

Journal of

# SCIENTIFIC AND INDUSTRIAL RESEARCH



[Vol. VI]

AUGUST 1947

[No. 8]



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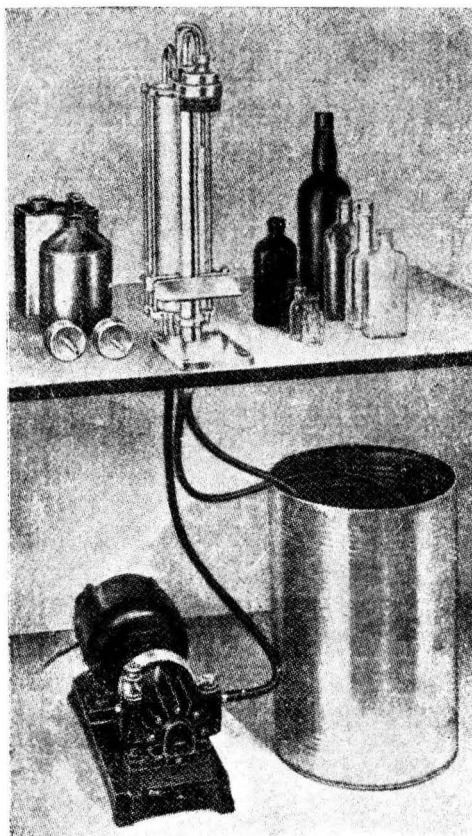
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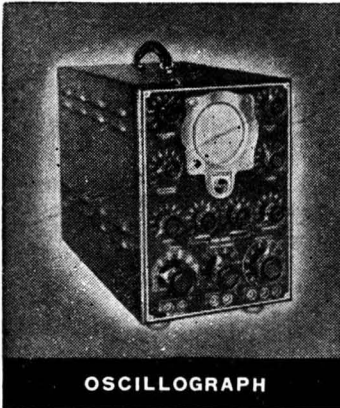
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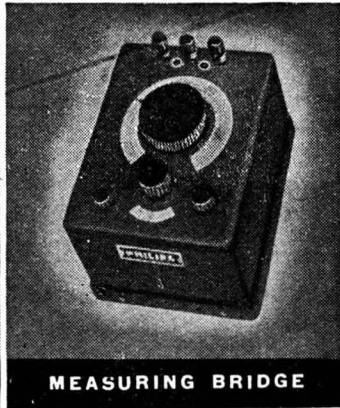


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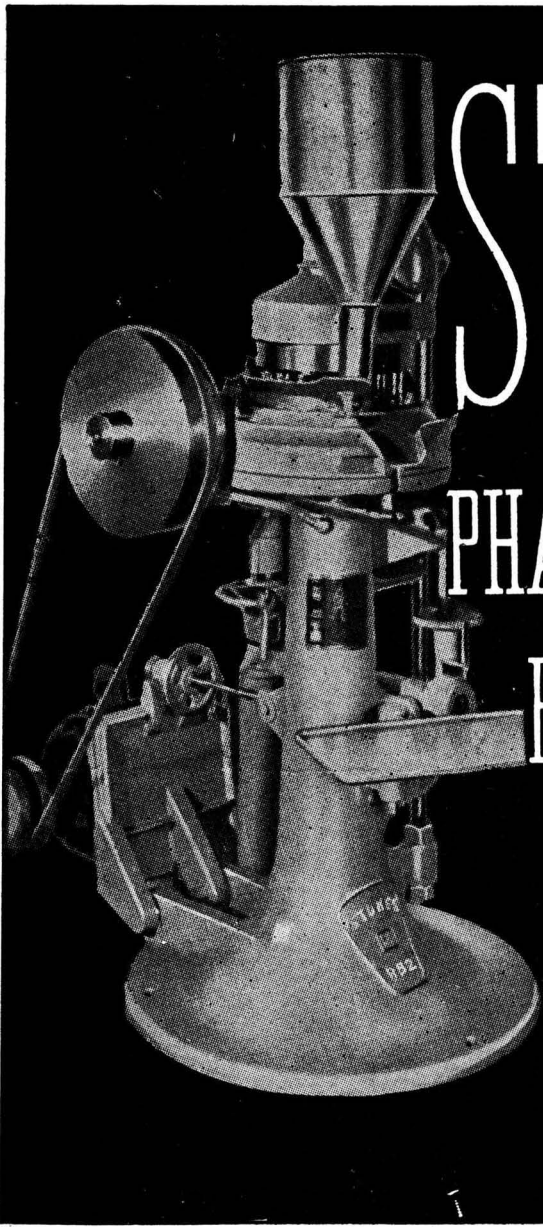
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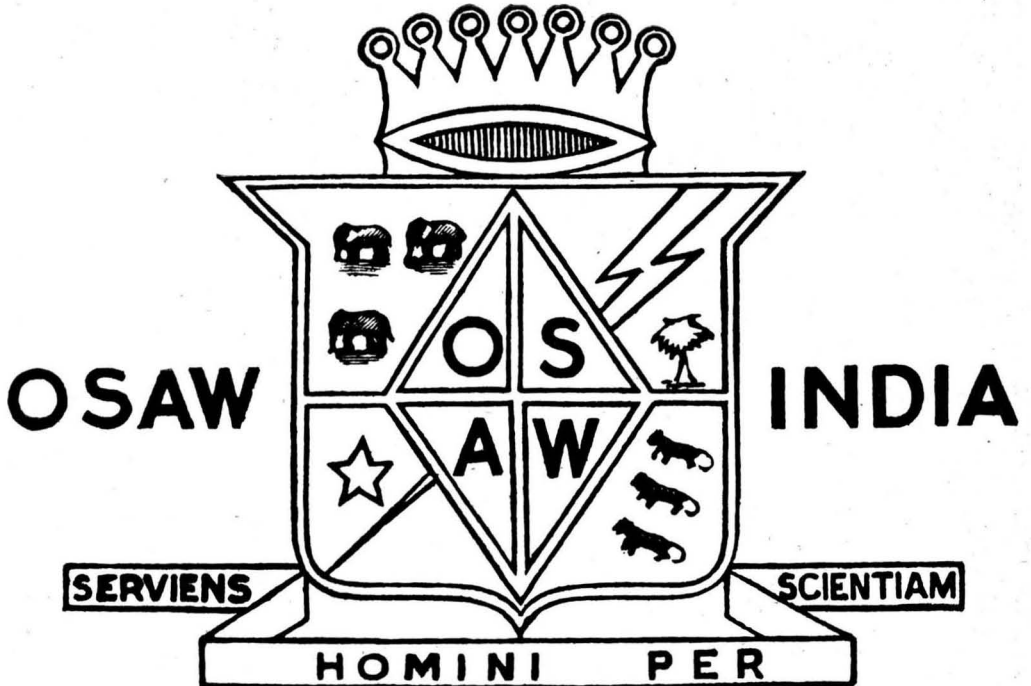
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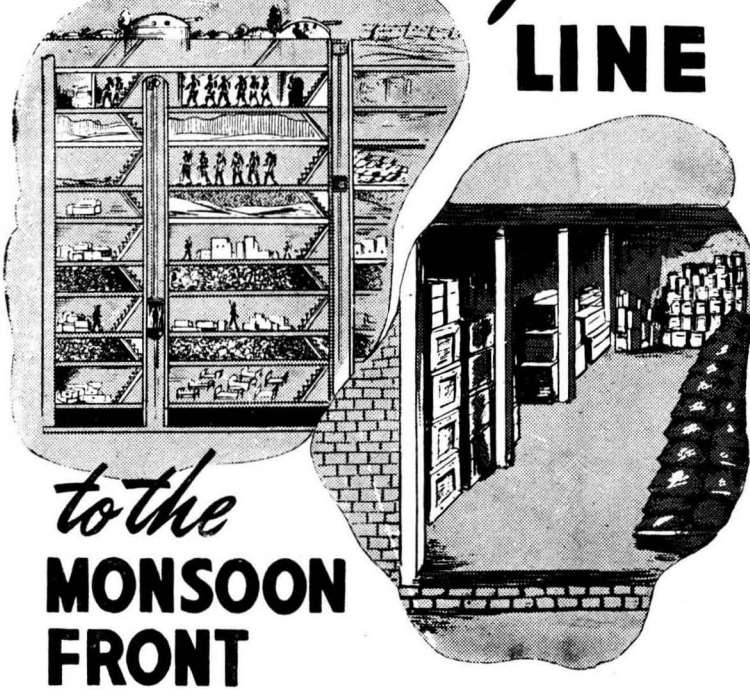
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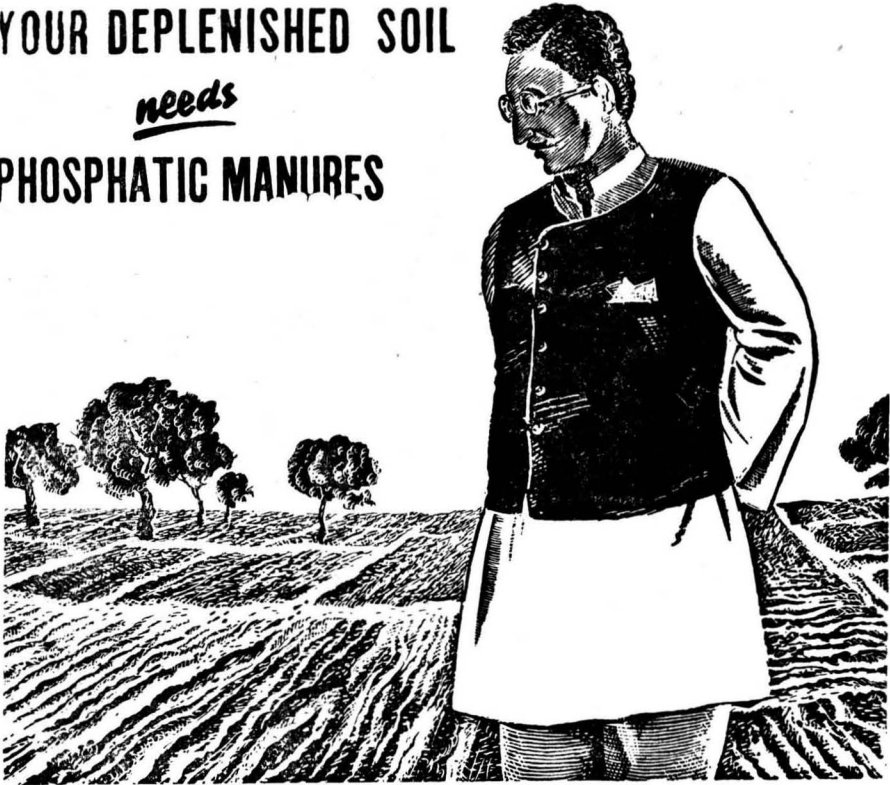
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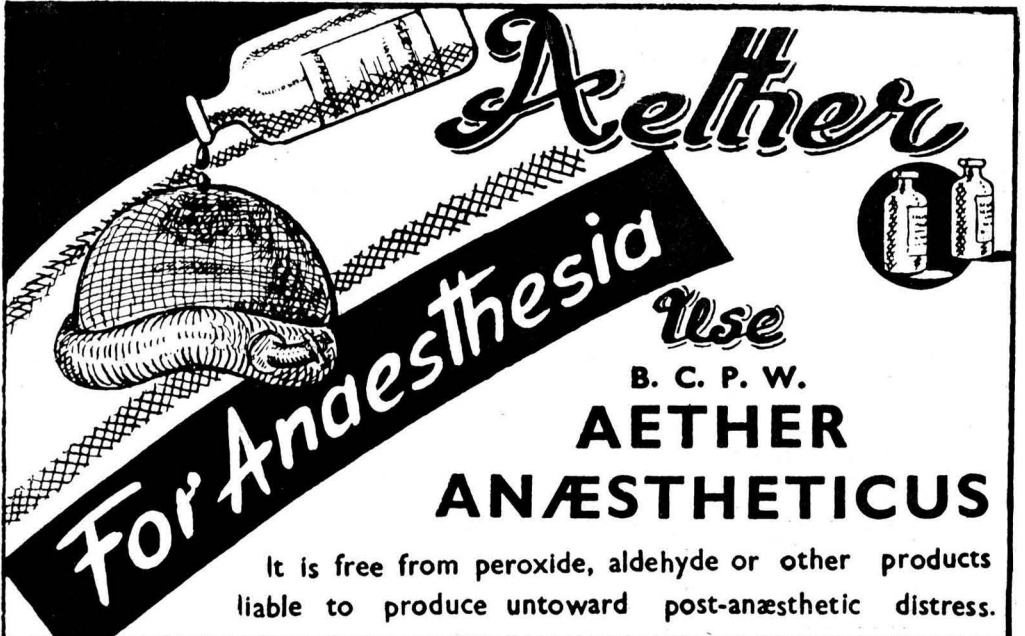
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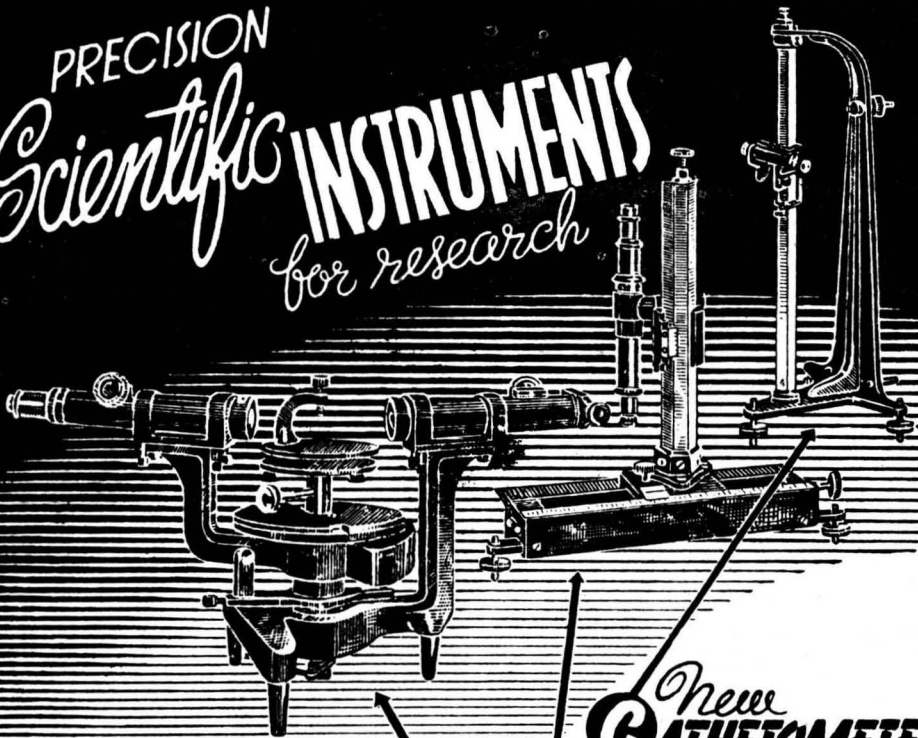
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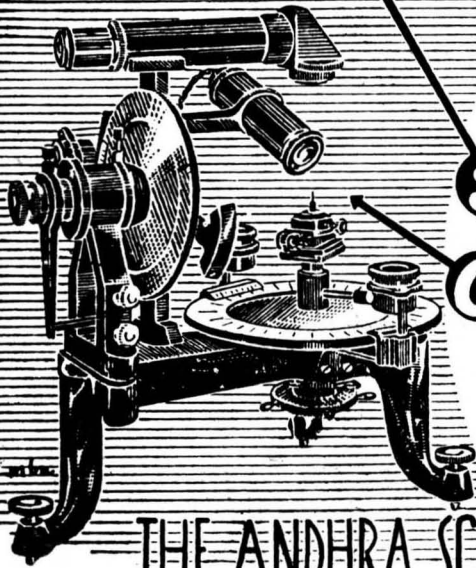


