. The GEC as main contractor, will be completely responsible for the whole project.

Power will be derived from the heat generated by a controlled nuclear fission chain reaction in two nuclear reactors. The principle of operation will be similar to that of the Calder Hall reactors in that the natural uranium fuel will be contained within a pile of graphite moderator material and the heat extracted by circulating carbon dioxide gas under pressure.

Generating capacity of the station will be 360,000 kW and the total output of electrical power is guaranteed to be not less than 300,000 kW, rising to 320,000 kW, all of which will be fed into the national grid. The station has been designed to operate on base load, so that every day between five and six million units of electricity will be sent out to consumers. The saving in coal from this one station will

amount to at least one million tons a year.

Each reactor with its eight steam raising units will weigh about 70,000 tons, the entire structures being supported on thick concrete rafts.

Associated with GEC and Simon-Carves Ltd. in this enterprise are The Motherwell Bridge & Engineering Co. Ltd., who will fabricate the pressure vessels, and John Mowlem (Scotland) Ltd., who will be the civil contractor.

The cylindrical core of each reactor, 28 ft. high and 50 ft. dia., is built up of 2,000 tons of graphite blocks containing vertical channels for the fuel elements and control rods.

**It is estimated that the saving in coal from the South of Scotland nuclear power station will amount to at least one million tons a year. On this basis a total of 12 million tons of coal per annum should be saved eight years hence. The Government's programme visualised 12 or more nuclear power stations before 1965 for an expenditure of about £720 million.**

Each of the 3,288 fuel channels contains 10 individual fuel elements stacked in a column one on top of the other. The element consists of a bar of natural uranium metal sealed in a magnesium alloy can which is finned to assist heat extraction. The total charge of uranium in the two reactors is 500 tons.

The reactor core rests on a grid made of steel plates arranged in 'egg-box' fashion. Surrounding the sides and top of the core is an inner steel shell made of relatively thin steel plate.

A 70 ft. dia. spherical pressure

vessel, welded from 3 in. thick steel plate and weighing nearly 1,000 tons, completely encloses the core and inner shell. The double-shell construction enables the best materials to be used for each purpose—a temperature resistant steel for the hotter, inner shell, and a tough, non-brittle steel for the spherical vessel which has to withstand the full working gas pressure.

Protection from nuclear radiation is provided by a 9 ft.-10 ft. thick concrete biological shield surrounding the core and forming one face of the 180 ft. high reactor building.

### Charge & Discharge

Charge and discharge of the fuel elements in a channel, 10 at a time, is accomplished by a single remotely-controlled machine situated in a shielded chamber beneath the reactor. The charge/discharge machine is designed so that the removal of spent or damaged fuel elements and the charging of fresh fuel can be carried out while the reactor is on load and under pressure. The machine is controlled from a room in the reactor building where the sequence of operations is viewed on close-circuit television screens.

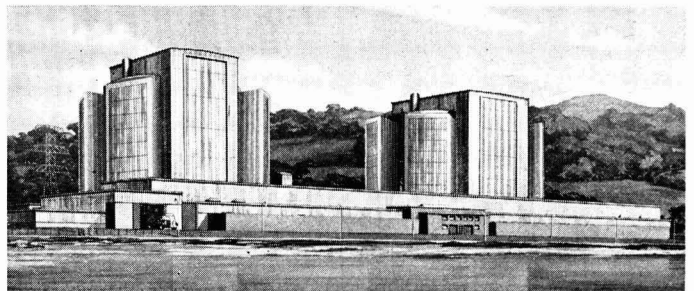
Heat is extracted from the nuclear reactor by a coolant gas, carbon dioxide, circulated throughout the system under a pressure of 150 lb. p.s.i. Gas passes upwards through the fuel channels in the reactor core, becomes heated, and is led away to the steam raising units where it gives up its heat to water with the formation of steam.

### Fuel Elements

Steam from the steam raising units is piped over a bridge to the 700 ft. long turbine hall containing six 60,000 kW dual pressure turbo-generator sets.

Between the two reactor buildings is an 18 ft. deep cooling pond in which the irradiated or spent fuel elements are allowed to 'cool-off' radioactively before being re-processed.

*An impression of the complete nuclear power station to be built by the GEC-Simon-Carves Atomic Energy Group for the South of Scotland Electricity Board. Negotiations for a site at Hunterston, about 30 miles from Glasgow, are taking place*





# Production of Titanium Metal

## RAW MATERIALS & METHODS REVIEWED

COMMERCIAL titanium sponge is at present manufactured by reduction of titanium tetrachloride. Other procedures, such as the electrolytic process or dissociation of halogen salts, are being investigated.

The important ores of titanium are rutile, which is a sand concentrate usually containing about 95 per cent of titanium dioxide, and various ilmenites which contain from 35 to 60 per cent of titanium dioxide. Rutile is essentially a naturally occurring form of relatively pure titanium dioxide, while the ilmenites are iron titanates and are given the generic composition of  $\text{FeO}\cdot\text{TiO}_2$ . Table I shows the analysis of rutile, a high grade Florida ilmenite, and a lower grade Canadian ilmenite from the Baie St. Paul region of Quebec.

TABLE I		
Analysis of Ilmenite		
	Florida ilmenite	Canadian ilmenite
TiO <sub>2</sub>	59.14 (Ti 39.5 per cent)	41.08 (Ti 25.1 per cent)
FeO	33.44 (Fe 25.9 per cent)	47.74 (Fe 37.2 per cent)
SiO <sub>2</sub>	0.16	2.23
CaO + MgO	0.51	4.26
Al <sub>2</sub> O <sub>3</sub>	0.50	1.58
	93.75 per cent	96.89 per cent
Analysis of Rutile		
TiO <sub>2</sub>	97.18 per cent	
FeO	0.37 per cent	
SiO <sub>2</sub>	0.50 per cent	
	98.05 per cent	

It will be noticed that the iron content is expressed as ferrous oxide, FeO, in Table I. This appears to be a traditional manner of reporting iron in the case of the several titanium ores. However, it is known that a considerable proportion of the total iron is present as ferric oxide,  $\text{Fe}_2\text{O}_3$ , in ilmenite and a large part of the difference between the analytical totals shown and 100 per cent is accounted for by the higher oxygen content of the ferric oxide.

### Desirability of Rutile

Of the ores of titanium, rutile is the most desirable from a chemical standpoint as it may readily be chlorinated to titanium tetrachloride with the production of only a minimum of undesirable by-products. This chlorination is done by contacting a mixture of rutile and carbon with chlorine at temperatures of 800-1,000°C.

However, two factors concerning rutile are in its disfavour and will undoubtedly make it a poor choice in the rapidly expanding titanium industry. These are its relative scarcity and its high price. A \$200 per ton rutile at 57 per cent titanium is equivalent to approxi-

mately 17.5 cents per lb. of contained titanium. It is necessary, therefore, to seek sources other than rutile for titanium, and ilmenite is a logical choice.

The production of high-grade ilmenite such as shown in Table I has been an established enterprise in the United States for a number of years and the technology of its mining and concentration is well-known, over half a million tons being produced annually in the United States. At a price of \$20 per ton, an ilmenite containing 40 per cent titanium would give a cost of about 2.5 cents per lb. of contained titanium compared with 17.5 cents for titanium in rutile.

### Direct Chlorination

Direct chlorination of ilmenite results in a large production of ferric chloride which adds substantially to the cost of chlorination and presents a difficult disposal problem. To prevent this it is necessary that the iron be separated from the titanium.

Ilmenite can be partially reduced in the arc furnace and a liquid separation of the iron and titania slag accomplished. However, in order to obtain a tappable slag, it is necessary to add fluxes, including lime, which overcome some of the refractoriness of the titania slag. The titanium now resides in a slag which still contains considerable iron, lime, magnesia, alumina and silica. For this reason, titania slag so produced—containing 72 per cent to 75 per cent equivalent titanium oxide—has part of the disadvantage of ilmenite.

The US Bureau of Mines and others have shown that it is possible to increase the equivalent titanium dioxide content to as much as 86 per cent, but there is an apparent limitation in making higher grade material due to the refractory nature of the titanium suboxides which makes tapping difficult. Particularly undesirable in a titanium raw material for tetrachloride manufacture are the oxides of calcium, magnesium and manganese which form fusible but non-volatile chlorides that add a burden to fluid bed techniques in chlorination.

A paper entitled 'A rutile substitute from ilmenite for the production of titanium metal' describes a process for the production of a rutile substitute for titanium tetrachloride manufacture in which disadvantages are minimised; it was given recently by J. H. Brennan, chief metallurgist, and J. G. Farmer, metallurgical engineer, both of the Electro Metallurgical Co., a division of Union Carbide & Carbon Corp., at a meeting of the Electrochemical Society.

Ilmenite is partially reduced by carbon in a resistance furnace of the Acheson type to yield a titanium oxycarbide from which iron is readily separated magnetically. The process has been found applicable

## Production of Titanium Metal

to Canadian ilmenites containing as little as 40 per cent titanium dioxide as well as to foreign and domestic ores containing upwards of 60 per cent titanium dioxide.

Conventional Acheson type furnaces, such as are used for silicon carbide and graphite manufacture, have been employed. Table II shows a typical charge for a 30 ft. long furnace using Florida ilmenite.

TABLE II

Ilmenite .. .. .	77,000 lb.
Petroleum coke .. .. .	13,300 lb.
Sawdust .. .. .	6,150 lb.
Water .. .. .	3,850 lb.

100,300 lb.

Sawdust and water are included to improve permeability for free evolution of gas. A central core of graphite or carbon is used to provide the initial path for current. As the run progresses, the solid product of the reaction becomes the conductor and then there is truly a direct resistance furnace. In a typical run, carbon monoxide flames make their appearance about 30 minutes after the initial application of power and burn freely over the top of the furnace charge. There is a steady settling of the charge during the entire period of the operation.

### Cooling the Furnace

Upon completion of the power cycle, the furnace is allowed to cool for about four days and then an envelope of unreacted mixture is removed from the top and sides. The product exists as a dense core surrounded by a small zone of sintered material. A thin layer of metallic iron lies under the core and this layer is largest, as would be expected, when low grade ilmenite (i.e. 40 per cent titanium dioxide) is used. The dense core consists of the titanium oxycarbide product in which metallic iron is dispersed in small globules, many of which could be distinguished macroscopically on a ground section. Power consumption usually averages between 2 and 2.5 kWh per lb. of dense product.

Crushing to 48 mesh and subsequent magnetic separation effectively removes the iron. An analysis of the magnetically separated titanium oxycarbide produced from Florida ilmenite is given in Table III.