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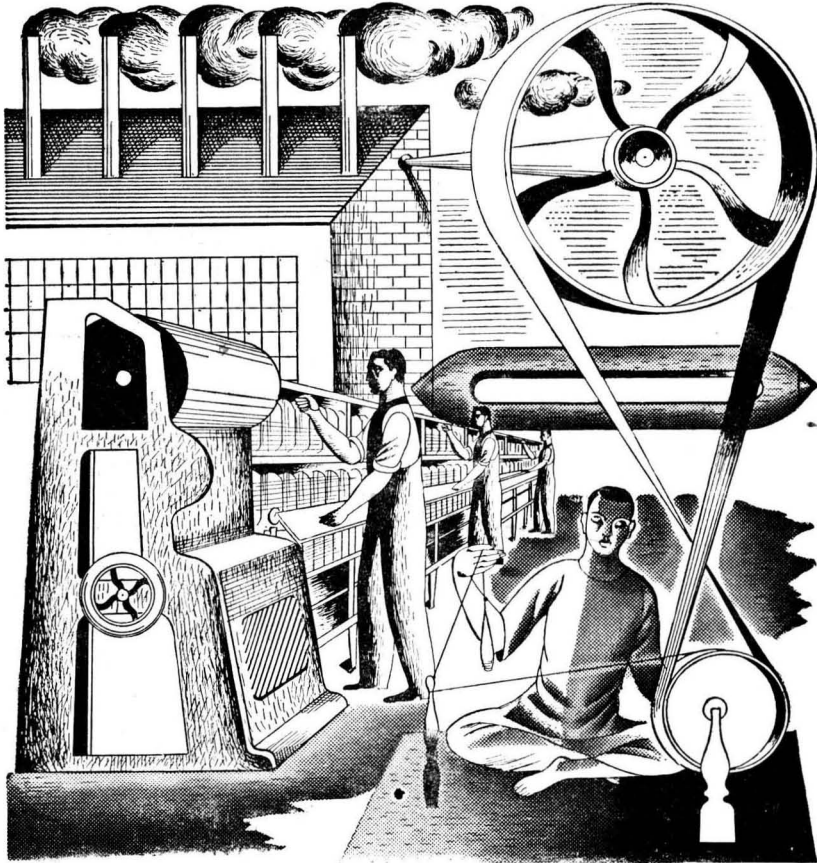
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## A Great Project

THE project report on Hirakund Dam issued by the Central Waterways, Irrigation and Navigation Committee (C.W.I.N.C.), a copy of which we have received, is a masterly document on a great project which will bring almost incalculable benefit to the people of Orissa. The credit for marshalling together the many details germane to the launching of an engineering project of a stupendous magnitude goes to the indefatigable chairman of the C.W.I.N.C., who is in fact, the author of the project and its begetter. The Mahanadi Development Plan (and the Hirakund Dam project which is the first stage in the development of the Mahanadi river basin) is the outcome of a long and detailed study, by a succession of able engineers, of the flood problem of Orissa—a problem which has been aptly described as a spectre haunting the Province. Starting from the first serious attempt of Sir Arthur Cotton in 1858, the flood problem of the deltaic regions of Orissa has been the subject of study by a series of committees upto 1945, when it was finally referred to the Government of India. These committees generally, took the view that all that was necessary to control the periodical floods was to let the water run into the sea in an orderly flow. The correct approach to the problem was taken by Mr. A. N. Khosla, who took up the investigation at the instance of the Government of India. As a result of local inspection, study of the previous reports and discussions with provincial experts, he came to the conclusion that the

only solution of the problem of floods, and of the many problems of Orissa generally, lay in the control, conservation and utilization of the enormous water wealth of the rivers traversing the Province by means of storage dams. Mr. Khosla proposed that the development of the Mahanadi river basin should be first investigated, and the studies which have since been carried out have confirmed the essential soundness of Mr. Khosla's view. A formal beginning of the project was made on March 15, 1946, when the Governor of Orissa laid the foundation stone of the Hirakund Dam.

To appreciate the magnitude of the project, it is necessary to point out that the Mahanadi river is the biggest among the Orissa rivers taking its origin in the hilly tracts of Central Provinces. It has a total length of 533 miles and a catchment area of 51,000 square miles with an average rainfall of 53.17 inches. The maximum flow discharge is estimated as 1.6 million cubic feet per second, which dwindles to 2,500 cubic feet per second during the dry months. The mean annual run off of the river at Naraj is 74 million acre feet which is more than twice the capacity of the biggest man-made lake in the world,—Lake Mead formed by the famous Boulder Dam in U.S.A. (capacity 32 million acre feet).

What adds importance to the project as conceived by Mr. Khosla is that it provides the key to the problem of Orissa, which is not

merely one of floods and droughts; it is the problem of undeveloped resources and lack of internal communications with the result that the Province as a whole remains undeveloped and in consequence poor and backward. Due to the maldistribution of rainfall in the river basins, floods and droughts are of frequent occurrence; only one food crop is raised in the year, and that too a precarious one. Malaria and other 'low in come diseases' are widespread. The enormous mineral wealth of the province,— coal, iron, chrome, graphite, bauxite, limestone and mica, to mention a few,— lies buried, unexploited and unutilized for the most part. Communication are few, and the vast hinterland is unconnected, either by rail or by canal, with the sea coast. The history of Orissa reveals that the Province has not witnessed any great constructional development during the past four centuries or more; its natural resources lie static and the people suffer from poverty and ill health. It was John Ruskin that wrote :

“Whenever you see want or misery or degradation then be sure either industry has been wanting, or industry has been in error. It is not accident, it is not Heaven—commanded calamity; it is not the original and inevitable evil of man's nature which fill your streets with lamentation and your graves with prey. It is only that, where there should have been providence, there has been waste; where there should have been labour, there has been lasciviousness; and wilfulness where there should have been subordination.”

The solution for stricken Orissa, and this is provided by Mr. Khosla's plan, lies in a determined effort to develop irrigation, power and navigation facilities through planned multi-purpose development of the enormous water wealth of the Province. Priority should be given to Mahanadi, the biggest among Orissa's rivers, which offers possibilities of giving immediate benefits to the people.

The Mahanadi development plan involves the construction of 3 dams at Hirakund, Tikarpara and Naraj. The first of them, the Hirakund dam, is the uppermost of the 3 component units and is expected to yield early results. Each one of the units admits of independent and stage development without in any way interfering with the development of the Mahanadi valley as a whole. The Hira-

kund dam, when completed, will rise 100 feet above the bed rock. The area submerged by the impounded water will cover 130 sq. miles, a good portion of which will be available for cultivation when the reservoir gets depleted during the dry months. The reservoir will have a capacity of four million acre feet, of which nearly half will be available for irrigation and power development. The storage will be sufficient to generate 50,000 KW. of hydro-electric power and the canal system taking off for the reservoir will provide perennial irrigation for nearly 800,000 acres. It will permit navigation to be developed from the port of Chandbali to Dholpur, a distance of 220 miles. The project will solve a fair portion of the flood problem of Orissa, besides offering recreation facilities and permitting the development of fisheries, reclamation of water-logged areas and effective control of malaria. It is expected to take 5 years for completion and the estimated cost is Rs. 47.81 crores.

The immediate execution of a plan with such great possibilities for the people of Orissa should be considered mandatory. Geological, hydrological, economic and other surveys show that the project is technically sound and financially feasible. The report has discussed the problem of resettlement of dispossessed people and suggests alternative sites, not far from the submerged area, which can be developed to combine the benefits of agriculture and industry. Economic surveys have revealed the possibilities for the establishment of iron and steel, cement, caustic soda and bleaching powder, fertilizer, sugar and textile industries in the region. The forest wealth of the river valley comprising sal, teak and bamboo forests can be utilized for developing lumber, plywood and paper industries. These developments will provide an outlet for the electrical power generated at the dam sites and the water ways will facilitate the transport of raw materials and finished products.

The project report should be studied as a whole to appreciate its many excellent features. It should serve as a model for the many projects of regional development which are awaiting investigation in many parts of India. The report rightly points out that the Mahanadi project should be planned, undertaken and executed by one single authority. The States and Provinces concerned in the develop-

ment of the river basin, should co-operate in the interests of the people and a co-ordinating authority, on the model of the Damodar Valley Corporation which was recently set up for the development of the Damodar river basin, should be appointed to take charge of the entire project. An equally important requirement for the success of the project, to which we have drawn attention while writing on the Damodar Valley Corporation, is to enlist the

enthusiasm, co-operation and active participation of the people, who should be encouraged to look upon it as their project. We trust that a determined effort would be made to implement Mr. Khosla's plan which promises immense industrial and agricultural developments and the economic metamorphosis of the Orissa Province, and that it would be taken up as an immediate programme rather than as an ultimatum aim.

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## Requirements for the Expansion of the Paper and Board Industry in India

By M. P. BHARGAVA

(Forest Research Institute, Dehra Dun)

**D**URING the last ten years the Paper and Board Industries have made more rapid strides than in any previous decade. Protection granted to the industry by the *Bamboo Paper Industry (Protection) Act*, passed in 1925, helped to resuscitate the struggling industry and to put it on the road to steady progress. A number of new mills came into existence by 1940. The practical cessation of imports of papers and boards during World War II and the necessity of supplying the greatly increased demands of the Government and Defence Departments gave a fillip to production. In 1937-38, the indigenous production of papers and straw-boards was 53,811 tons. During 1946, the actual production was as follows:

	<i>Tons</i>
(1) Writings and Printings ..	65,139
(2) Wrappings and Imitation Kraft ..	15,809
(3) Other varieties ..	6,825
(4) Boards ..	18,480
(5) Straw-boards ..	14,504
Total ..	120,757

The production covers a wide range of papers and boards including tissue, air-mail, banks, bonds, ledgers, cartridges, duplex and triplex boards, etc. The quality of several of these products compare favourably with those

of the imported ones. The production also includes certain lines, e.g. kraft, tissue, duplex and triplex boards, which were not manufactured before the War.

The aggregate potential capacity of the existing mills is estimated to be about 130,000 tons per annum. This capacity would have been further increased had it not been for the difficulties of procuring the required machinery and equipment. A number of new ventures have been projected and it is hoped that these will materialise as soon as imports of plants from abroad are possible.

The Paper Panel, appointed by the Government of India to report on the development of the paper and board industries in the country, anticipate a considerable increase in the consumption of papers and boards as a result of the development of commerce and industry and of the gradual introduction of mass education in the country. According to the Paper Panel the annual consumption of all kinds of papers and boards, including newsprint, cheap packing and printing papers and insulation, pressed and jointing boards etc., is anticipated to be about 355,000 tons in 1951-52 and 540,000 tons in 1956-57. The targets for indigenous production recommended by the Panel are 264,000 tons in 1951-52 and



471,000 tons in 1956-57. Supplies of raw and auxiliary materials (chemicals, coal, etc.,) in adequate quantities, transport facilities, procurement of the required capital goods, technical personnel and lastly research are some of the important problems which require to be tackled urgently for the achievement of the above targets.

### Raw Materials

Based on certain assumptions with regard to the furnish of papers and percentage yields of pulps, the quantities of various raw materials, required per annum for the 1951-52 and 1956-57 targets respectively, are as follows:

	1951-52 (tons)	1956-57 (tons)
Bamboo	370,000	625,000
Sabai grass	90,000	160,000
Rags	45,000	75,000
Others	80,000	140,000
Cereal straw	80,000	140,000
Soft woods	60,000	100,000
<b>Total</b>	<b>725,000</b>	<b>1240,000</b>

Surveys of bamboo and grass areas carried out in the past show that about 730,000 tons of bamboo could be made available per annum in different Provinces and Indian States, and about 60,000 tons of *Sabai* grass per annum in the United Provinces, Punjab, Behar and Orissa. Large quantities of the grass are available in Nepal also. A number of other grasses, the paper making suitability of which has been tested at the Forest Research Institute, though inferior to *Sabai* grass, are found in different parts of the country. It is estimated that about 120,000 tons of these grasses can be exploited per annum.

Bamboos and grasses can be supplemented by a number of other raw materials, e.g. cotton rags, flax, hemp, and jute wastes and reclaimed papers. Collection of these very useful waste materials is still largely unorganised. More than 40,000 tons of these materials were consumed by the industry in 1944. In addition, there is the possibility of utilising cotton linters, surplus stocks of short staple cotton and bagasse (crushed sugar-cane). It is estimated that about 13,000 tons of cotton linters can be made available per annum and about two million tons of bagasse could be had provided the sugar mills are given coal in exchange, since a large number of sugar mills use bagasse as fuel for steam and power

generation. Straws and stalks of the numerous agricultural crops, which are grown over an enormous acreage, are also very large potential sources of cellulose.

The estimates given above for the various raw materials are very rough indeed being based on surveys carried out years ago. Moreover, during the last War certain bamboo areas have been extensively exploited. In order to provide for a planned development of the industry, it is essential that fresh and systematic surveys of all the important paper making materials should be carried out. The Government of India have already addressed the Provincial Governments and the Indian States in the matter. The survey would be useful in helping to regionalise the location of new factories so as to avoid their concentration in certain localities, leading to uneconomic competition for raw material supplies. According to old surveys, about 158,000 tons of bamboos in the Bombay Presidency and about 140,000 tons in the Madras Presidency should be available per annum. At present there is not a single mill in the Bombay Presidency using bamboo, and in the Madras Presidency there is one small mill using bamboo for paper manufacture. Obviously, therefore, Bombay and Madras Presidencies offer good prospects, from the raw material supply point of view, for putting up new mills.

The manufacture of newsprint would require in 1951-52 and 1956-57 respectively about 60,000—100,000 tons of soft woods largely for the production of mechanical pulp. Conifers, spruce (*Picea smithiana*) and fir (*Abies pindrow*) are admittedly the best raw materials for the purpose. Large supplies of these conifers exist in the Himalayan regions but the high altitudes at which they occur, the extremely rugged and broken nature of the mountainous tracts and lack of suitable floating streams have, hitherto, rendered impossible their exploitation for the newsprint industry. Recent advances in forestry and mechanical engineering may provide a solution either for the transport of woods as logs or of pulp in slush form, through pipe lines, after grinding wood in small units at suitable places in the hills. Surveys and preparation of forest working plans for the exploitation of stands of conifers in the hills are also called for.

In case the exploitation of conifers for the newsprint industry is not found feasible and

economic, attention will have to be concentrated on the utilisation of soft woods of the broad-leaved species found in the plains. Investigations in this direction have been carried out at the Forest Research Institute and are still in progress. The object of these investigations is to select a few species suitable for the purpose, so that plantations may be raised on short rotation basis and supplies made available to the industry in the near future. There is not a single species in any part of India, suitable for mechanical pulp production, which is available in quantities sufficient to warrant the establishment of a newsprint mill.

However, as mentioned above, surveys carried out in the past indicate the existence in the country of raw materials enough to support the growth of the industry for the next few years. For further development, it is obvious that it would be necessary to raise plantations of bamboos, grasses and soft woods.

#### **Auxiliary Materials**

The total requirements of chemicals for the expansion of the industry are estimated to be about 250,000 tons in 1951-52 and about 400,000 tons in 1956-57. Coal requirements for steam and power purposes would also be in the neighbourhood of 1 million and 1½ million tons in 1951-52 and 1956-57 respectively. Manufacture of heavy and other chemicals occupies an important place in the post-war development schemes for chemical industries. Increased raisings of coal and hydro-electric power developments are also being actively sponsored by the Indian Government.

#### **Transport Facilities**

The expansion of the paper and board industries in the next decade would involve haulage and transport of about 3-4 million tons of materials. This would necessitate development on a large scale of waterways, roads, railways and introduction of mechanised means of transport. Various projects for these developments are being matured by the Central as well as the Provincial Governments.

#### **Capital Goods**

It is estimated that about 8 crores of rupees have already been invested in the industry. Replacements and renovations of the worn-out machinery and establishment of new mills are estimated to require a further investment of about ten crores of rupees. It is hoped that normal conditions would soon be restored to enable the industry to import the required plant and machinery from abroad in the near future. The Paper Panel have recommended the encouragement of workshops for the fabrication of certain spare parts and accessories required by the industry.

#### **Technical Personnel**

At present there is a great dearth of properly qualified and trained Indians to man the existing mills and those projected for the future. Plans for imparting advanced technical training at the Forest Research Institute, Dehra Dun, are under way, and are expected to materialise in the near future. The Government of India have sent a few young men for training abroad.

#### **Research**

Research alone, both of a fundamental and applied character, can sustain and provide the required nourishment for the healthy growth of any industry. The paper and board industries are highly specialised industries and depend solely on research for their development. A scheme for expanding the research activities of the Paper Pulp Section Forest Research Institute, Dehra Dun, has been prepared. The Officer-in-Charge of the Section is proceeding abroad on deputation to U.K., Sweden, Germany, Canada and the U.S.A to acquaint himself with the latest developments in cellulose, pulp and paper technology. It is hoped that, on his return, early measures will be taken to implement the finalised scheme of research.

The Cellulose Research Committee of the Board of Scientific and Industrial Research is also actively fostering research on problems connected with the cellulose and paper industries.

# Recent developments in Tin and Tin Alloy Coatings

By JOHN IRELAND

THE industrial use of electro-plated tin coatings is a development of the past decade in which British scientists have played an important part. In particular the *Tin Research Institute* has developed two types of bath, and has worked out methods by which the baths can be controlled and maintained in such conditions that smooth, continuous, tightly-adherent coatings are formed which are totally different from the coatings obtained by the earlier processes. In the first years of the war effort about one hundred new installations for electroplating were made in England and many other installations have been made since.