TABLE III

Titanium .. .. .	68.08 per cent
Iron .. .. .	1.96 "
Carbon .. .. .	9.34 "
Silicon .. .. .	0.04 "
Ca+Mg .. .. .	0.43 "
Aluminium .. .. .	0.50 "
Vanadium .. .. .	0.09 "
Chromium .. .. .	0.27 "
Zirconium .. .. .	0.08 "
Manganese .. .. .	0.70 "
Phosphorus .. .. .	0.008 "
Nitrogen .. .. .	0.42 "
81.92 per cent	
Oxygen (by difference) .. .. .	18.08 "
100.00 per cent	
Ti/Fe ratio .. .. .	34.7

The ore has thus been upgraded from a 39.5 per cent titanium content in ilmenite to a 68 per cent titanium level in the non-magnetic oxycarbide. In comparison

it is seen that rutile contains approximately 58 per cent titanium, showing that the product represents a substantial improvement in titanium content not only over ilmenite but also over rutile. The titanium to iron ratio is improved from 1.5 in ilmenite to 34.7, while impurities such as lime, magnesia and manganese, are minimised in effect because of the large increase in the titanium content of the beneficiate.

While the magnetically-separated product is generally characterised as titanium oxycarbide, X-ray examination shows TiC, Ti<sub>2</sub>O<sub>3</sub> and small amounts of FeO.2TiO<sub>2</sub> and FeO.TiO<sub>2</sub> types. The titanium carbide has a lattice parameter of 4.30 Å compared with a normal of 4.32 Å for pure titanium carbide. This indicates the probability of oxygen in solution in the carbide.

The product has been chlorinated on a large scale for the production of titanium tetrachloride. The threshold temperature for chlorination to start is approximately 200°C lower for this oxycarbide than it is for the rutile-carbon mixtures in use today. Moreover, the higher titanium content of the oxycarbide—68 per cent against 57 per cent for rutile—increases the production rate of the chlorinator.

Sodium reduction of this titanium tetrachloride, after copper purification and distillation, has yielded titanium metal which is the equivalent in quality to concurrently produced metal using rutile as the source of titanium tetrachloride.

Thus, an acceptable substitute for rutile in the production of titanium metal can be made from ilmenite by partial reduction in an Acheson type furnace and magnetic separation of the reduced product. Commercial interest relates to the differential in price between rutile and ilmenite at any given time and the probability that sufficient rutile may not be available to support a rapidly expanding titanium metal industry. This development may well eventually constitute a stabilising influence on the market price of rutile, and, more important, it will make the US self-sufficient in the matter of domestic titanium ore supplies. A semi-commercial scale plant has been designed and construction will start shortly.

## Nickel Alloys

*Wiggin Nickel Alloys No. 42* contains an article dealing with the welding of Nimoply 75, a composite metal sandwich comprising Nimonic 75 bonded on both sides of a copper inter-layer. For some years the nickel-chromium alloy, Nimonic 75, has been used for combustion chamber linings. The new material has been developed to meet the need for a material with the high-temperature characteristics of Nimonic 75 but with improved thermal conductivity. Other articles in this issue describe the Inconel components of gas carburising furnaces in the Ford Motor Co.'s works, the use of Nimonic DS for flare stack tips, uses of Monel in pickling plant and the development of a special tungsten nickel for cathode sleeves of valves. *Wiggin Nickel Alloys* is the technical journal of Henry Wiggin & Co. Ltd., and free copies are available from the company's publications department at Thames House, Millbank, London SW1.

## FIRST SYNTHETIC RUBBER PLANT IN UK

### Dunlop Development at Birmingham

FOR THE first time in the United Kingdom, experimental production of synthetic rubber on an appreciable scale is shortly to begin at the Dunlop Rubber Co.'s £600,000 new plant near Birmingham. Output is expected to be about 1,500 tons a year.

For many years the company has been carrying out research in the fundamental science and technique of synthetic rubbers. Progress has been in a series of steps from the laboratory bench to pilot plant stage.

Main objects of the plant are. Production of general purpose rubber for tyres, and of new synthetic rubbers. It

**Said a spokesman of the Dunlop Rubber Co., 'Our venture is the advance guard of synthetic rubber production in Britain, the latest link in the chain of events begun in Birmingham in 1884 when Sir William Tilden made the first synthetic rubber in his laboratory.'**

has been designed so as to be flexible in operation and will thus be able to produce the wide range of synthetic rubbers likely to be developed in the future.

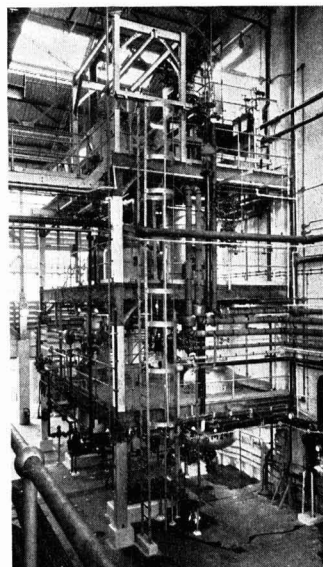
Ordinary general purpose synthetic rubber is made from the monomers butadiene and styrene. These are first of all emulsified in a dilute soap solution. Modifiers etc. are then added, which cause the small globules of liquid to polymerise into solid particles of rubber. The latex is then coagulated by adding salt and sulphuric acid and the curds of synthetic rubber are washed and dried.

Basic raw materials of synthetic rubber—the monomers butadiene and styrene—are supplied from the oil refineries at Fawley and elsewhere. These are stored in the tank farm at Fort Dunlop and drawn as required into the pre-treatment area for treatment by alkali or distillation to remove the added polymerisation inhibitors, and are then fed to the reactors. These are in a building covering about 11,000 sq. ft.

Beyond the reactors is the solution make-up area, where the emulsifiers are prepared. The liquids are pumped in metered quantities into the reactors; if cooling is necessary they pass through a heat exchanger on their way to the reactor. The reactors are of steel, glass lined and water-jacketed. Polymerisation is carried out with constant stirring; a range of controlled temperatures suitable for the production of various types of rubber can be obtained—low temperatures are achieved if required by circulating cooled brine.

Reaction is stopped when the required conversion has been reached. From the blowdown tanks the unstripped latex is piped for stripping of unreacted monomers—butadiene is flashed off and styrene removed by low pressure steam stripping. The finished latex passes to storage tanks and from there to the coagulation unit.

Coagulated crumb is freed from surplus serum on a vibrating screen and washed and filtered in a series of operations during which a squeeze-roll conveyor converts it into sheet. This is shredded back to crumb and dried



*The illustration above shows the styrene recovery unit of the synthetic rubber plant at Fort Dunlop, Birmingham. Part of the reactor unit is shown below*

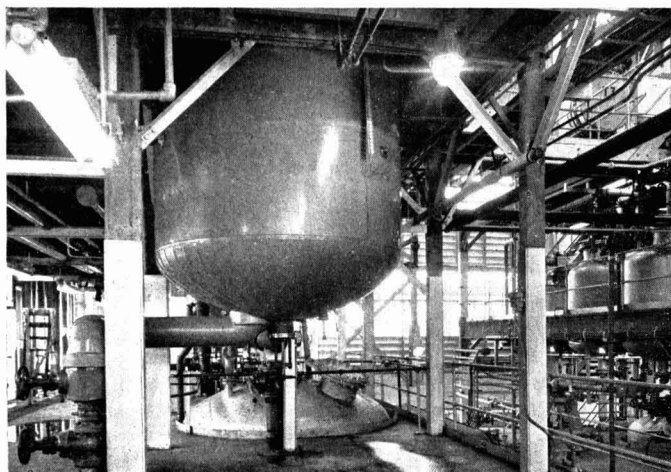
in a three-pass steam heated drier. The dried crumb is fed to the weigher which automatically delivers the required weight to the baling press. The bale of synthetic rubber is dusted in soap-stone, wrapped in a polythene-lined paper bag and stitched ready for despatch.

The new plant is highly mechanised; in fact, the whole finishing process is mechanical until the wrapper is placed in position by hand.

Design and construction of plant buildings and civil work on the site was the responsibility of the company's architects' department in conjunction with the building contractors, Robert M. Douglas (Contractors) Ltd., Birmingham 23.

Dunlop's central purchasing department was responsible for the commercial and contractual aspects of the project, while the works engineers provided all services and utilities and fire protection equipment, sub-contractors for the latter being Mather & Platt Ltd., Manchester.

Foster Wheeler Ltd., London SW3, was the main engineering contractor handling detailed engineering, equipment purchasing and erection supervision. Pipework was installed by Toon & Partners; steelwork by Allen Erecting & Engineering Co. Ltd., Birmingham 23; electrical equipment by Harris & Sheldon (Electrical) Ltd., Birmingham 4 and insulation etc. by Joseph Nadin Ltd., Birmingham 7.



● Metropolitan-Vickers Electrical Co. Ltd. announces that as from 1 January 1957, MR. H. H. LUSTY is appointed manager, publicity department. He succeeds MR. E. E. WALKER, who is retiring but will act in an advisory capacity until the end of January. Mr. Lusty is at present chairman of the Council of the Plastics Institute, a member of the British Plastics Federation publicity committee, and industrial adviser on plastics to the Council of Industrial Design.

● MR. EVERETT M. BARBER, inventor of the Texaco combustion process, and supervisor of special engine research at the Texaco Research Centre, Beacon, NY, was presented with the Richards Memorial Award by the American Society of Mechanical Engineers on 28 November.

● MR. C. B. CAMPBELL, an expert of the atomic energy division of DSIR, is now in New Zealand to examine the uranium deposits found about a year ago in the Buller district on the West Coast. With geologists from the NZ Department of Scientific & Industrial Research, he will spend about three months taking ore samples and completing tests to find out whether the ore is of high enough grade to be worked economically.

● The chief metallurgist of the British Welding Research Association, MR. H. E. DIXON, who has accepted a senior appointment with Richardson Westgarth Atomic Ltd., resigned from the staff on 30 November 1956. During the past eight years Mr. Dixon has made valuable contributions to the BWRA researches.

● DR. ALEXANDER KING, C.B.E., chief scientific officer, Department of Scientific & Industrial Research, has been appointed deputy director of the European Productivity Agency from 1 January 1957. He will succeed MR. EDWIN FLETCHER, who is returning to the TUC from which he was seconded in 1955.

● MR. G. L. PITZER has been appointed vice-president—production, of Bakelite Co., a division of Union Carbide & Carbon Corporation. Mr. Pitzer started with Union Carbide in 1933 in the South Charleston, West Virginia, laboratory of Carbide & Carbon Chemicals. He has held various positions in the Union Carbide organisation including that of superintendent

# People in the NEWS

of the Canadian Resins & Chemicals Ltd. plant at Shawinigan Falls, PQ, Canada, and assistant superintendent, Texas City plant, of Carbide & Carbon Chemicals Co. He moved to New York in 1952 to fill the post of works manager for Bakelite Co.

● MR. C. W. BLOUNT has been appointed vice-president—marketing and MR. J. D. BENEDITO has been appointed vice-president—sales of Bakelite Co. Mr. Blount joined Bakelite Co., a division of Union Carbide, in 1924 as a sales engineer. In 1932 he was appointed assistant sales manager, becoming vice-president and general sales manager in 1944. He was made vice-president in charge of sales in 1953.

● MR. P. G. ASTLES has joined the board of Union Oxide and Chemical Co.

● MR. J. M. REID and MR. W. H. SMITH have been elected to the board of Staveley Coal and Iron Co.