The most interesting property of the electroplating process is that, by its means, coatings of more than one metal can be applied, or even alloy coatings can be formed which could not be got by the old established hot-dipping process used for the production of coatings of pure tin. In America the tendency has been to obtain the effect of alloy coatings by applying two or more thin coatings of different metals successively, and then using a heat treatment to procure some form of alloy by diffusion. In England the art of plating alloys direct in one operation has made great progress. At least two new alloy coatings have been put on to a commercial basis.

## Speculum

The first and most striking alloy consists approximately of 45 percent tin and 55 percent copper. Its properties are however quite different from either of the constituent metals. In appearance the coating is almost indistinguishable from silver, its hardness is intermediate between that of nickel and chromium and it has a high resistance of corrosion and tarnish. The alloy has been called "speculum" because its composition and qualities are similar to the bronze mirrors made and used in the Mediterranean civilisations more than 2,000 years ago. Silver was also employed but, al-

though it has a higher initial reflectivity, it becomes tarnished and scratched rather rapidly.

From the users point of view the warm colour of speculum is more attractive than the rather bluish tint of chromium. And where used for interior metal trim, as for instance in restaurants and bars, it permits a wider choice of decorative colour schemes. For jewelry and tableware speculum is as attractive as silver and has the merit of not requiring so frequent cleaning. Speculum spoons and forks have been in use for several years in hotels and in private houses and have remained perfectly bright with only the usual washing. The hardness of the coating has shown a very satisfactory resistance to mechanical damage and scratching.

Speculum coatings are also outstandingly satisfactory for bathroom metal work. Wash basins with speculum plated taps have been in constant use for seven years and are still in perfect condition. Indeed, for all interior metal work speculum has many advantages. It is however not advised to use speculum for out-of-doors metal work or for automobile trim.

*Cost Aspects.*—The cost aspects of speculum are attractive. As it is applied in a single plating operation it offers economies as compared with nickel + chromium not only in the matter of labour charges, but also in capital expenditure tied up in plant and floor space needed. In addition, both the initial preparation of articles and the final polishing are less exacting in the case of speculum. Satisfactory finishes have in fact been obtained on spinings and pressings without any preparatory polishing, and brass castings are stated to require only half the preparation usual for nickel chrome. The throwing power of this bath is exceptionally high which greatly simplifies the plating of fabricated articles.

It should be noted that the process of speculum plating is covered by patents in some countries. This arises because the plating solutions used are in part covered by earlier American patents designed for the production of red bronze, (that is alloys of copper and tin containing up to about 10 per cent. tin). The modern bath is easy to operate, although competent chemical control is necessary to maintain correct conditions of deposition. The procedures are however fully worked out and have proved well within the capacity of the normal works control laboratory.

The electro-plating bath employed is based on a combination of two widely used plating solutions; the sodium stannate bath and the copper cyanide bath. The composition of the alloy deposit is controlled by maintaining the cyanide and free caustic soda content of the bath at the correct figures. Separate anodes of copper and of tin are used to replenish the bath as the alloy is so resistant to attack that alloy anodes would not dissolve.

Speculum plating was first adopted commercially in England, in 1939. Its use was of course suspended during the war period, but in the past year a number of plants have been put into operation, and the work being done includes tableware and cutlery, domestic irons, shop fittings, cafeteria urns, cigarette cases, boxes, lighters, toilet fixtures, metal furniture, car headlight reflectors and a large assortment of renovations to pewter, Sheffield plate, wheel hub cover and perambulators, toys, etc. Under present conditions the output of these plants is not yet large, but several firms have extended their plant after the first few months of successful operation, which gives reason for believing that this new finish will take a substantial and permanent place in industry.

A new alloy coating has recently been devised in the laboratories of the *Tin Research Institute, London*, to protect steel from rusting. This consists of an electro-deposited alloy of tin and zinc. The use of zinc coating or galvanizing, and of tinning are very widespread. Each gives good service under certain conditions, but there are many purposes for which the new coating appears to be greatly superior to either of the constituent metals used alone.

A zinc coating protects the underlying metal from rusting by a kind of sacrificial action, and the protection is excellent as long as the zinc lasts. But zinc is itself a fairly active metal, and it corrodes with comparative rapidity. Tin, on the other hand, has a high intrinsic resistance to corrosion, but it has no sacrificial action. The tin coatings used in industry are usually very thin, and the underlying iron is liable to be attacked at pores or at points of mechanical damage.

The new alloy coating appears to combine the advantages of both metals. It is able to protect the steel even at exposed points, and yet the coating offers a high intrinsic resistance and in consequence lasts much longer.

Some idea of the protective qualities and intrinsic resistance to corrosion of these alloys is illustrated in Fig. 1. This contrasts the result of corrosive attack of warm salt spray on steel sheets covered with tin-zinc alloy and with tin and zinc used alone. There is comparatively little attack on the top row of alloy coated specimens, and only the thinnest coatings have begun to break down. The second row of tin coated specimens show little attack on the tin, but severe rusting. The two lower rows have pure zinc coatings which have been severely attacked. They are covered with a thick deposit of whitish corrosion product, and patches of rust are already apparent. An examination of the actual specimens shows that as great protection is provided by a thin coating of tin-zinc alloy as by a much thicker coating of zinc alone.

Similar tests carried out over many months show equally favourable results when the specimens are exposed to tropical conditions and to impure urban atmospheres.

The tin-zinc coating is sufficiently ductile to withstand considerable deformation without failure. This is exemplified in Fig. 2 which shows mild steel specimens which have been "cupped" after coating, and have then been exposed to salt spray tests. The tin-zinc alloy shows no corrosion, the tin and cadmium coatings are partially injured and rust spots are visible, whilst the zinc coating is heavily corroded.

This alloy coating is applied electrolytically. The solution used is a combination of the well-known sodium stannate and zinc cyanide



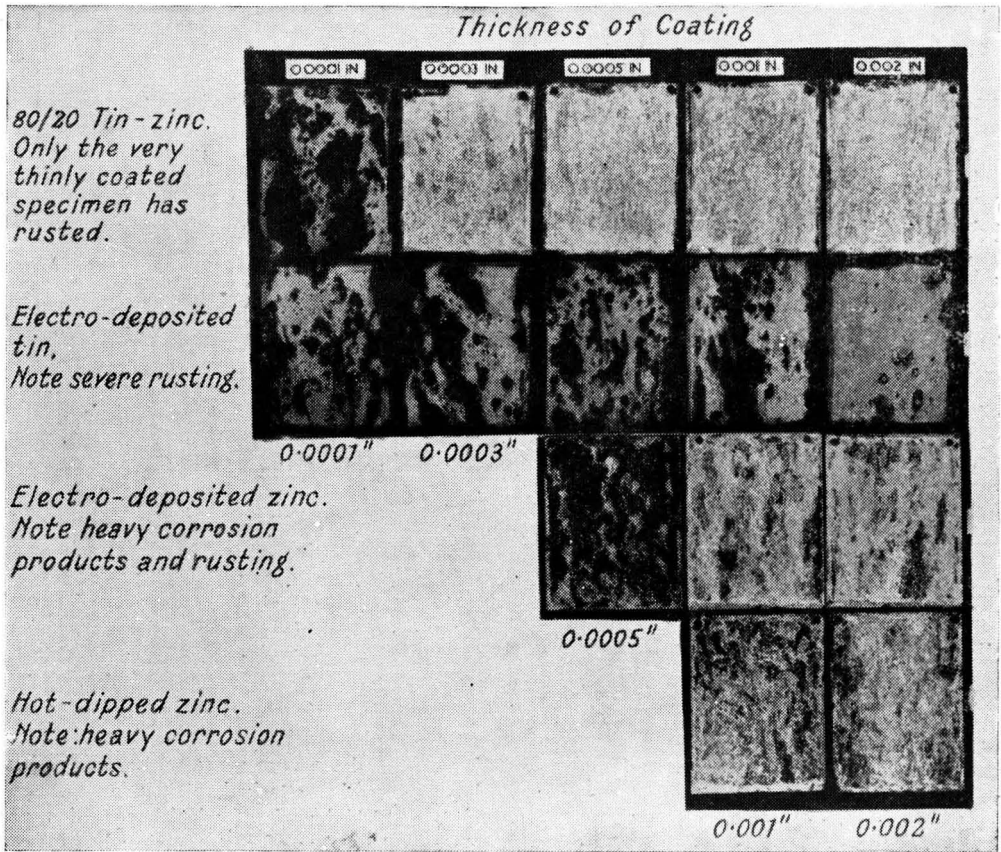


Fig. 1.—Corrosion of various coatings after exposure to salt spray tests for 672 hours.

baths. It is exceedingly simple to operate and although the bath composition must be controlled within limits, there is a fairly wide latitude, and the chemical checks are easy and not too frequent. Alloy anodes are used to replenish the bath. The following conditions are recommended:—

	50% tin 50% zinc plate
Tin content of bath	30 gm. per litre
Zinc " " "	8 " " "
Free sodium hydroxide	6-8 " " "
Sodium cyanide	50 " " "
Cathode current density	15-30 amp/sq. ft.
Temperature	70° C.
Anodes	50% tin 50% zinc.

The deposit, although not recommended as an ornamental finish, is of a pleasing white appearance and polishes with extreme ease to a silvery colour which is maintained for a considerable time. The alloy coating is easy to

solder. For this reason it has been welcomed by manufacturers of radios for the steel frame on which their sets are assembled. These frames are frequently spot welded into box-like structures and then numerous soldered connections are made to "earth" various circuits. Cadmium is protective and solderable, but gives off poisonous fumes when spot welded. Zinc is protective, but soldering is difficult and unreliable. Tin-zinc alloy combines greater protection with solderability in a higher measure than any other known coating.

It is, however, as a protective coating that its chief market is envisaged. It has been very carefully tested by manufacturers of steel window frames, electric water heaters, aero-engines, electrical instruments, etc., in comparison with other industrial protective coatings and in every case it has given the highest per-



formance. Articles of domestic hardware, flyscreen, rabbit wire, fencing and electric power-line hardware, would all be worth the extra protection and longer life that the new coatings provide. It is also likely that many applications will be found on yachts and sea-going ships of all sizes as this alloy is outstanding resistant to maritime conditions.

This alloy coating is only at the beginning of its career, but without doubt as it becomes known, its use will expand rapidly.

### Protective Coatings

The problem of preventing the destruction of steel is an exceedingly important one which has been studied from many angles. The use of thin sheet steel and of light steel structures is increasing both in industrial and in domestic equipment. But the advantage of the low first cost of steel can be fully exploited only if manufacturers can guarantee a satisfactory length of life for their products.

The usual protective treatment is based in applying a coating of paint, lacquer or varnish, and great skill has been expended on improving the quality of those coatings. Unfortunately, they all meet a common end.

Rusting occurs below the paint and before long the paint flakes off leaving unsightly areas over which corrosion proceeds rather rapidly.

It has recently been established by the *Tin Research Institute* in London, that if steel be given a very thin coating of tin before the paint is applied, the resistance to destruction of the paint is greatly increased and the life of the steel is multiplied several times.

This effect, though novel, is not surprising when one recollects that tin has a high inherent resistance to corrosion and a strong affinity for oils. The peculiar ability of tin to retain an oil film is, for example, an important factor in the use of tin in bearings. Printing in colour on tinplate has long been an art that rivals the best that can be done on paper. Tinplate is also coated with lacquers for packaging certain foodstuffs, and lacquered tinplate can afterwards be fabricated into containers without spoiling the bond between the tin and the lacquer. It might be agreed therefore, that a thin coating of tin on steel would improve the adhesion of paints to steel and thus increase the useful life of the product. There is, however, evidence that the presence

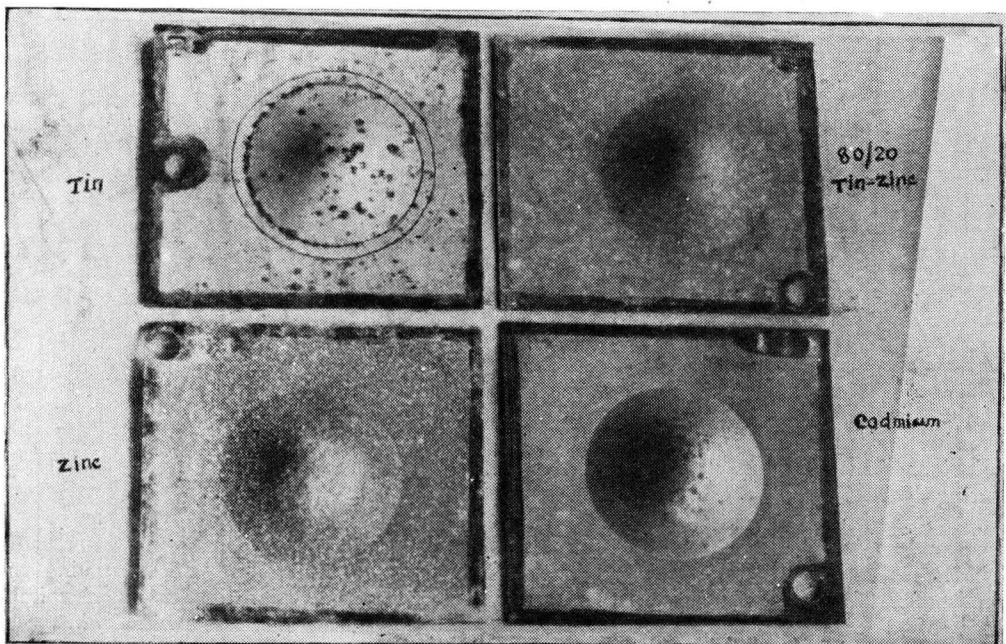


Fig. 2.—Corrosion on steel specimens deformed after plating (Thickness of film, 0.0007" in each case) and exposed to a humid atmosphere for 1,430 hours.



Fig. 3.—Cold rolled and pack rolled steel plates painted after various pretreatments and exposed for 1,500 hours to accelerated weathering tests. The lower set of photographs were exposed for 1,500 hours to accelerated weathering tests. The lower set of photographs were taken after stripping the point and removing the phosphated coating to exhibit the extent of rusting.

of the thin tin film improves corrosion resistance independently of the favourable effect on adhesion. This point has been brought out during the war in the use of very thinly coated tinplate. It has been impossible to produce a lacquered finish for non-tinned steel sheet which provides a satisfactory container for wet foodstuffs. If, however, a thin tin coating is used of 8 ozs. per basis box, or 0.00003" (0.00077 mm.), greatly increased resistance is obtained.

With these facts in mind, a joint research

was undertaken by the *Tin Research Institute* and the *Paint Research Station*, to ascertain the degree to which tin coatings can improve the corrosion resistance of painted steel. Some hundreds of specimen were made and tested. These included different types of steel, different thicknesses of tin coating and for comparison untinned steel and chemically phosphated steel. Twelve typical paints used in industry, including linseed oil and synthetic paints, cellulose, and air and stove drying enamels were used. After painting, and before

exposure to corrosion, each specimen was given a deep scratch so that behaviour after mechanical damage could be assessed.

The painted specimens were tested in a variety of ways which experience has shown are representative of severe service. Some sets have been exposed to natural weathering for over three years. Others have been given a variety of accelerated weathering tests including intermittent exposure to strong electric light, rain water, and/or salt spray. The appearance of the specimens after these tests depended on the nature and duration of the exposure and on the quantity and quality of the paint used, but the relative performance of the different pretreatments agreed closely throughout. In each case where paint had been applied direct to steel, the corrosion has been severe. The specimens phosphated before painting were much better and had held paint well, but rust spots had developed. (Fig 3).

Those specimens which had been tinned before painting were outstandingly good. The paints had adhered closely, and little or no rust had formed. It was also established that an exceedingly small amount of tin was required if the coatings were applied by electrolytic plating. 1 lb. of tin is sufficient to cover 900 sq. ft.

The value of this treatment may be appreciated in the case of an automobile body which

could be coated both inside and out with tin costing less than 2 sh. As a result the body and mudguards would last several times as long without damage to its paint by corrosion.

In another instance where steel sheets are shipped for fabrication overseas, the cost of the thin grease coating now used to prevent rusting before manufacture is greater than the cost of a tin coating which would give greater protection. And it would not be necessary to remove the tin before using the sheets, as the tin facilitates drawing and forming operations, greatly aids soldering and does not interfere with spot welding.

It is impossible therefore to say how widely this process may be used and how greatly the life of steel equipment can be extended until the industrial trials, now being made, yield some conclusions. But this study has shown that tinning is outstandingly the best pretreatment yet devised for painted steel, and that by this means its useful life may be multiplied several times.

Such longer life will undoubtedly open wider markets for light steel products including motor car bodies, gasometers, light boats and barges, steel window frames, refrigerators, gas and electric fires, water heaters, steel furniture, desks, cabinets, storage bins, and many others.

(Courtsy, U. K. Publicity Office, New Delhi).

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## Industrial importance of Magnesium

**M**AGNESIUM, the white, ultra-light metal derived principally from the magnesium chlorides in sea water, is gaining prodigious industrial importance. New alloys with increased strength, mass production techniques for casting, forming, forging, fabricating and welding, and corrosion-resistant finishes have given this remarkable metal a firm foothold as structural material for industrial products.

The tremendous wartime demand for magnesium, particularly for airplanes, brought new manufacturers into the field, and initiated an unprecedented amount of private and govern-

mental research into its properties and metallurgy. The total annual consumption of magnesium in the United States amounted to about 5,000,000 lbs. before the war; by January 1942, the capacity of American plants had risen to 322,000,000 lbs. The new techniques developed fill 45 voluminous reports published by the Office of Technical Services of the United States Department of Commerce.

The peacetime results of magnesium's wartime growth were demonstrated late in February, 1947, in an exhibit of industrial products held at Dayton, Ohio. Scores of manufacturers showed their products, ranging from baby

strollers, stepladders and lawn mowers to truck bodies, textile machinery, machine tool parts, tyre moulds, power saws and jet engine castings. But the most remarkable feature of the show were the methods used to make the products rather than the products themselves. The Dow Chemical Co. and the American Magnesium Corp., a subsidiary of the Aluminum Corp. of America, alone demonstrated more than a 100 examples, illustrating every process by which the metal is worked.

Practically all types of extrusion can be applied to magnesium. New alloys with higher temperature tolerance and wider stress range, consequently better adaptability to casting and forging, are being tested. In casting, getting magnesium into the mould and taking off the impurities once posed considerable problems. Today, however, permanent metal moulding for engine parts and other forms weighing up to 125 pounds, semi-permanent mouldings (metal with sand core) for intricate shapes are as applicable to magnesium as sand moulds.

In forming, the use of heat,—both sheet and dies are warmed—, permits so-called "deep draws" for bowl or dome shapes. The heat treatment lowers the elasticity so that the "deep draw" shapes will not "spin back" but keep their form. Deep draw pieces are true to size, require no expensive hand work. Heat also helps spinning, a process comparable to the way a potter shapes a bowl, and it is used in drop and press forging.

In machining, J. Colin Smith, development sales engineer of the Aluminum Corporation of America, thinks magnesium is the easiest metal to handle at a maximum rate of speed, especially where a great deal of drilling is needed. As a casting, he claims, magnesium's breaking strength under tension is equal to that of other metals.

Corrosion has been overcome through the development of finishes which protect the metal and often also provide abrasion resistance and colouring. Plastic coating, chemical dipping, electroplating, anodizing and painting

are the processes used. A new caustic chemical treatment is being developed, reportedly applicable on top of mechanical finishes.

Under the new processing methods magnesium has proven its wide range of usability. Production of civilian magnesium goods has gained greatly since the end of the war and in some instances output has been proceeding on a sizeable scale. One firm is turning out 6,000 magnesium griddles a day. Wheel barrows, lawn chairs, artificial hands, parts of railroad sleeping cars, trucks, buses, portable timber saws, spindles for the textile industry, elevators, canoes and golf clubs are in steady production.

Magnesium suppliers and fabricators are convinced of the great future for magnesium. They point to its light weight (one fourth that of steel), to its machinability, its high strength-to-weight ratio, and the progress in processing still possible. The Dow Chemical Company has increased its production schedule to 20,000 tons in 1947, compared with much less than half that tonnage in 1946. That is a far cry from wartime peaks but a multiple of any previous peacetime year.

With new processes, prices will have to be brought in line. Unless saving of operating costs—less weight needs less power—or saving of weight itself are considered prime requirements, some products appear overpriced for mass distribution. Pianos with magnesium frames for the string-mountings have been reduced in weight by 103 lbs. with little effect on the price. A magnesium wheelbarrow weighs only 12 1/2 lbs. but its price of \$34 marks it as a piece of luxury equipment for gentlemen gardeners rather than a tool for everybody. The magnesium mower at \$35, however, is only \$5.25 above the aluminum mower, but about 25 per cent. lighter.

Weight is an important factor for everything that moves or has to be moved. In the long run, savings in operating costs might make magnesium more economical than heavier metals at a lower purchasing price. Therein lies the metal's greatest virtue. —USIS.



## Fuel Industry based on Gasification of Coal

**A** CHEMICAL process of gasifying and liquifying coal, America's greatest source of energy (commercial reserves total 3,180,000,000,000 tons) and converting it into gas, high octane gasoline, oils and alcohol, has given birth to a new multi-million-dollar fuel industry. Capable of utilizing coal regardless of quality, the new industry will add practically inexhaustible reserves to America's fuel economy by producing highly desirable oil out of less avidly demanded coal.

Sponsored jointly by the largest United States commercial producer of bituminous coal, the *Pittsburgh Consolidated Coal Co.* and the nation's largest oil company, the *Standard Oil Co. of New Jersey*, a \$300,000 pilot plant is expected to go into operation by the end of this year; by 1950 full commercial utilization of the process is expected.

The process on which the new industry is based includes two major chemical steps in the transformation of coal into gas and oil. The first step,—extensively studied in the laboratory, found feasible, but still to be perfected—, is gasification of coal by breaking it down into carbon monoxide and hydrogen. This results in a synthetic product of comparatively low heat value which provides the raw material, or "feed" gas, for the second step of chemical transformation.