● The Treasury announces that LT.-GEN. LORD WEEKS has been appointed an ex-officio director of the British Petroleum Co. in the place of SIR GORDON MUNRO, who is retiring at his own request because of pressure of other work. The Government holds nearly 56 per cent of BP's ordinary stock and has the right to appoint two ex-officio directors.

● At a recent meeting of the electrical and electronics section of SIMA, MR. L. A. WOODHEAD, director and general manager, Cossor Instruments Ltd., was elected chairman. MR. A. E. EVANS, managing director, Evans Electro-selenium Ltd., was elected vice-chairman. Mr. Woodhead joined A. C.

Cossor Ltd. in 1930, and in 1950 was appointed manager of the company's instrument division. On the formation of Cossor Instruments Ltd., he took over his present position.

● Officers of the American Institute of Chemical Engineers for 1957 are: DR. J. HENRY RUSHTON (*president*); MR. GEORGE E. HOLBROOK (*vice-president*); DR. GEORGE G. BROWN (*treasurer*); MR. F. J. VAN ANTWERPEN (*secretary*); *directors*: MR. JOHN J. HEALY, JR.; MANSON BENEDICT, MR. RICHARD H. WILHELM and MR. J. H. KOFFOLT.

● MR. E. L. HARRISON, sales director, has completed 21 years' service with Quickfit & Quartz Ltd., manufacturers of interchangeable laboratory glassware, and has become a member of the Triplex 21 Club. Mr. Harrison joined the company in 1935, and has held the positions of works superintendent at the Stone factory, commercial sales manager, and sales manager. He joined the board as sales director in January 1955. The Triplex 21 Club, which was formed in 1950, has nearly 150 members—all Triplex group workers with at least 21 years' service.

● DR. ALBERT PARKER, director of fuel research in the Department of Scientific and Industrial Research since 1943, retires on 31 December after 28 years' service with the Department. The Council for Scientific and Industrial Research have appointed DR. A. C. MONKHOUSE to be acting director of Fuel Research from 1 January 1957.

● The Royal Society of Arts Bicentenary Medal has been awarded to DR. W. J. WORBOYS, director of Imperial Chemical Industries. The award is given annually to a person, other than an industrial designer, who has exerted exceptional influence in promoting the development of art and design in British industry.

## WILLS

DR. FREDERICK SODDY, of 39 Overhill Drive, Brighton, Emeritus Lee's Professor of Chemistry, University of Oxford and a pioneer of research into atomic disintegration, who died on 22 September, aged 79, left £36,725 9s 0d. £36,476 16s 6d net value. (Duty paid £8,777). He left scientific equipment to Sir Richard Southwell of Trumpington, and money to cover the publication of his memoirs and for the setting up of a trust for educational purposes.



# Industrial Research

THE WIGGINS TEAPE GROUP ORGANISATION

PAPER technology is a relatively new branch of applied science. It is, therefore, not surprising that the Wiggins Teape Group research organisation is a young organisation. It was founded in 1947 to serve the needs of the group of paper-making, converting and selling companies operating under the parent company, Wiggins Teape & Co. (1919) Ltd.

Actually, it was decided that a research chemist should be appointed before World War I, but the appointment was not in fact made until 1923 when the late Stephen Edge began duty with Alex. Pirie & Son, at Stonywood, Aberdeenshire, a mill making high class writing and printing papers.

In 1922 Wiggins Teape and Alex. Pirie combined. This was a year after a new machine was started up in the former company's Glory Mill, High Wycombe, for making photographic paper. Alex. Pirie ceased manufacture of this grade in 1923 and Mr. Edge became research chemist for the Group. He moved his location several times, but by 1934 it was decided that Glory Mill needed a research laboratory for photographic base alone. Mr. Edge, therefore, returned to Stonywood and Dr. Rance (the present director of research and development) set up the laboratories at Glory mill.

## Continuous Research

Photographic base demands continuous research and the research laboratory at Glory Mill contributed vastly to the many improvements in this paper; Mr. Edge's work in Aberdeen led to a greatly improved knowledge of high class paper making. A review of the patent literature on melamine and urea formaldehyde wet strength resins, reveals that very many patents are held by Wiggins Teape & Edge or Wiggins Teape & Rance. Work in this field and in the field of extractable matter—pitch, uronic acids, lignin etc., led to Glory Mill's being able to satisfy the whole of the war demand for photographic base paper.

On account of ill-health Mr. Edge left Aberdeen in 1947. Dr. Rance was then asked to form a central research organisation. This was based, initially, in Aberdeen and took over the premises and staff of the Stonywood research laboratories. Mr. Edge, meanwhile, ran a technical information service for the Group from Cambridge.

WTGRO, as it is called, started with four graduates and few assistant staff. Its work was divided into sections, but mainly carried out in the laboratory. The sections comprised pulp, paper, chemistry and development. Between 1947 and 1950 chemical research—into extractable residues from pulp, into hemicelluloses, and chemical adhesives for paper continued as did that

started by Dr. Rance on paper structure and rheology. Simultaneously, work was carried out on the beating process (maceration of wood pulp).

In 1950 the staff was doubled and the work changed as the advice of WTGRO was sought 'on the plant floor.' By 1952 there were 11 graduates and as many auxiliary staff.

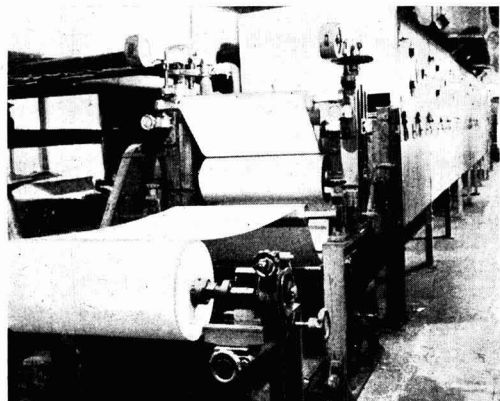
Because of rapid expansion in the south Dr. Rance made Glory Mill his headquarters again. The acquisition, in 1955, of Thomas Owen, papermakers with factories in Cardiff and New Bury, the development of the mills at Chartham, Kent, and Bridgend, South Wales, and the projected building of a pulp mill near Newport, have made necessary a large southern base. The company has, therefore, bought Butler's Court, at Beaconsfield, to serve as research headquarters. Ultimately, about 15 graduates and 15 auxiliary workers will be at Butler's Court and about eight and 12 respectively at Aberdeen.

At the present time, Wiggins Teape has 14 paper mills and a part interest in three others, two coating plants, five envelope and stationery factories as well as warehouses at home and overseas. A pulp mill is under construction, and most of the mills are being modernised and expanded.

Each mill has a technical staff responsible for complete laboratory services on the production side and who assist the sales side on technical matters. New chemicals and processes are investigated and new papers are developed. In all, 120 scientists are employed.

Projects on which WTGRO teams work are decided

*A pilot-plant coating machine at the Wiggins Teape Group research organisation laboratories, Glory Mill, High Wycombe*



## Wiggins Teape

by a committee consisting of three directors of the parent company. The projects are categorised as: (1) Process developments; (2) product development; (3) instrument development; (4) investigations into the characteristics of paper and pulp; (5) raw material evaluations; (6) investigations arising from a customer's query or complaint; and (7) short-term testing work.

One project recently undertaken by WTGRO was the production in UK of a carbonless copy paper made in the US. This paper gives perfect copies without the use of carbons. The research organisation started work in April and delivered 20 tons of paper in July. Output is now hundreds of tons a year, but it is still the responsibility of the research organisation to control the raw materials and check the finished product. This control will continue until the process has reached such a 'mechanical' stage that it can be handed over to the production control laboratories.

Another project which has involved much study has been that dealing with the hygroscopic nature of paper. Because paper is hygroscopic it must be sold in equilibrium with prevalent humidity conditions if it is not going to shrink or stretch, cockle or curl.

Hitherto, it has been customary to overdry paper and then re-wet it in a later process, but as a result of research, this later process is being dispensed with, and paper in equilibrium with any desired ambient humidity can now be produced directly from the machine.

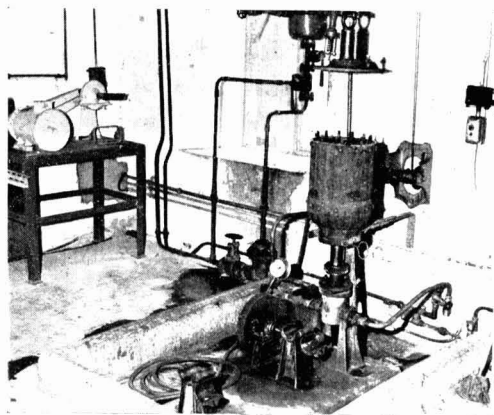
### Projects on Process Development

Several projects on process development are in hand. One concerns the efficient screening of paper pulp suspensions to remove dirt. As a result of this project, paper is now being made cleaner than was at one time thought possible. Work continues also on the installation of statistical quality control schemes on the company's 62 paper and coating machines.

Product development is another undertaking for WTGRO and its work in this field has resulted in plastic laminates, vacuum cleaner bags, and air filter papers; oil and petrol filters, leathercloth base, paper for transmitting photographs by radio, paper for teleprinters, paper chamois leathers etc. Manufacture of a particular paper may take months of laboratory and pilot plant work but this does not cease with the paper development. It is not left until the paper has become easy and economical to make.

Instrument development is a natural auxiliary of process development and the instrument development section has pioneered the use of instruments to measure the thickness, weight, moisture content, formation and opacity of paper as well as the amount of dirt in it. Manufacture is taking place at speeds of from 20 to 1,000 feet per minute. Other instruments are also being developed to measure colour, permeability and gloss.

Naturally process and product development cannot continue without research into the fundamental nature of paper and its raw materials. During the past seven years some outstanding work has been done on the nature of the beating process (by which the same fibre



*Apparatus in the pulping laboratory at Glory Mill*

can be made suitable for blotting paper or tracing paper): work on the structural properties of paper has been done as a corollary to the machine investigations. The presence of 'fatigue' by which, after repeated loading and unloading, paper can be made to break at a fraction of its normal load—or even while it is being unloaded—was found partly as a result of experiments by WTGRO.

Raw material evaluation can pay handsome dividends. In this respect the work done by the organisation on gelatine has reduced variability in tub sized paper considerably. Work is continually being done on additives though this only rarely takes place as a major project—it has to be fitted in as products become available. In this way new products are continually checked and, where possible or economical, absorbed into general use. For instance, sodium hexametaphosphate, activated silica, sodium aluminate, wax emulsions, and other products are now commonplace in paper mills.

Customers' queries are dealt with by the research organisation which also provides technical information. In this connection a large library of books has been built up under a technical information officer who is responsible for arranging the distribution of information to all interested persons in the group. Every report issued in any of the mills is sent to the technical information service for indexing, filing and summarising in a bulletin of abstracts.

Every two months, all heads of laboratories meet under the chairmanship of the director in charge of research and development to discuss their mutual problems. The manager of research is present at meetings of mill managers and a representative is sent to the engineers' meetings.

WTGRO provides chairmen for four paper industry technical committees and sub-committees, two BSI committees, and members for committees in the paper industry, Royal Statistical Society, British Hydro-mechanics Research Association and other bodies.

As a whole the paper industry is short of technically trained staff and, in this respect, Wiggins Teape has a continuing demand for technologists of high quality. The research organisation will continue to grow within the limits set by the economic needs of the Group, and Butler's Court will cater for this expansion.

## SA Petrochemicals

### Sasol Factory's Future Development

SPEAKING at the recent convention of the South African Chemical Institute in Johannesburg, the works manager of the Sasol oil-from-coal undertaking said that as a result of the establishment of the Sasol factory, South Africa now had the basis of a petrochemical industry which could develop to impressive dimensions, was diverse enough to meet the country's needs for many years to come and would also provide scope for profitable export. This result would only be achieved, however, if secondary industry seized its opportunities and made use of the products offered by the oil-from-coal plant and the Iscor iron and steel industry. He insisted that 'if secondary industry is alive to these opportunities, the next decade will witness a notable development in the chemical industry of this country.'

### Rubber Lacquer

A NEW rubber lacquer for application to rubber either before or after curing has been developed by Swale Chemicals Ltd., of Croydon, Surrey. Known as Flexigloss, it is supplied in drums as a complete product ready for use without the addition of catalyst hardeners.

The standard lacquer is a fluid of moderate viscosity which can be applied by brushing or dipping. Good mechanical properties are claimed for the lacquer film. It is said that lacquered samples of rubber can be folded in two, creased or crumpled in the hand without cracking the film.

A piece of rubber about 1/20 in. thick coated on one side can be stretched almost to breaking point before the lacquer is damaged.

The coating is water repellent and can be washed in soapy water. It is claimed to be non-toxic.

Witco Chemical Co. Ltd., Bush House, Aldwych, London WC2, are distributors for Flexigloss.

### First Fertiliser Shipment

MOVEMENT of the first carload shipment of ammonium nitrate fertiliser from the new plant at Medicine Hat, Alberta, of Northwest Nitro-Chemicals Ltd., has been announced by Harrisons & Crosfield (Canada) Ltd., sales agents for the company. The shipment was consigned to buyers in Washington State, under contracts made through the sales agency's Seattle office. Other units of the new plant will begin producing phosphate fertilisers very soon.

## Duty Suspended

UNDER the Supplies and Services (Temporary Provisions) Act 1946, the Irish Republic Revenue Commissioners announce that the Government have made an Order the effect of which is to suspend until further notice the customs duty on superphosphate (ex Tariff Ref. No. 245). Prior to the suspension the duties were: full rate, 20 per cent *ad valorem*; preferential rate, 'nil.' Ground mineral phosphates and compound manures remain chargeable with the duty.

### Nobel Prize Winner

SIR CYRIL HINSHELWOOD, of Oxford University, was among eight people who received Nobel prizes from King Gustav of Sweden at a ceremony at Stockholm on 10 December. Sir Cyril shared the chemistry prize with Professor Nikolai Semenov, of Moscow, for researches into the mechanism of chemical reactions (see THE CHEMICAL AGE, 10 November, p. 244).

### Iraq Oil

IRAQ'S PRODUCTION of oil in September is reported as amounting to 3,014,704 tons, bringing the total for the first nine months of this year to 25,885,334 tons. The Iraq Government's oil revenues for this period amounted to £59 million, and before the present stoppage of the oil flow, were expected to be of the order of £80 million for the whole year. Oil products consumption in Iraq is now at a rate of one million tons a year and estimates for 1960 suggest this consumption will be doubled.

### Detergent Committee

IN THE House of Commons on 18 December, the Minister of Housing and Local Government made a statement on the report of the committee on synthetic detergents.

He said that the main conclusion reached by the committee was that the use of synthetic detergent is, or could be, a source of difficulty in sewage disposal etc. The committee recommend research, by manufacturers and others, aimed at finding the means of ensuring that these substances would be destroyed during sewage treatment.

The Minister further stated that he is to appoint, in consultation with the Secretary of State for Scotland, a standing technical committee to secure that the desired research is carried out effectively.

## Laboratory Equipment

### Interesting Gadgets in New Publication

SCIENTIFIC INSTRUMENT and laboratory equipment suppliers, Glencreston Ltd., Church Road, Stanmore, Middlesex, have published the November industrial issue of *Lab Mail*. Information is given on the p.v.c. laboratory pump. The pump housing and impeller are made from hard synthetic p.v.c. based material which is highly corrosion resistant and physiologically neutral. Three gallons per minute at 2 ft. head can be dealt with; maximum total discharge head is 12 ft.

A quick-acting tubing compressor is illustrated which provides a very rapid and easily operated flow control for use on rubber tubing up to 15 mm diameter. With a flick of the lever the flow in the tube can be stopped instantly.

Another interesting gadget is a siphoning starter which eliminates the unhygienic and very often dangerous practice of suction by mouth to start flow in siphoning tubes. Rubber tubing used for siphoning is compressed between the two rollers of the tongs. By pulling the tongs downwards, vacuum is created in the tubing which lifts the liquid and causes it to flow when the tongs are released. Even heavy wall tubing can be easily compressed with very little effort.

### Oil Supplies

ON 30 NOVEMBER, OEEC set up a Petroleum Emergency Group (OPEG) which will represent Europe at Washington negotiations with the American Middle East Emergency Committee.

In OEEC's new oil survey, Europe's serious situation is stressed. At present Europe can count on receiving little more than 60 per cent of her estimated requirements, unless Western Hemisphere supplies are greatly increased. European stocks will also diminish rapidly.

Until Middle East oil is fully renewed, the most that Europe can hope to receive is some 75 per cent of her requirements. It is estimated that to achieve this, Western Hemisphere production must increase by 600,000 to 800,000 barrels a day. Experts consider, however, that this increase is within the capacity of the producers.

### Change of Address

FROM 10 December Charles Lennig & Co. (Great Britain) Ltd. will have its offices at 26-28 Bedford Row, London WC1 (telephone CHAncery 6634-6).



# From all Quarters



## Isotopes in Finland

MONETARY VALUE of imported isotopes from the UK into Finland amounted to £200 in 1954, to £1,100 in 1955 and during the first ten months of this year to £1,100.

Co-60 and Cs-137 are being used for non-destructive testing purposes and recently there has been a keen interest in the use of isotopes in the study of flow phenomena, leakage and mixing. Na-24 has been used in tracing the mass transfer in melting glass.

## Insecticides

TWO NEW INSECTICIDES will be marketed shortly in Malaya by a Singapore company, the Guaniseit Co. One is a domestic insecticide which, it is claimed, will destroy all types of insects. The other type is designed to protect trees, plants, vegetables and flowers against all types of insects and fungus.

## Japanese Uranium

A NEW PROCESS for refining uranium ore of poor quality is claimed by a Japanese scientist in a Ministry of International Trade and Industry research centre. The process is described as an evaporation method of uranium extraction through chlorination. The pulverised ore is heated to between 700° C and 800° C in a furnace through which a mixture of chlorine and carbon monoxide gases are then blown. Metallic uranium is extracted from the resulting uranium chloride vapour by the usual electrolytic method.

## South African Symposium

THE SOUTH AFRICAN Institute of Mining and Metallurgy is participating in a joint symposium on the Development of the South African Uranium Industry in collaboration with the South African Chemical Institute, the Geological Society of South Africa, the South African Institute of Mechanical Engineers and the Institution of Chemical Engineers, South African Branch. Papers will include a survey of the develop-

ment of the process, formulation of uranium plant design, reviews of uranium leaching and precipitation practice, accounts of pyrite and uranium flotation and a description of uranium laboratory control.

At the conclusion of this joint symposium (March, 1957), it is intended to have all the papers combined in a single volume to form a comprehensive record of the geology, mineralogy, chemistry extraction and processing of uranium oxide from mines of the Transvaal and Orange Free State goldfields.

## Turkish Oil

OIL from the Raman and Garzan fields is now being refined at the State-owned refinery at Batman, Turkey. A thousand tons a day are refined and are supplying all the requirements of Turkey's eastern provinces as well as asphalt sufficient for the country's present needs.

Negotiations are to be resumed on the proposed oil refinery near Istanbul on the Sea of Marmara. The refinery, to be jointly operated by four companies, would cost over \$50 million and have a throughput of 3 million tons annually.

## Italian Mercury & Sulphur

MERCURY OUTPUT in Italy increased by 14 per cent from 883 tons to 1,011 tons. Exports of mercury during the first seven months of this year increased to 1,722 tons, against 583 tons during the first seven months of 1955.

Production of sulphur for the first six months of this year was 96,804 tons of which 54,964 tons were exported. Stocks, however, are still reported to be considerable.

## Indian DDT

A PLANT for formulating 1,400 tons of technical DDT per annum to 50 per cent and 75 per cent water dispersible powder conforming to WHO's specifications is to be set up in the vicinity of the technical DDT plant at Alwaye, Travancore-Cochin, India.

## Canadian Hexylene Glycol

BA-SHAWINIGAN LTD., Montreal, jointly owned by British American Oil Co. Ltd., and Shawinigan Chemicals Ltd., will commence production of hexylene glycol at its plant in Montreal East in March 1957.

The chemical is derived from acetone, a product now made by BA-Shawinigan. It is used in hydraulic brake fluids, printing inks, and in the treatment of textiles. It is not now made in Canada, and the new production will be large enough to supply the Canadian market.

Hexylene glycol is the second of a series of commercially important derivatives of acetone to be produced by BA-Shawinigan. Isopropanol has been produced since early 1955.

## N. Zealand Conference

QUICKFIT & QUARTZ interchangeable scientific laboratory glassware is to be exhibited at the International Conference of Scientists to be held in Dunedin, New Zealand, in January. Over 3,000 scientists will visit New Zealand to attend this congress. The glassware will be exhibited by Quickfit & Quartz's New Zealand distributors, Townson & Mercer (New Zealand) Ltd., of Auckland.

## Trichlorethylene in Portugal

IT IS REPORTED that trichlorethylene is now being manufactured by the Belgian controlled chemical manufacturing company of Soda Povoá S.A.R.L., Povoá de Santo Iria, near Lisbon, Portugal.

## European Office

SHORTLY after 1 January the Stanford Research Institute, of Menlo Park, California, US, will open a European office, probably at Zurich. For the past eight years SRI has been working in Europe and the new office is being opened in response to mounting requests for assistance that could best be met with a central location and resident staff.



by  
Olof Samuelson &  
Nils-Herman Schöön

# Ammonia from Spent Liquor

Magnesium Oxide Distillation Method—Part 1

SEVERAL methods have been suggested by various authors to recover ammonia from ammonium bisulphite spent liquor. None of these methods have as yet found industrial application. Among the suggestions should be mentioned: ammonium uptake by ion exchange (1); pyrolysis of the evaporated liquor (2); or distillation with an alkali, such as black liquor (3) or magnesium oxide (4).