The second and final step, already accomplished on a commercial scope, with natural gas serving as "feed", in a plant of the *Standard Oil Development Company* in Baton Rouge, Louisiana, leads through a hydrocarbon synthesis. In the synthesis, the two components of the "feed" gas, carbon monoxide and hydrogen, are converted into the end products: gas of high heat value and liquid fuel, in proportions which can be varied practically at will.

The pilot plant and a \$120,000,000 commercial production plant to be finished by 1950

will be located in the heart of the rich coal region near Pittsburgh, Pennsylvania. The pilot plant, meant to show how to commercialize the process already developed in the laboratory, will produce, 2,400,000 cw. ft. of "feed" gas from 50 tons of coal daily. Following approximately a year of pilot operations, the construction of the commercial plant would begin, the expenses being met by interested industries or through a public sale of securities.

A plant of the size considered would consume 20,000 tons of coal a day and yield the equivalent of 400,000,000 cw. ft. of gas of high heat value. In summer, when no heating fuel is needed, it would switch to the production of 200,000,000 cw. ft. of gas, 14,000 barrels of high octane gasoline and certain amounts of other oils and alcohol.

In announcing the plans, the director of Standard Oil's technical "Development Company," Robert P. Russel, said the new industry would assure the United States of an "enormous potential reserve of liquid fuel" the demand for which is increasing this year. Geologists, he stated, are convinced that the United States crude oil reserves will be as large 20 years from now as they are today, but the costs of finding oil and of its production are mounting. The new synthetic fuel, on the other hand, could be made from cheaply mineable coal. As to costs, use of the synthetic fuel, even today in the stage of its experimental production, would not increase the daily operating costs of a car by more than 10 cents.

Russel estimated that half of the United States coal reserves synthesized into the new fuel would equal an oil reserve of 3,700,000,000 barrels of oil, or enough to last more than 3,000 years.

—U.S.I.S.



# Petroleum\*

By E. S. PINFOLD

**P**ETROLEUM, mineral oil, earth oil, are terms used to denote the liquid hydrocarbons occurring in nature as minerals; in a wide sense, they include also the gaseous and solid hydrocarbons, waxes and asphalts, frequently associated with mineral oil, and many of the products obtained from the crude oil by refining. Petroleum oils are distinct from oils of vegetable and animal origin, not only in their mode of occurrence, but also in chemical constitution.

Petroleum occurs at the earth's surface as oil springs or seepages, as emanations of natural gas, sometimes forming mud-volcanoes, and as pitch lakes: these surface occurrences have been known from pre-historic times, and many of them, more especially the gas shows, some of which burn continuously as 'eternal fires', have been made objects of veneration and worship, and have determined the sites of shrines and temples. In India, the temple to the goddess *Devi* at Jawalmukhi in the Kanera District is erected over a large gas spring which is kept burning continuously. Similar occurrences at Baku in Russia, and elsewhere, have been sacred to fire-worshippers from very early times. Bitumen or asphalt, resulting from the inspissation of seepage oils, was used as mortar for damp courses and for ornamental purposes, by the Sumerians in Mesopotamia. Similar material, obtained probably from oil-seepages in Baluchistan, was used in buildings ascribed to about the same period (3000 B.C.) at Mohenjo-daro in Sind, and Harappa in southern Punjab.

Mineral oil has been produced in Burma, by means of shallow hand-dug wells, from very early times, and production at the end of the eighteenth century was reported by British visitors to have been over 90,000 tons per annum. It was used probably as a wood-preserved, for medicinal purposes and possibly in lamps.

The refining of petroleum had its origin

in the processes devised by James Young and others for the treatment of shale oil produced in Scotland from 1853 onwards. Mineral oil was first produced in quantity from the Drake well drilled at Oil Greek, Pennsylvania, in 1859, and development of the oil industry was very rapid thereafter. The main products were kerosine, used as an illuminant to supplement and eventually to replace the dwindling supplies of whale oil, and lubricants. Petrol and fuel oils were at first regarded as waste products. With the rapid increase in production, supplies soon exceeded the home demand and additional markets were developed in other parts of the world. Towards the end of the nineteenth century fuel oil began to replace coal, especially for marine engines, and a few years later, the invention of the petrol engine provided a use for the lighter products of the refining process. In a short time, petrol or gasoline had replaced kerosine as the main product of the industry. The remarkable increase in the demand for petrol led to the development of a new refining process, known as 'cracking', in which, by destructive distillation under high temperatures and pressures, the kerosine and heavier products are broken down to give a further yield of motor fuels. A later phase has been the production of special quality gasolines for use in the high compression engines of the modern car and aeroplane. In the last few years there has been a great expansion in the production from petroleum of chemicals, plastics, artificial rubber and other useful materials as by-products, more especially from the unsaturated compounds which are formed in the cracking process.

The immense and unparalleled expansion in the petroleum industry has resulted in oil displacing coal as the world's most valuable mineral product; the use of petroleum and its derivatives has so revolutionised transport, industry and modern life, that the present is sometimes referred to as the 'Petroleum Age'.

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\* Contribution to the *Dictionary of Economic Products and Resources of India*. Suggestions are invited by the Chief Editor, 20 Pusa Road, New Delhi.

Mineral oils are composed essentially of carbon and hydrogen combined as hydrocarbons; they may contain, as impurities, a small percentage of sulphur, present as hydrogen sulphide, thiophenes or mercaptans, and traces of oxygen, nitrogen, vanadium, present as acids, bases, phenols and other compounds.

Although the hydrocarbon gases and lighter petroleum oils are of simple constitution, the detailed investigation of the ultimate composition of mineral oils is one of the most difficult problems of modern chemistry. The difficulty increases with the increase in molecular weight of the heavier constituents, not only because of the decomposition which may take place at the high temperatures required for their distillation and separation, but also because of the large numbers of isomers and homologous compounds which may be present. Despite the great amount of chemical research that has been and is being done in this connection, the composition of only the lower boiling point compounds has been determined, that of the heavier products being practically unknown.

Crude oils are frequently referred to as of paraffin, asphaltic or mixed base types, according to the character of the residues obtained from them after partial distillation. Most oils are of mixed base type and contain both wax and asphalt, though either may predominate. This classification of crude oils is of limited use in that it is concerned only with the heavier and less valuable constituents and gives little or no indication as to the character of the economically more important lighter fractions. Investigation has shown that these are composed of three classes of hydrocarbons, which may be present in very different proportions. The three classes are: The paraffin or aliphatic series of straight-chain compounds and their branched isomers, with the general formula  $C_n H_{2n+2}$ ; the naphthenes or cycloparaffins, ring compounds with the general formula  $C_n H_{2n}$  and the aromatic or benzene hydrocarbons with the benzene ring ( $C_6H_6$ ) as nucleus. No unsaturated compounds of the ethylene or acetylene series have yet been identified in the natural crude petroleum though such compounds occur in the products of refining and play an important part in the reactions which take place when petroleum oils and gases are subjected to high temperatures and pressures.

Classification according to the proportions of the hydrocarbon groups in the petrol and kerosine fractions is now being adopted and may displace the older method, though no suitable nomenclature has yet been devised. Table I (after B.T. Brooks, *Science of Petroleum*, Vol II, p. 987) indicates the proportion in which these hydrocarbon groups may be present:—

Type of Crude	Wax %	Asphalt %	Composition of 250-300° C. fraction.		
			Paraffin %	Naphthenes %	Benzenes %
Light Paraffinic	1.5-10	0-6.0	46-61	22-32	12-25
Paraffin-Naphthenic	1.0-6	0-6.0	5-42	9-28	16-20
Naphthenic	trace	0-6.0	15-26	61-76	8-13
Benzenoid	0-0.5	0-20	0-8	57-78	20-35

The importance of this classification is the indication it gives of the products which can be obtained from the crude oil by refining: Oils rich in paraffins usually yield high grade kerosine and lubricants, the oils of Pennsylvania being the best known examples; naphthenic crudes usually contain asphalt and require a more elaborate refining process. Oils rich in aromatics for example, from oil fields in British North Borneo, yield anti-knock gasolines, valuable for blending with other fuels, and toluene, used in the manufacture of explosives.

### Units and Specifications

Oil analyses and specifications usually include specific gravity, viscosity, percentage of sulphur, initial boiling point and the percentage of fractions distilling within a given temperature range, or the temperature ranges over which given percentages of the total volume distil.

The characteristics of crude oil from Burma are given by W. J. Wilson as follows\*:

Specific gravity (60° F)	0.835—0.842.
Colour (liquid)	Brown by transmitted, Green by reflected light.
Setting point	780° —81° F.
Closed flash-point	< 50° F.
Sulphur content	0.15%.
Wax content	From 14.8% of m.p. 127° C. to 16% of m.p. 123° C.
Resins and asphalts	< 3%.
Naphthenic acids	< 0.02%.
Ash content	< 0.0035%.
Calorific value	10,900 cal. per gm.; 19,630 B. Th. U. per lb.
Ultimate analysis	Carbon, 86.45%; Hydrogen, 13.25%; Nitrogen and Sulphur, 0.30%.

\* This, and the foregoing Table, are quoted with the permission of the Publisher, Oxford University Press.

## Distillation Test :-

		Initial boiling point, 64° C.			
		%			
<	100°, C.	5.9	5% distils at	96° C.	
100	150°	17.4	10	" "	112°
150	200°	10.5	20	" "	138°
200	225°	5.2	30	" "	180°
225	250°	6.6	40	" "	228°
250	285°	11.7	50	" "	263°
285	300°	5.0	60	" "	292°
>	300°	37.5			

The range of products obtained from any crude varies mainly with its composition and partly according to the refining treatment to which it is subjected.

Production and other figures are given in tons, metric tons, barrels or gallons; 1 USA gallons = 0.83268 Imperial gallons, 1 USA barrel = 42 USA gallons, or 34.973 Imp. gallons, the barrel used in India is 40 Imp. gallons.

In most British possessions, specific gravity is used to indicate the density and quality of crude and refined petroleum oils. A different hydrometer scale is used in America: this is graduated in degrees API (American Petroleum Institute). This scale is related to specific gravity by the following formulae :-

$$SG (60^\circ / 60^\circ F) = \frac{145.5}{131.5 + \text{API degrees.}}$$

$$\text{Degrees API} = \frac{141.5}{SG (60^\circ / 60^\circ F)} - 131.5$$

The following are equivalent readings on the two scales :-

° API	SG	° API	SG
0	1.0760	30	0.8762
10	1.0000	40	0.8251
20	0.9340	50	0.7796

## Occurrence

Oil occurs in the sedimentary rocks of all geological ages in which suitable oil-source materials were present originally, and where the structural conditions and later geological history have been favourable to its preservation. Oil can migrate freely through fissures and cracks and through porous strata, and the rocks in which it accumulates eventually are not necessarily those in which it was first formed. Vast quantities of oil-bearing rocks have been removed by denudation and erosion during the history of the earth's crust, and, at the present day, the seepage of oil and gas from surface springs represents, in the aggregate, a continuous loss of petroleum minerals; such losses must have been greater in the older

than in the younger rock systems, more especially during those periods now represented by unconformities; partly as a result of this, the larger part of the world's petroleum is found in the comparatively young strata of the late Cretaceous and Tertiary periods.