In a previous paper (5) it was shown that when ammonium bisulphite spent liquor was distilled with sodium hydroxide, a part of the ammonia remained bound in the spent liquor because of the formation of so called 'bound nitrogen.' A loss of ammonium ions ('free nitrogen') occurred already during sulphite cooking (6). These observations suggested the usefulness of a closer investigation of the conditions for the recovery of ammonia.

## Ammonia Recovery

This paper is chiefly concerned with ammonia recovery by distillation with magnesium oxide. The distillations described below were performed with varying temperatures, varying rates of distillation and varying amounts of magnesium oxide. The influence of the reactivity of the oxide will be dealt with in a later investigation. Ammonium sulphite spent liquor used in most experiments came from Toten A/S, Nygard, Norway, and from Billeruds AB Jössefors. In some experiments a calcium sulphite liquor from Mo & Domsjö AB Hörnefors, was used. The calcium ions were exchanged for hydrogen ions by passing the liquor through a cation exchange resin and ammonia was added to the effluent.

Distillations were performed at varying distilling rates with 100 ml. sulphite spent liquor and a constant amount of magnesium oxide, 15 g. per litre in a 600 ml. flask. The flask was equipped with a tightly fitting rubber stopper, in which three holes were drilled, for distilling away ammonia, for stirring and for addition of spent liquor. After passing through a splash head, the vapours were condensed in

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a cooler. A wing stirrer was connected via a mercury seal. The distillation was carried on until ca. 60 ml. distillate were obtained.

Several runs were performed by steam distillation, in which cases the 600 ml. flask was replaced by a 500 ml. Kjeldahl flask, which was heated by a heater mat in order to keep the volume of the solution constant throughout the experiment.

The distillation was carried out in an autoclave with a magnetic stirrer (from Andreas Hofer, Mühlheim-Ruhr). A number of splashheads were connected to the autoclave, as well as coolers with receivers. In the autoclave two litres spent liquor were distilled at 150°C during 1.5 hours, with 30 g. magnesium oxide. The amount of distillate obtained was one litre. The apparatus was constructed for batch runs only, and unfortunately, the heating up time (two hours) was rather long. Therefore these experiments do not give an entirely satisfactory picture of the conditions under elevated pressure.

## Vacuum Evaporator

These experiments were carried out in a laboratory vacuum evaporator, built in stainless steel and glass (from Kurt Herbert, Lahr, Baden). The evaporator was heated indirectly by steam from an electric steam boiler.

The construction of the apparatus allows a feed and removal of liquor during the evaporation.

Two litres of liquor were used in each run. This was the greatest volume which could be evaporated without the necessity of removing liquor from the evaporator during the course of an experiment. In all experiments, 1.0—1.2 litres water were distilled off. After cooling the distillate was taken up in sulphuric acid.

In the experiments at atmospheric pressure the yield of ammonia in the distillate was determined by direct titration of the excess acid with 0.1N sodium hydroxide. In the other series, the receiver contained N sulphuric acid, from which a small part was taken for analysis, and ammonium determined in the customary way by distillation with excess sodium hydroxide. Foaming of the spent liquor was suppressed by the addition of an alkylaryl polyglycol ether (Berol VMA 09). In order to decrease corrosion 0.13 per cent sodium arsenate was added to the receiver in the vacuum evaporator.

## Distillation Results

The ammonium bisulphite spent liquor on which the experiments were carried out contained 26.6 mmol free nitrogen per 100 ml. and 2.4 mmol bound nitrogen per 100 ml. The results from distillation experiments performed at atmospheric pressure for varying times are given in Table I.

The table shows that a considerable formation of bound nitrogen occurs, particularly at long distilling time. The values given in the table were obtained by Kjeldahl digesting of the alkaline solution remaining after the determination of free nitrogen in the residue. A correction was introduced for the original

TABLE I  
Distillation at atmospheric pressure

Time of distillation min.	Ammonia recovered in distillate		Ammonia in residue mmol.	Conversion into bound nitrogen		Nitrogen accounted for mmol.
	mmol.	per cent		mmol.	per cent	
14	24.6	92.5	1.0	1.1	4.1	26.7
27	23.8	89.5	1.2	1.8	6.8	26.8
38	23.6	88.7	0.9	2.2	8.3	26.7
42	23.4	88.0	0.8	2.3	8.6	26.5
64	22.8	85.7	0.8	3.0	11.3	26.6
87	22.3	83.8	0.8	3.3	12.4	26.4
135	21.3	80.1	0.8	4.5	16.9	26.6
393	19.4	72.9	0.7	6.7	25.2	26.8

## Ammonia Recovery

amount of bound nitrogen in the liquor. The remaining amount of free nitrogen in the residue was determined by steam distillation with sodium hydroxide according to a method especially devised to prevent additional formation of bound nitrogen (5).

As mentioned above, the experiments were carried on for 1.5 hours at 150° C. After this distillation 29 per cent only of the original amount of free nitrogen could be found in the distillate and 10 per cent in the residue. Of the originally present ammonium ions 61 per cent have thus been transformed into bound nitrogen.

### Reaction Temperatures

From the above distillation experiments it is evident that high reaction temperatures and low distilling rates contribute to an increased formation of bound nitrogen. Thus a change towards distillation under vacuum appears in several ways advantageous. Firstly, the distillation characteristic for the system ammonium-water is much steeper at low pressures (7); and secondly, the solubility of magnesium hydroxide increases at low temperatures. According to Travers and Nouvel (8), the solubility at 35° is 4.6 times that at 150° C.

The experiments were carried out with two litres of sulphite spent liquor, containing 272 mmol. free nitrogen per litre. In the individual runs the amounts of magnesium oxide were varied. In Table II two series of runs are recorded, differing from each other by the fact that in the latter series the evaporation was driven a little further. As can be readily seen, there was no or only an insignificant formation of bound nitrogen in these experiments. A considerable part of the free nitrogen is present in the reaction vessel.

### Different Situation

Quite a different situation arises in the case of distillation with sodium hydroxide under similar conditions. The distillation of two litres of sulphite spent liquor with 25 g. sodium hydroxide gave a complete yield in the same apparatus. This difference between sodium hydroxide and magnesium oxide can be explained by the fact that magnesium oxide dissolves slowly at low temperature.

In order to study more closely how pH, reaction time and temperature influence the formation of bound nitrogen, the present distillation investigation was completed by experi-

MgO added	Time of distillation	Temp. of heating steam	Boiling temp.		pH in residue	Ammonia recovered in distillate		Ammonia in residue	Ammonia accounted for
			°C.	°C.		mmol./l.	per cent		
g/l	min.	C.						mmol./l.	mmol./l.
5	30	70	38	5.7	20.5	7.5	250.0	271	
10	30	70	35	8.0	121.0	44.5	150.0	271	
15	30	68	34	8.0	187.0	68.8	81.5	269	
20	30	68	34	9.6	228.0	83.8	41.5	270	
20	40	72	33	9.9	252.0	92.6	17.6	270	
25	40	73	32	9.9	255.0	93.8	14.8	270	
30	40	74	32	10.1	256.0	94.1	13.8	270	
35	40	73	30	10.1	260.0	95.6	11.0	271	

ments where the liberated ammonia was not driven off. Thus 200 ml. ammonium sulphite spent liquor were treated in steel autoclaves at 145° C in the pH interval 2.2—7.2.

Sodium hydroxide solution was added to the spent liquor to obtain the desired pH and the solutions were diluted with water to equal volumes. The pH value in the liquor before the experiment was calculated with the aid of a previously determined titration curve. The solutions were heated for three hours to 145° C. At this temperature, treatment was carried on for 3.5 hours. The sulphite spent liquor used contained 121 mmol. free nitrogen per litre and 10 mmol. bound nitrogen per litre.

After the completion of the run, the pH of the solution was determined, as well as the free and bound nitrogen contents. The amount of bound nitrogen formed during the experiment was calculated as the difference between total nitrogen and free nitrogen, after subtracting the original amount of bound nitrogen in the liquor.

One series of runs was also performed at 35° C. Here the ammonium

bisulphite spent liquor was shaken together with magnesium oxide (25 g. per litre) in a thermostat. After neutralisation with N sulphuric acid the remaining amount of free nitrogen was determined. The liquor used contained 182 mmol. free nitrogen per litre and 26 mmol. bound nitrogen per litre.

The results related in Table III show that bound nitrogen also forms in an acid medium, which result confirms earlier experience in connection with sulphite cooking (6). From Table IV it can be seen that in a weakly alkaline solution a noticeable loss of free nitrogen occurs already after a comparatively short treatment at 35° C.

### Considerable Influence

The experiments described above show that the reaction time has a considerable influence on the formation of bound nitrogen. Attempts were therefore made to reduce the conversion of ammonium ions into bound nitrogen by carrying out the stripping of ammonia as a steam distillation. 100 ml. sulphite spent liquor, containing 26.6 mmol. free nitrogen, were distilled after the addition of varying amounts of magnesium oxide. As can be seen from Table V, a greater yield of ammonia is obtained than for distillation without the passage of steam.

The yield increases with the amount of oxide and for an amount of 18—20 g. per litre the yield is 97 per cent. For continued addition of magnesium oxide, the amount of distilled ammonia decreases. When distilling with excess concentrated sodium hydroxide in the same apparatus, a yield of 98.2 per cent was obtained.

Thanks are due to Mr. Nils Karlson for his assistance with the laboratory work. Statens Tekniska Forskningsrad has supported the research financially, which support is gratefully acknowledged.

### REFERENCES

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- (2) Markham, A. E., Peniston, O. P., & McCarthy, J. L., *Paper Tr.*, 1948, 127, 1, 64.
- (3) Jenness, L., C. Durst, R. E., & Tode, E. F., *TAPPI*, 1953, 36, 337.
- (4) Helleur, D., US Patent 2596241.
- (5) Samuelson, O., & Schoon, N.-H., *Svensk Papperstidn.*, 1954, 57, 850.
- (6) *Ibid.*, 1955, 58, 894.
- (7) Wucherer, J., *Z. ges. Kalteind.*, 1932, 39, 97, 136.
- (8) Travers, A., & Nouvel, *CR Acad. Sci. Paris*, 1929, 188, 499.

pH before treatment	pH after treatment	Free nitrogen mmol./l.	Total nitrogen mmol./l.	Conversion into bound nitrogen per cent
2.2	1.9	113	130*	5.8
3.2	2.5	112	131	7.4
4.2	3.3	109	131	9.9
5.2	3.9	104	131	14.0
6.2	4.3	101	131	16.5
7.2	4.5	97	129	18.5

\* The original amount of bound nitrogen in the liquor is 10 mmol./l.