Oil is known, though not in commercial quantities, in the oldest fossiliferous rocks, the Cambrian, and important oil fields occur in the next younger system, Ordovician, in Central and Eastern North America, where the oil source rocks have remained buried under great thicknesses of impermeable material and where subsequent earth movement has been slight. The Silurian and Devonian rocks of the same regions are also oil bearing. Large production obtained from the Carboniferous and Permian in Eastern North America, in Germany and in the recently developed Russian fields, west of Urals. Oil is less plentiful in the Trias and Jurassic, and no important oil deposits have been proved in rocks of these systems. The most prolific oilfields obtain their production from the Upper Cretaceous and from the Eocene, Oligocene and Miocene.

Petroleum occurs in sedimentary rocks, usually of marine or estuarine deposition; it is frequently associated with rock salt, gypsum, dolomitic limestones and red marls, laid down under inland sea conditions. Conditions are known to have been unfavourable to the formation and occurrence of oil in many regions now forming a large proportion of the earth's surface. Such barren regions are :- (i) The 'ancient shields',—stable portions of the crust occupied by Pre-Cambrian gneisses, schists and very old sedimentary deposits formed by their denudation; Peninsular India, South and Central Africa, Australia, Scandinavia and Labrador, belong to this category; (ii) The great accumulations of basaltic lavas of which the best example is the Indian Deccan. The oily material once reported from the Deccan traps, near Bombay, proved on investigation, to be the altered droppings of bats; (iii) The central cores of the great Alpine mountain belts, the Himalaya, the Alps, the Andes and the Rocky Mountains. These tracts have been subjected to intense earth movements, accompanied frequently by igneous intrusion and dynamic metamorphism, and any oil which may have been present has been lost by erosion and dispersal.

Regions favourable to the occurrence of oil, both as surface shows and in commercial quantities, have been described by Van der Gracht as the following:— (i) The foot-hills of the great mountain chains, where suitable source rocks have been deposited which have later escaped the violent diastrophism of inner mountain belt; (ii) Unstable plateau regions, further from, but still adjacent to, the mountain chains, where, due to vertical movements, considerable thicknesses of suitable deposits have later undergone only minor compression and folding. These two categories are not sharply differentiated. The main Indian occurrences are in regions which are too distant from the Himalayas and Assam-Arakan ranges to be considered as in the foot-hills or 'fore-deeps', and are sufficiently sharply folded to be distinguished from the mobile plateau areas. The Punjab oil occurrences, associated closely with deposits of rock salt and gypsum, have more affinities with Van der Gracht's second class than those of Assam and Burma.

The close association of oil when the geosynclinal regions, the centres of which became the great mountain chains, accounts for the present distribution of oil fields in 'belts' or provinces parallel and adjacent to the main ranges. Thus, the oil occurrences of India and Burma are confined to Baluchistan, the Frontier Province and northwest Punjab, Upper Assam and the Burma Valley, and the Irrawady valley, all at no great distance from the Tertiary mountain ranges. This Indian 'oilbelt' may be regarded as the continuation of the oil occurrences in Iran and Iraq on the west, and of the oil field of the Dutch East Indies to the south east. Within these regions, however, there are several separate petroleum provinces: the Iranian oil occurs in the Asmari Limestone of Oligocene/Miocene age; the seepages of Baluchistan are at or near the base of the Laki stage of the Eocene; the Punjab oil originates in the upper part of the Laki; whilst the Assam and Burma oilfields are in sandstones and shales ranging in age from Eocene to Miocene.

### Origin

Despite much world-wide research the exact method of oil formation is still unknown. Traces of hydrocarbons occur in the gaseous emanations of some volcanoes. Early experiments proved that oils could be obtained from the interaction of water with metallic carbides

under special conditions of temperature and pressure, and this led to the conception that petroleum was formed by similar reactions taking place in the earth's interior. This and related 'inorganic' theories have now been abandoned for several reasons, some of which are: the rare association of oil deposits with past or recent volcanic activity and the presence in oil of optically active compounds, such as phytosterol and cholesterol which would not have survived high temperatures.

The constant association of petroleum with sedimentary deposits indicates that oil is of organic origin, and that the source material has been the remains of animal and vegetable life buried in the muds and other deposits of shallow seas. The exact process by which such material is transformed into oil is still uncertain. Wide research on sediments now being laid down has failed to reveal a single instance in which petroleum (as apart from methane or marsh gas) is being formed at the present day; some oil has been obtained from muds from the sea, but this may have had its source in submarine seepages and the oil may not be indigenous to the sediments in which it now occurs.

It is now generally agreed that the process by which oil is formed from organic material may be of a biochemical nature, and that special and as yet unknown bacteria or other organisms may have played an important part. The subject offers a promising field for further research. The frequent association of oil with inland sea deposits suggests that high salinity, or the change from open sea to inland sea conditions, may have been favourable to oil formation. In the case of salt domes, however, the accumulation of oil is sometimes adventitious and due solely to the favourable structure in the rocks adjacent to the salt plug.

The nature of the source material has also been the subject of much discussion. For some years it was supposed that it was vegetation, mainly terrestrial, which, under other conditions have formed coal. Oil and coal occur frequently in rocks of the same age and of not very different lithological character: the Barail rocks of Upper Assam contain important coal-seams and oil source rocks in the same region. Most oil-bearing rocks are, however, of marine or estuarine origin, and the prevailing opinion is that the source materials of petroleum were the remains of marine

organisms; marine algae may have been important contributors. Beeby Thompson and others have drawn attention to the presence of abundant fish remains in the petroliferous rock of Russia and elsewhere, and have suggested that fish may have been the main source of the oil. This theory has not obtained wide acceptance, one of the arguments against it being that oil occurs in Lower Palaeozoic rocks which were laid down before fish had appeared; this, however, is not conclusive, for the progenitors of fish and other similar forms were abundant in Cambrian and Ordovician times. It may be noted that fish remains occur, sometimes abundantly, in the oil-source rocks of Baluchistan, north-west India and Burma.

### Accumulation and Exploitation

In those regions where the geological conditions are favourable to oil occurrence, a study of the surface evidence and geological history will indicate the stratigraphic horizons at which oil-source rocks occur and suitable reservoir rocks in which it may have accumulated. Oil seepages occur where the source-rocks outcrop at the surface and in conditions which allow the continuous escape of the oil; such conditions are not usually favourable to the accumulation of oil in quantity and under pressure. The presence of a seepage, therefore, cannot necessarily be taken as an indication that workable oil will exist in the immediate vicinity; seepages do occur on some oilfields, but many large fields have been discovered in areas devoid of oil of seepages or gas shows.

Oil prospecting was at first dependent entirely on surface shows, but geologists quickly recognised that successful wells were obtained in areas elongated parallel to the general strike of the regional structure, and more particularly along the crests of arch-like folds of the strata, known to geologists as anti-clines. The troughs, or synclines, between the arches were unfavourable areas, and wells drilled in them usually obtained water only at the equivalent depth of the oil sands. This association of oilfields with anticlinal rock structure, known as the anticlinal law, at first received scant recognition, and for almost half a century wells continued to be located on seepage evidence, on the general trend of earlier discoveries, or entirely haphazard or 'wild cat'. It was only after the beginning of the present century that, with the accumulation

of evidence, the anticlinal law was admitted to be of fundamental importance in the search for new fields. The only modification in this law since its inception is the inclusion of other geological structures as being also favourable. The feature common to all these is that they provide an obstacle to unward migration of the oil and so form a trap in which the oil accumulates as it separates from water under the influence of gravity, assisted by compression and earth movements. The anticline is the most favourable of such structural traps, more especially at points along the fold axis where opposed pitches in conjunction with the flank dips form an inverted bowl or dome. Most of the world's oilfields, and those so far developed in India and Burma, are located on dome or semi-dome structures.

Another form of trap is provided by the abutment of oilbearing beds against the plane of an unconformity or non-sequence in the strata column. Seepages frequently occur at the outcrops of such unconformities, the oil migrating from the burine source or reservoir rocks through the porous basal rocks of the over-lying series.

Important structural traps are also formed by fractures caused by differential movement of adjacent parts of the earth's crust, known as faults; such structures are of increased importance when they are transverse to an anticlinal axis: The Pynma oil-pool of Burma occurs in a fault trap of this kind.

Stratigraphic traps are formed by lenses of oil-bearing sand in impervious shales or clays, conditions usually associated with shallow water and shore-line conditions of deposition.

For the occurrence of a workable oil field, the presence of an oil source of sufficient magnitude is a first essential, and the richness of an oil-bearing region is directly dependent upon the thickness of such source beds and the abundance of the material from which the oil was formed. Local conditions of deposition have frequently played an important part in this connection, and have probably been responsible for the considerable variation in productivity which occurs in many petroliferous regions. Unfortunately the effect of such local conditions is often difficult to assess until the results of several test wells are available.

Other conditions essential to the occurrence of oil in quantity are: the presence of an im-



permeable cap-rock or cover which forms the actual trap and prevents the further upward movement of the oil and gas and retains them, possibly under considerable pressure; and sufficient permeability or fissure space in the reservoir rock to admit of the oil accumulating in sufficient volume. The reservoir rock is either a sand, grit or conglomerate, in which the oil can occupy the cracks, fissures and joints, sometimes widened by solution.

An oil-pool is the result of the accumulation of oil by migration laterally, and possibly vertically, over considerable distances, and the size of the area so drained, actually the size of the favourable trap or closed structure, is another important factor in determining the amount of oil which is contained ultimately in the productive area. That oil migration is often a slow process, continuous over long stretches of geological time, is indicated by the continuous escape of oil and gas at surface seepages.

The final requisite for a commercially exploitable deposit is that the oil reservoir must be within reach of the drilling methods available. There has been, and is likely to be, continued progress and improvement in drilling apparatus and technique. In the early years of the industry, a few hundred feet was the limit to which wells could be drilled, and, as late as the early years of the present century 2,000' to 3,000' was seldom exceeded. With the introduction of the rotary method of drilling, about 1915, this limit has been rapidly increased; many oil-fields are now being worked at depths of 7,000' to 8,000' and some of the fields in northwest India are producing from these depths. The deepest well yet drilled for oil has reached over 16,000' (1945). Much of the new oil production obtained in recent years has resulted from the deeper drilling of oilfields which were on the point of exhaustion from the older shallower development. Additional depth, however, may involve special difficulties connected with high rock and fluid pressures and high temperatures, and further progress in deep drilling may eventually be limited by these factors.

### Oil Prospecting

The search for workable oil in any region require as complete a knowledge of the local geology as can be obtained in the time available, from records of previous work and from the surface evidence; where the surface geology

is obscured by alluvium it may be necessary to use geophysical methods described in a later paragraph. Seepages of oil or gas are usually well known to the local people and mentioned in the literature. The first stages of the investigation should reveal the geological succession, the presence or absence of unconformities and the conditions under which the various rock groups were deposited. Oil seepages are most abundant in the actual source rocks or in adjacent porous beds, and this evidence may be sufficient to indicate the stratigraphic horizons in which oil is likely to occur. This preliminary work will also indicate probable reservoir rocks and their impervious covers. Much of this evidence will be obtained in areas in which the oil-bearing beds outcrop at the surface and where structural conditions are unfavourable to the accumulation of oil in quantity. The geological survey must then be extended over adjoining regions in which the possible oil reservoirs are buried beneath a considerable thickness of barren sediments, in order to determine whether suitable trap structures exist in which the oil-bearing rocks are within reach of the drill. The final stage will be the detailed mapping of the more favourable areas to decide the best position for the test well or wells.