Time of reaction min.	Free nitrogen mmol./l.	Conversion into bound nitrogen per cent
0	182	0.0
40	175	3.8
60	173	4.9
120	171	6.0
240	171	6.0
360	170	6.6

MgO added	Ammonia recovered in distillate	pH in residue
g/l	mmol./l.	per cent
10	203	76.3
15	248	93.2
18	258	97.0
20	258	97.0
30	256	96.2
40	253	95.1
50	251	94.4
60	253	95.1

# INDIAN NEWSLETTER

by Our Special Correspondent

**A**N IMPORTANT DECISION recently taken by the Government of India relates to the manufacture of intermediates required for the production of dyestuffs, drugs, plastics and explosives. The National Development Corporation of India, a private limited company owned by the Government of India, will be in charge of the project which is estimated to cost Rs. 18 crores. This decision on the multi-purpose project is a part of the development programme of the dyestuffs and drugs industry. The project envisages the setting up of a central unit, under the public sector, for the manufacture of primary intermediates. Companies in the private sector, already licensed or registered for undertaking the manufacture of dyestuffs have been asked to indicate the lines of development which they are willing to undertake regarding the production of late intermediates.

## Dyestuffs' Imports

India has been importing increasing amounts of dyestuffs in the past few years and by 1960 the consumption is expected to be of the order of 10,000 tons valued at Rs. 30 crores. There are at present 11 units in the private sector producing dyestuffs based on imported intermediates and penultimate products.

Project reports on the dyestuffs industry submitted by Montecatini of Italy, ICI, and Bayer of Germany have indicated the practicability of establishing a dyestuffs industry in India. According to one report, it is necessary to manufacture 57,076 tons comprising 153 intermediates. For this purpose the setting up of two factories, one in Bombay State and another in West-Bengal-Bihar, has been proposed. The factories are estimated to cost Rs. 8 crores, half of which will be needed for plant and machinery. The Bombay factory will comprise plants for chlorination, sulphonation, acetylation, alkali fusion, nitration and reduction. The other will have plants for the manufacture of phthalic anhydride and maleic acid, alkyl resins, phenol,  $\beta$ -naphthol, and equipment for refining benzene and toluene. The Bombay factory would

be manufacturing 90 products and the rest (excluding alkyd resin,  $\beta$ -naphthol, phenol, phthalic anhydride, maleic acid, benzene and toluene) will be produced in units other than these two. The report also indicates that products acetylene may be manufactured in other units. The manufacture of the following products, viz. Fast Blue BD Base, acid urea,  $\beta$ -naphthylamine, diphenylether, cyanuric chloride, phenyl isocyanate, diethyl meta-anilic acid, 5-chloro-2-toluidine has not been recommended as their manufacture would be too intricate and costly.

The Expert Committee set up last year by the Synthetic Oil Committee of the Government of India to draw up a project report for the production of synthetic oil has now submitted it. The committee recommends the establishment of a plant for the production of 800,000 tons of soft coke and 120,500 tons of synthetic petrol per annum, at an estimated cost of Rs. 20 crores including capital expenditure on coal mining. The oil produced will meet about 15 per cent of the present requirements of the country. About 1.2 million tons of coal from Jambad seams will be treated per annum.

A Committee of eleven has been appointed by the Government to advise on the location of a refinery for refining crude oil produced in Assam. The refinery will be set up in the public sector. A representative from France and from Rumania are on the Committee which is expected to submit its report shortly. The probable output of crude oil in Assam is estimated at two million tons per annum.

## Fertiliser Committee

The technical Committee appointed by the Government of India to consider the question of increasing the production of nitrogenous fertilisers has proposed expansion of the existing units at a cost of Rs. 2.5 crores. The Committee has pointed out that the cost of production could be brought down by producing ammonia employing electrolytic hydrogen and nitrogen from liquid air. One of the proposals to increase the output of fertilisers is to expand the manufacturing capacity of Travancore Ferti-

lizers and Chemicals Ltd. which includes (1) increasing ammonia production to 70 tons per day (2) stepping up the production of ammonium sulphate from 135 tons to 200 tons per day and (3) production of new fertilisers such as ammonium phosphates.

Experts of the Government of India and the representatives of the Associated Electricals, UK, who are the technical consultants for the proposed heavy electrical equipment factory, have agreed on the items to be manufactured during the first stage of production. The factory which is expected to go into production by 1960 will attain full capacity in 1967. A private limited company known as the Heavy Electricals (Private) Ltd., entirely owned by the Government, has been set up to manage the project. The schedule of manufacture includes a.c. and d.c. rotating machinery, rectifiers, transformers, gears, motor starters, capacitors, water-wheels, turbines etc. Switch-board instruments, meters, and relays, insulating materials and steam turbines with ancillary equipment will be manufactured at a later stage.

## Scientific Instruments

Problems relating to the production, standardisation and testing of scientific (surveying, drawing and mathematical) instruments in the country to meet increasing requirements were discussed at the first meeting of the Scientific Instruments Committee held at New Delhi during October 1956.

The Committee was set up in June 1956 by the Government of India to examine the progress made in the manufacture of scientific instruments, suggest methods for further development of this industry and for co-ordination between the existing small scale industries and the factories under the Ministries of Production and Defence.

## Demand Increasing

Union Minister of Consumer Industries, Shri Kanungo observed at the Delhi meeting that the demand for scientific instruments in the country was increasing due to the engineering, housing and industrial sectors. The objective should be to step up production of tools, equipment and instruments of the country. With this end in view, new industrial undertakings and factories have been sponsored by the Government during the last few years. The National Instruments Factory at Calcutta is being expanded, to cope with the increased demand. The Ordnance Factory at Dehra Dun, which was set up for the manufacture of special instruments for the Services, has gradually taken on the manufacture of scientific instruments for civilian needs.

## LATIN AMERICA'S MINERAL RESOURCES

### US Capital Playing Major Role in Development

TODAY Latin America's resources of copper, lead, zinc, tin, oil, bauxite and iron ore gave it increasing prominence as a world source of minerals, says the current *Monthly Review of The Bank of Nova Scotia*, which is devoted to a broad survey of Latin American mineral resources, including those of the Caribbean area.

Like Canada, the *Review* points out, Latin America has long been a major producer of base metals; indeed they compete in world markets. As in Canada, newer resources have recently come into prominence, US capital playing a major role in their development in both areas.

Transportation and basic government services are, however, generally much less adequate in Latin America and power is scarcer and more costly. It is for purposes such as these that most of the \$653 millions in World Bank loans to Latin American countries and several hundred millions of the \$1,000 millions in post-war Export-Import Bank loans to the area have been granted.

#### Investment

Before the war, says the *Review*, the direct private investment of American capital in Latin American mineral industries greatly exceeded that in Canadian mineral industries. But today the difference is small—\$2,200 million for Canada and \$2,800 million for Latin America at the end of 1955. The greater post-war rise in Canada reflects not only the growth of the oil industry, but also the fact that American investors generally appear to have regarded Canada as a more stable area for investment than the majority of Latin American countries.

However, in the last two or three years, new legislation in several countries has provided tangible evidence of the desire to encourage foreign participation in the development of mineral resources. In Chile, for instance, the passage in 1955 of a new copper law improved taxation and marketing conditions. With this new legislation and high prices, copper appears to be entering a new era of development. Indeed, a revived flow of new capital into major mining and petroleum undertakings is again in prospect in several countries.

The importance of Latin America as a world supplier of minerals is illustrated by some statistics. In 1955 this region produced approximately one-fifth of the world's output of copper, lead and zinc. Bolivia supplied some

16 per cent of the tin, and the Caribbean area about one-half of the bauxite.

For petroleum, the area is one of the two great surplus-producing regions of the world, contributing in 1954 about one-third of the petroleum and petroleum products entering into world trade. And its commercial reserves of iron ore are estimated at some 25,000 million tons, or 30 per cent of the estimated world total.

Several current projects financed by US capital show the growing diversity of the Latin American mineral picture. A manganese deposit is being developed in Brazil north of the Amazon and a titanium deposit in Mexico. The expanding production of sulphur from the salt domes in the Isthmus of Tehuantepec is making Mexico an important source of supply for this mineral. And work is being carried on to solve the metallurgical difficulties of producing nickel from the large Cuban deposits.

In discussing the effects of mineral development on the various Latin American economies, the *Review* points out that there is a tendency for an increasing proportion of the mineral output to receive some degree of processing before being exported. A good deal of the post-war development has involved the building of smelters and refineries, and this trend is likely to continue.

#### Export

As yet, fabricating industries based on local resources have been little developed, and it seems likely that for a long time to come Latin America will export the bulk of her mineral production in simply processed form.

Nevertheless, the recent exploitation of iron ore resources has helped to bring about the development of small steel industries in several countries. And in Brazil and Mexico a marked growth of secondary steel-using industries is proceeding. The *Review* notes that European capital is involved in some of these secondary industries, in contrast to recent primary developments which have been largely financed by US capital.

### German Ethylene Oxide

AN ETHYLENE OXIDE PLANT, with a capacity of 12,000 tons a year is to be built by the Bayer chemical works in Germany. The process of the US Scientific Design Co. will be used.

## Fibre Damage

### Dyeing Has Little Effect on Wool

IN HIS ADDRESS on 'Dyeing and Fibre Damage' to the Dewsbury Textile Society on 27 November, Dr. G. H. Lister, of Bradford, said that there were many complaints that dyeing reduced the properties of wool. However, on technical examination it was found that 90 per cent of the complaints referred to effects occasioned by the manufacturing processes rather than to any weakening in or damage to fibres. He asserted that fibre damage by dyeing in relation to the consumer life of a garment was very small. In reply to a question, he said that a solution had yet to be found to the problem of dyeing rags containing synthetic fibres for this was likely to become more serious in the future with the increasing use of artificial fibres.

## Sulphur Topics

A MOST INTERESTING COLLECTION of articles and news items on sulphur are contained in the *Quarterly Bulletin*, September 1956, No. 14 of the British Sulphur Corporation Ltd. Canada's sulphur industry and US production of Frasch and recovered sulphur are reviewed, sulphur prices and trends are discussed, the Bonnie Chemical plant near Bartow, Florida, is described, and reports are given of sulphur production in the UK, Italy, Mexico etc. There is a useful account of the use of sulphur in nickel refining and a description of sulphur recovery from total coal gasification at the new plant of Steinkohlengas AG, Westphalia, Germany.

## US Oil Sales

THE US Bureau of Mines forecasts that US oil producers will sell 25 million barrels of oil more in January and subsequent months than in November because of increased demand from Britain and Western Europe.

Sales in December are expected to rise by 20 million barrels. According to the Bureau, the 25 million-barrel-monthly increase will continue until the Suez Canal is cleared. An increase of some 700,000 barrels a day is expected in December sales.

An export level of 400,000 barrels a day is understood to have been reached by the end of November. By mid-January exports are expected to increase to at least 500,000 barrels a day. An additional 100,000 barrels a day is expected to be sold on the US domestic market as US imports of Middle East crude are further curtailed.



# PATENTS

By permission of the Controller of HM Stationery Office the following extracts are reproduced from the 'Official Journal (Patents)'. Copies of this publication may be obtained from the Patent Office (Sale Branch), 25 Southampton Buildings, Chancery Lane, London, WC2, price 2s 6d per copy (including postage); annual subscription £6 6s. The letter P or C preceding the number indicates that the application was accompanied by a provisional specification or a complete specification respectively.

## APPLICATIONS

- P32972 Detergent compositions. Hedley & Co. Ltd.  
 C33024 Carboxylic acids salts rearrangement process. Henkel & Cie Ges.  
 C33271 Nitrosamines catalytic hydrogenation. Hercules Powder Co.  
 C32886 Quinolizone derivatives. Hoffmann-le Roche & Co. AG.  
 C32926 Antifreezing agents corrosion inhibitor. Holm, H. W.  
 C32920 Chemical process. Horizons Titanium Corp.  
 C33360 Gasoline &c. catalytically reforming process. Houdry Process Corp.  
 P33205 Mononitric esters. Imperial Chemical Industries Ltd.  
 P33204 Organic substances oxidation. Imperial Chemical Industries Ltd.  
 C33132 Silicon hydride compounds. Kali-Chemie AG.  
 C33077 Chrome plating bath. Kampschulte, H., and Farfsing, P.  
 C32744 Fuels gasifying arrangement. Koppers Ges., H.  
 C32678 Antibiotics salts. Laboratorio Atral Ltd.  
 C32994 Nicotinic acid esters. Lannacher Heilmittel Ges.  
 P32825 Material evaporating methods. Lubszynski, H. G.  
 P32975 Viscous liquid materials deposition apparatus. McCorquodale Colour Display Ltd.  
 P33423 Frequency spectrum analysers. Marconi's Wireless Telegraph Co. Ltd.  
 P32801 Amides. May & Baker Ltd.  
 P32802 Heterocyclic compounds. May & Baker Ltd.  
 C32933 Steroid compounds. Merck & Co. Inc.  
 C32736 Steroids. Merck & Co. Inc.  
 C33300 Gases mists separating method. Metallges AG.  
 P32864 Nickel-chromium-cobalt alloys. Mond Nickel Co. Ltd.  
 C32889 Rubbery copolymers modification. Monsanto Chemical Co.  
 P33185 Mycobacteriostatic compositions. Monsanto Chemicals Ltd.  
 P32703 Chemical etching. Mullard Ltd.  
 C32671 Epoxy ethers curing process. Naamlooze Vennootschap de Bataafsche Petroleum Maatschappij.  
 C33136 Organic titanium complex. National Lead Co.  
 C33442 N-methyl-a-phenyl-a-methyl-succinimide. Parke, Davis & Co.  
 C32686 Antibiotic compositions. Pfizer & Co., Inc., C.  
 C32687 Industrial fermentation. Pfizer & Co., Inc., C.  
 C33216 Steroid compounds. Pfizer & Co., Inc., C.  
 C32928—C32929 Nuclear reactors fuel elements. Physikalische Studienges Dusseldorf.  
 P33324 Lubricating compositions. Ranson Grease Co. Ltd.  
 C33428 Hydroxychromone derivatives. Recordati-Laboratorio Farmacologico Soc. per Azioni.  
 C32930 Catalysts regeneration. Reichhold Chemicals, Inc.  
 C32931 Chlorinated phenols. Reichhold Chemicals, Inc.  
 C32709 Meat tenderising devices. Sala, A., and Catellani, R.  
 P33034 Thermo-plastic materials. Shaw, H., and Bentley, F. H.  
 C32961 Anthraquinones hydrogenation process. Solvay & Cie.  
 C32962 Hexamethylene diisocyanate. Solvay & Cie.  
 P33021 Iron oxides. Spence & Sons Ltd.  
 C32896 Fluids flow rate ratio maintaining device. Staribacher, A.  
 C32921 Ferrites. Steatit-Magnesia AG, and Steatite Research Corp.  
 C32718 Resins. Stoubendorff, A. de.  
 P33255 Polymers bonding method. Stuart, N.  
 C33410 Cyclopentanophenanthrene derivatives. Syntex Soc. Anon.  
 P32754 Vegetable fibres treatment. Turnbull, R. H.  
 C32855 Polymerisable silane derivative. Union Carbide & Carbon Corporation.  
 C32856 Polysiloxane compounds. Union Carbide & Carbon Corporation.  
 C32857 Cyclsiloxane polymers. Union Carbide & Carbon Corporation.  
 C32858 Ethylvinylsiloxane cyclic polymers. Union Carbide & Carbon Corporation.  
 C32859 Polysiloxane compounds. Union Carbide & Carbon Corporation.  
 C32860 Polysiloxane compound. Union Carbide & Carbon Corporation.  
 C32747—C33400 Steroids. Upjohn Co.  
 C32734 Polyethylene terephthalate threads. Vereinigte Glanzstoff-Fabriken AG.  
 C32807 Liquids removing process. Wacker-Chemie Ges.  
 C33402 Liquid solids incorporation. Wallace & Tiernan, Inc.  
 P32820 Zirconium alloys. Wanklyn, J. N.  
 P32996 Moisture content determining apparatus. Weeks, F., and Weeks, H.  
 P33403 Polyacrylonitrile fibres dyeing process. Wolsey Ltd.  
 C33082 Carbon-substituted piperazines. Wyandotte Chemicals Corp.  
 P32918 Epoxide resins. Yarsley Research Laboratories Ltd.

## ACCEPTANCES

*Applications in the following list, and the specifications filed in pursuance thereof, will be open to public inspection in due course. Persons interested may give notice of opposition to the grant of a Patent on any of the applications included in the list by filing Patents Form number 12 at any time within the prescribed period. See 'Official Journal (Patents)' for dates on which these applications will be open to public inspection.*

- 765 356** Colouration of fibres having a polyvinyl derivative base. Compagnie Francaise des Matieres Colorantes.  
**765 357** Ammonia and hydrogen chloride production and recovery from ammonium chloride. Olin Mathieson Chemical Corporation.  
**765 696** Manufacture of pesticidal compositions. Berk & Co. Ltd.  
**765 750** Detergent compositions. Hedley & Co. Ltd.  
**765 485** Detecting corrosion of metals by gases. Imperial Chemical Industries Ltd.  
**765 700** Mixtures of perhaloacylhalides and preparation of same. Union Carbide & Carbon Corp.  
**765 752** Solvent process. Esso Research & Engineering Co.  
**765 585** Polymers. Du Pont de Nemours & Co., E. I.  
**765 486** Carbon black dispersions. Columbian Carbon Co.  
**765 233** Purification of relaxin by ion-exchange. Warner-Lambert Pharmaceutical Co.  
**765 590** Derivatives of purine. Wellcome Foundation Ltd.  
**765 591** Esters of 4-amino-2-hydroxybenzoic acids monosubstituted at the nitrogen atom; production process. Rheinpreussen AG, Fuer Bergbau und Chemie.  
**765 488** Vinyl chloride resins for application as solvent solutions. Firestone Tire & Rubber Co.  
**765 489** Radioactive phosphorus. UK Atomic Energy Authority.  
**765 372** High temperature lubricant. Esso Research & Engineering Co.  
**765 597** Polytetramethylene terephthalate. Imperial Chemical Industries Ltd.  
**765 374** Thermoplastic synthetic resin-coated fabrics. Wardle (Everflex) Ltd.  
**765 602** Manufacture of alkali metal carbonates. Diamond Alkali Co.  
**765 494** Magnesium base alloys. Soc. D'inventions Aeronautiques et Mecaniques SIAM.  
**765 603** Titanium ester complexes. National Lead Co.

## PATENTS ACCEPTANCES

- 765 500** Copolymers of linear polyesters and vinyl monomers. Union Carbide & Carbon Corp.
- 765 501** Polymerisation of ethylene propylene and *isobutylene*. Ethyl Corp.
- 765 607** Preparation of benzelic acid esters. Farbwerke Hoechst AG.
- 765 715** Beater sizing of paper with keytene dimers. Hercules Powder Co.
- 765 608** Stabilised polychlorotrifluoroethylene resins. Union Carbide & Carbon Corp.
- 765 609** Preparation of esters of dicarboxylic acids. Goodyear Tire & Rubber Co.
- 765 779** Improving properties of mineral lubricating oils particularly. Shell Research Ltd.
- 765 505** Hydrocortisone derivative. Merck & Co., Inc.
- 765 506** Pyrimidine compounds. Badische Anilin- & Soda-Fabrik AG.
- 765 507** Thiomolybdate lubricants and their manufacture. Institut Francais du Petrole des Carburants et Lubrifiants.
- 765 296** Lubricants for extruding magnesium metal about a core wire. Dow Chemical Co.
- 765 509** Steroids and the preparation thereof. Pfizer & Co., Inc., C.
- 765 510** Amino-nitriles having therapeutic action. Kalichemie AG.
- 765 511** N-butyl-3-phenylsalicylamides. Cutter Laboratories.
- 765 385** Organo-siloxane compositions. Midland Silicones Ltd.
- 765 512** Method of separating tar acids and tar bases. Pittsburgh Consolidation Coal Co.
- 765 514** Purification of diphenylol propane. Koninklijke Zwavelzuur-Fabrieken Vorheen Ketjen NV.
- 765 515** Substituted tricyclohexyl borates. Borax Consolidated Ltd.
- 765 516** Unsaturated alcohols and esters and a process for preparation of same. Hoffmann-La Roche & Co. AG., F.
- 765 518** Desulphurising phenols contaminated by organic sulphur compounds. Naamlooze Vennootschap de Bataafsche Petroleum Maatschappij.
- 765 519** 2-(Carboxyalkylamino)- $\Delta^2-1$ , 3-diazacycloalkenes. Minister of National Defence of Canada.
- 765 520** Interpolymer compounds. Kodak Ltd.
- 765 246** Fractional distillation of petroleum hydrocarbon oils. Shell Research Ltd.
- 765 614** Fungicidal compositions. Monsanto Chemical Co.
- 765 522** Stabilisation of chlorohydrocarbons. Diamond Alkali Co.
- 765 615** Fluidised coking process. Esso Research & Engineering Co.
- 765 525** Process for the preparation of dialkyl monoalkali metal phosphates. Naamlooze Vennootschap de Bataafsche Petroleum Maatschappij.
- 765 526** Pure vitamins and analogs thereof. Lepetit Soc. Per-Azioni.
- 765 527** Fluorinated anisoles. Farbwerke Hoechst AG. Vorm Meister, Lucius, & Brüning.
- 765 528** Pentaerythritol chlorideacylates. Hercules Powder Co.
- 765 529** Tetrahydrofuran derivatives and conversion to pyridinediols. Aktieselskabet Sadolin & Holmblad.
- 765 531** 3,3-Bis(chloromethyl)cyclooxabutane. Deutsche Gold- und Silberscheideanstalt Vorm. Roessler.
- 765 261** Catalytic cracking of hydrocarbon oils. Naamlooze Vennootschap de Bataafsche Petroleum Maatschappij.
- 765 533** (Addition to 728 990.) Crystallisable pentaene aldehyde complexes. Eastman Kodak Co.
- 765 535** Polymerisation of ethylene. Naamlooze Vennootschap de Bataafsche Petroleum Maatschappij.
- 765 536** Organic carboxy-compounds. Celanese Corp. of America.
- 765 537** Production of vitamins of the B<sub>12</sub> group. Aschaffenburg Zellstoffwerke AG.

## SAFETY PIPETTE

### Range of Sizes Available

A PIPETTE which does away with the dangers inherent in mouth suction is made by W. G. Flaig & Sons Ltd., 39 Waterloo Road, London NW2. A greased glass barrel with a small aperture in the cap is fitted over the top of the pipette. To operate the pipette the barrel is raised with the finger over the aperture until liquid is drawn past the graduation mark. The finger is then removed and the barrel pushed back to its original position. Liquid is allowed to flow down to the graduation mark and the finger is then replaced. The pipette is allowed to deliver in the normal way by removing the finger. This type of pipette, known as the Exelo, is available in six different designs and in sizes ranging from 0.5 to 100 ml. and in a micro-range from 0.001 to 0.5 ml.

## Stanford Research Projects

THE November edition of *News Bulletin* published by the Stanford Research Institute, Menlo Park, California, US, reviews the history of SRI from the first tentative suggestions in 1939 to the present day. Today one half of the physical science division personnel of SRI are chemists. In addition there are chemists working in other sections of SRI. Among work carried out by staff chemists was the devising and construction of an automatic sampling and analytical device which continuously records the concentration of gaseous fluorides, even when they are present in only a few parts per billion parts of air. Another project is the conversion of solar energy into chemical energy through the production of hydrogen from water for fuel and other uses.

- 765 617** An azepine derivative. Hoffman-La Roche & Co. AG., F.
- 765 761** Filter for dust-contaminated gaseous medium. Aktiebolaget Svenska Fläktfabriken.
- 765 658** (Addition to 762 723.) 6-(1-aziridinyl) quinoline-diones-5, 8. Farbenfabriken Bayer AG.
- 765 281** Production of tetrachlorofurane. Badische Anilin- & Soda-Fabrik AG.
- 765 544** Amides and ketones having basic substituents. Hillel Remedy Factory Ltd.
- 765 718** (Addition to 741 630.) Production of metals by reduction of their halides. Deutsche Gold- und Silberscheideanstalt Vorm. Roessler.
- 765 282** Antibiotic products and their production. Chemie Grünenthal Ges.
- 765 725** 1-hydroxy-4-(2'-cyano) - ethylamino - anthraquinone. Sandoz Ltd.
- 765 284** Surface activation of passive polymers. Minnesota Mining & Manufacturing Co.
- 765 285** (Addition to 734 232.) Penicillin compound. Biochemie Ges.
- 765 547** Heterocyclic amines. Farbenfabriken Bayer AG.
- 765 636** Sulphur dyestuffs. Cassella Farbwerke Mainkur AG.
- 765 549** Spinning solutions of acrylonitriles polymers and copolymers and synthetic threads. Vereinigte Glanzstoff-Fabriken AG.
- 765 642** Silicone-alkyd resins. Midland Silicones Ltd.
- 765 728** 3- or 5-amino-1, 2, 4-triazoles. Farbenfabriken Bayer AG.

## Commercial Intelligence

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

### Mortgages & Charges

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

E. DU VERGIER & Co. LTD. Brimsdown, fertiliser manufacturers &c. 14 November, mortgage, to Martins Bank Ltd. securing all moneys due or to become due to the Bank; charged on land and factory premises at Essex Road, Hoddesdon, with plant, machinery &c. \*£2,000. 31 December 1955.

LEYLAND PAINT & VARNISH Co. LTD. 19 November, debenture to District Bank Ltd. securing all moneys due or to become due to the Bank; general charge. \*Nil. 1 May 1956.

ROLINX LTD. Wythenshawe, plastic manufacturers &c. 12 November, puisne mortgage securing to District Bank Ltd. all moneys due or to become due to the Bank; charged on leasehold land with factory and buildings at Harper Road, Wythenshawe. \*£15,923. 14 July 1955.

### New Registrations

#### Chemical Research (London) Ltd.

Private company (575,525). Registered 12 December. Capital £100 in £1 shares. Objects: To carry on the business of specialists in chemical research, manufacturers of, agents for

and dealers in cleaning materials, soaps, abrasives, powders, detergents etc. The permanent directors are: Eileen F. Pearce-Smith, 25 Sherrington Avenue, Hatch End, Mdx; Edward S. Smith, 10 Blenheim Road, N. Harrow, Mdx, director of Nicholls Adair Engineering Co. Ltd., and Lenpol Ltd.; and Alec J. Belither, 'Burleigh,' Easthorpe, Birch, Essex. Secretary: E. S. Smith. Reg. office: 5 Great Winchester Street, London EC2.

#### Scientific Installations Ltd.

Private company (575,421). Registered 10 December. Capital £4,000 in £1 shares. Objects: To carry on the business of consulting, mechanical and electrical engineers, tooling and chemical engineers etc. The subscribers (each with one share) are: Peter W. Brassington, B.Sc., A.M.I.E.E., and Mrs. M. V. Brassington, both of Summerfield, California Lane, Bushey Heath, Herts. The first directors are Adml. Sir Algernon U. Willis, G.C.B., etc., and Peter W. Brassington, B.Sc., A.M.I.E.E. Solicitors: Rollo Walker & Co., Bushey, Herts. Reg. office: Summerfield, California Lane, Bushey Heath, Herts.

### COMPANY NEWS

#### The Distillers Co. Ltd.

The board of The Distillers Co. Ltd. reports that the over-all results for the six months to 30 September show an encouraging increase in profits. In the main, this improvement is attributable to the increased volume of matured Scotch whisky which it has been possible to release. Despite disturbed conditions, common to most industries, the earnings of the industrial group have so far been well maintained. The board has declared an interim dividend on the ordinary capital for the year ending 31 March 1957, of four and four-fifths pence per share of 6s 8d (equivalent to six per cent), less income tax at 8s 6d per £.

#### ICI of Australia & N. Zealand

New sales and profit records were achieved in the year ended 30 September by Imperial Chemical Industries of Australia & New Zealand. Sales rose from £A.32.4 million to £36.7 million. The parent company's profit, subject to audit, totalled £1,940,760, an increase of £188,717 over the previous year.

### MARKET REPORTS

**LONDON** A steady business on home and export account has been reported from most sections of the industrial chemicals market. With the approach of the Christmas holiday, however, conditions generally are a little less active and there has been a seasonal falling off in contract deliveries. Hydrogen peroxide, borax and boric acid are items for which there has been a good call, but there has been no feature on the week. Zinc oxide is dearer and higher rates have been notified for a number of solvents; otherwise prices are unchanged with a firm undertone. Creosote oil, carbolic acid and cresylic acid are moving well in a steady coal-tar products market.

**MANCHESTER** Fairly steady trading conditions have been reported for heavy chemicals during the past week. Contract deliveries to the textile and allied trades and most other leading industrial consumers are keeping up reasonably well, though so far as additional enquiries and actual fresh business are concerned there are already signs of the usual year-end influences at work. Apart from a few sections of the fertiliser trade, trading conditions at the moment are on the quiet side. A steady demand for most tar products is reported.

**GLASGOW** A reasonably busy week has been experienced in most sections of the heavy chemical industry, and a varied range of chemicals have been demanded. Enquiries and the placing of contracts for next year's supplies are now in evidence. Prices generally have been firming. Some increases have been reported, due mostly to the increase in carriage charges, which have taken place.

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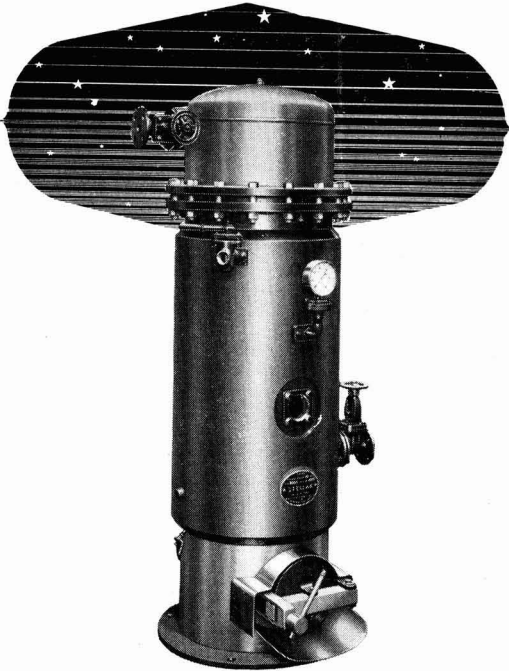
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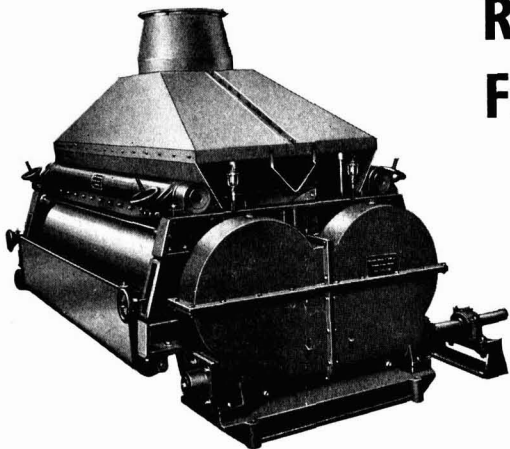
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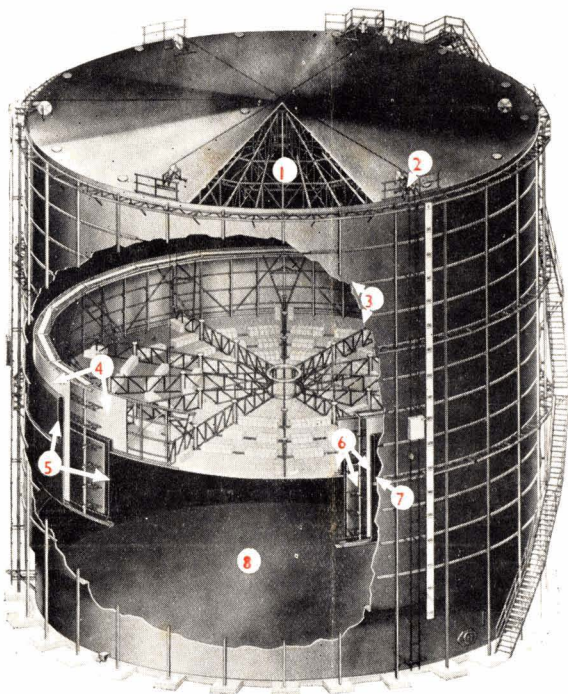
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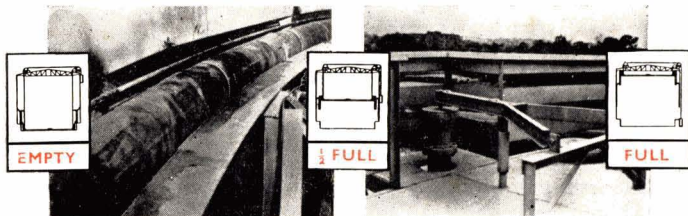
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