It is only rarely that all the conditions necessary for a successful result are known to be present: The structure may be imperfect; the oil source may be of unknown capacity, or even doubtfully present; the reservoir rock may be too deep. These risks, however, have to be taken, and the drill must be the final arbiter. It is this which introduces the considerable hazard usually present in oil prospecting, and notwithstanding competent geological advice, a considerable proportion of failures must occur. An oil field, from the outset of production, is a wasting asset and will become exhausted within a period dependent upon the conditions and upon the rate at which the oil is produced. All the larger and conservatively managed oil companies regard prospecting as an essential part of their activities, and set aside large sums for this purpose. Oil prospecting in India and Burma has been continuous for almost forty years and the expenditure estailed has reached many crores of rupees; successes have been few, and much of this heavy expenditure has shown no return. The history of most of the great oil producing regions of the world has

been the rapid and successful development of a few obviously favourable propositions, followed by only moderate successes and many failures as areas in which the evidence is less favourable or less complete are later included in the prospecting programme.

### Aerial Surveys

Before air transport became available, the search for oil necessitated a survey by geologists working over a region, mostly on foot; such a survey was costly, both in time and money. Much of the evidence can now be obtained more quickly and at considerably less cost by the examination of photographs taken from the air, and much use is now being made of aerial surveys, more especially in regions which are difficult of access by other means. The aerial method is particularly well adapted to the reconnaissance of desert country in which the rock outcrops are not obscured by surface deposits or vegetation; even in jungle covered tracts, useful information may be obtained from variations in the topography and vegetation. In favourable conditions it is possible to locate from the air the parts of a region that require more detailed examination, and to eliminate large tracts in which the geological conditions are revealed as unfavourable. It is invariably necessary to supplement the work done from the air by detailed work on the ground, in order to establish key beds and their relation to likely source and reservoir rocks. Detailed ground work will also be required before test wells are finally located, but in this, as in the selection of suitable routes, etc., the air survey will provide a useful topographic base.

### Geophysical and Geochemical Prospecting

In regions where the geological structure is obscured by recent deposits, other sciences are called in to provide what evidence they can of conditions at depth. The torsion balance and gravimeter enable the variations in the forces of gravity to be plotted; this is equivalent to the determination of the relative weight of the rock column at given points, and these results, in conjunction with what is known of the rock sequence in neighbouring areas, are sometimes sufficient to indicate areas favourable for testing. Variation in the elasticity of the rocks at depth can be determined by the production of miniature earthquake shock and the

measurement of the velocity of the resulting earth-waves. The waves are generated by an explosion, and their arrival at selected points is recorded by seismographs; variations in velocity reflect variation in the elasticity of the underlying formations. The reflection of earthwaves at underground rock surfaces may be determined by a modification of these seismic methods. Geophysical work has been conspicuously successful in the detection of salt domes and associated oil occurrences in the Gulf Coast of America.

More recently, detailed analyses of the surface rocks, and the detection and determination of any hydrocarbons present, has also been used in the search for oil in areas other evidence may be insufficient or requires further confirmation.

### Drilling and Development

The early production from the Yenangaung field in Burma was obtained entirely from hand-dug wells, and this primitive method of drilling has continued in use to the present day. A shaft about 5' square is excavated by hand; and as the hole becomes deeper, the debris is lifted to the surface in tins or baskets by means of a rope passing over a roller and pulled by one or more coolies; the upper part of the hole is timbered to prevent collapse of the walls. When working in oil and gas-bearing formation the well digger had to be hauled to the surface at intervals of a few minutes in order to avoid asphyxiation; he is now provided with a helmet and supplied with air by means of a hose connecting with a primitive air pump worked by hand at the surface. Sunlight is reflected to the bottom of the hole by a mirror. Many hundreds of wells have been carried to over 300' by these methods and many thousands of tons of oil have been produced from them. Other primitive drilling methods used in Japan, China, and elsewhere, employ a percussion principle,—a heavy drilling bit or pipe, is alternately raised and allowed to drop on bottom, thus pulverising the rock. The motion is obtained by means of a rope passing over a pulley at the top of a tripod of mast, the bit being raised by coolies pulling on the rope, or by attaching the rope to a spring board or pole on which the coolies jump.

The percussion system was also the basis of

first steam driven drilling outfits, the percussion action being applied by means of a crank and 'walking beam' to the end of which the drilling cable is attached. A screw clamp allowed the driller to feed the line as the bit cut deeper. This process, known as cable-tool drilling, was used, with only minor modifications, from the time of the Drake well (1859) for more than half a century, and it is still in use for shallow wells and for special purposes as for drilling through productive zones. A timber or steel derrick is necessary to allow the long string of tools to be lifted to the surface. The cuttings are removed by means of a bailer lowered into the hole on a separate winch. The main limitation of cable-tool drilling is that the hole has usually to be cased with iron or steel pipes continuously as it progresses in order to prevent the walls of the hole falling in. A hole of sufficient size to accommodate the casing is obtained by the use of expanding bits or reamers, but in caving formation the depth to which a string of casing can be carried is limited by the increasing friction against the wall of the hole. When an upper water-bearing formation requires to be sealed off, the string of casing is cemented in the hole by forcing cement round the casing shoe and between the casing and the wall of the hole; sufficient time is allowed for the cement to set, and the cementation is then tested; drilling proceeds thereafter in a smaller diameter hole, another casing string being inserted and carried ahead. A deep well must therefore commence drilling in a large diameter hole (up to 30") and several successive strings may have to be set, so that the eventual diameter may be reduced to 4.5" or less, the hole being lined by up to six strings of casing, each extending from the surface to a greater depth than the next larger in diameter. For this and other reasons cable-tool drilling is both slow and expensive, and it has been almost completely displaced by the rotary system.

This method has much in common with the rotary core drill used in mineral prospecting. The string of drilling tools is given a rotary instead of a percussion movement and the debris is removed by a continuous mud or water stream pumped by way of a swivel down the tubular drill stem, emerging at or near the cutting edge of the bit, and returning to the surface between the drill stem and the walls of the hole. The drill-stem consists of pipes

screwed together in sections or stands; a high derrick permits these to be stacked vertically when the drill-stem is withdrawn, and additional sections are added as the hole gets deeper. The string as a whole is suspended by the swivel from a travelling block and raised or lowered by cable and winch. The bit is chisel-shaped or 'fish-tail' and eats its way into the formation by a direct cutting action, or, for harder formation, it may consist of toothed rollers which roll on bottom with a pounding action so pulverising the rock.

The rotary method has revolutionised the drilling technique, and is now used in all fields where moderately deep or deep wells are necessary. Not only is the drilling rate much faster with this system but there is a considerable economy in the amount of casing required. A short conductor string is set at the surface in order to enable the mud flow to be controlled, thereafter the mud plastering, and the weight of the fluid column in the hole, is usually sufficient to hold up the walls and prevent caving. In this way, casing need not be required until immediately above the oil zone, and in many cases this may be the only long string used; a short length of perforated liner is frequently set through the producing beds. Recent advances include building the drilling machinery in units to increase portability, the use of internal combustion engines as prime movers, and the adoption of smaller hole diameters.

Great care is taken to isolate the oil-bearing sands or limestones from any water which may occur in the formations above serious effect on the production of an individual well and may affect a whole field and result in a considerable loss of ultimate yield. Methods used in Burma are discussed in detail by C. T. Barber<sup>2</sup>.

### Production Methods

When first tapped, an oil-sand is usually under considerable pressure, sufficient to cause the oil and gas to flow at the surface; this flow is controlled by a series of valves at the well-head, known as the 'Christmas tree'. When there is free fluid communication in the oil sand, the pressure is normally equivalent to that of a water column from the reservoir to the outcrop of the oil sand at surface. In some fields, notably in the Punjab, pressures greatly in excess of the normal artesian pres-

sure are encountered; no satisfactory explanation of these excess pressures has yet been suggested.

The removal of oil or gas from an oilfield more rapidly than it can be replaced by water from the lower portion of the structure causes a fall in reservoir pressure at a rate varying with the offtake. After a period depending upon the conditions, the pressure becomes insufficient to force the oil to the surface; it has then to be lifted by artificial means. The methods used are pumping, gas-lift, bailing or swabbing.

In pumping, a piston or working valve, connected by sucker rods to the surface machinery, moves in a working barrel attached to a string of tubing and placed at or near the bottom of the well. A reciprocating movement is given to the rods by means either of a system similar to that used for cable tool drilling, or a specially designed geared pumping unit. In the case of small shallow wells, a central power may pump perhaps a hundred wells by means of an eccentric and rod or cable connection to 'jacks' at the well head.

Gas-lift is artificial or artificially assisted flowing, and entails the compression and injection of natural gas into the column of oil. The gas reduces the weight of the column of fluid in the hole and induces flowing. Several systems of intermittent gas-lift are in use; these involve the employment of two strings of tubing, and the oil is blown out by the admission of gas at regular intervals.

Bailing may be used when the oil carries with it a large amount of solid particles which would interfere seriously with the action of a pump, or would entail heavy wear and depreciation of the equipment. The majority of the hand-dug wells in Yenangyaung are produced by bailing, the capital cost of the equipment needed being low.

Swabbing is occasionally resorted to in order to clean the well before installing a pump to induce flowing, or to remove large volumes of liquid. The method consists of running a 'swab' and pulling it out by means of a cable and winch, the swab being essentially a valve and rubber sleeves which fits tightly into the casing.

### Secondary Recovery Methods

Ordinary production methods of necessity leaves behind a high proportion of the oil originally in the reservoir rocks. Various methods of reducing the amount of oil not recovered have been devised; these include gas-drives, water-drives, vacuum, the use of acids and explosives.

With gas drives, air or natural gas is compressed and injected into one well and in passing through the sand, drives the remaining oil to the producing wells, gas is preferable to air since the latter may cause corrosion troubles and explosive mixtures. This method, although of wide application in the U.S.A., has not shown outstanding results in Burma. Water drives are conducted on similar principles; care has to be exercised to see that solids are not precipitated in the water which would clog the sand in the injection well. Water drives or water flooding have not been used in India or Burma, although, in special conditions in America, they have proved profitable.

The pressure in the wells can be lowered below atmospheric by means of vacuum pumps, and in the later stages of the life of an oilfield, this method of increasing the differential pressure between the oil sand and the well may result in moderate increase in ultimate recovery. Vacuum has been used extensively in the Native Reserves of the Yenangyaung field.

The use of acid is confined to fields in which the reservoir rock is a limestone. Hydrochloric acid, with a small proportion of an inhibiting agent to prevent the corrosion of the steel casing, is pumped into the well and forced into the strata; part of the limestone is dissolved, and this enlarges the channels and fissures and increases the rate of flow into the well. The use of explosives is confined to hard compact strata; the explosion shatters the rock in the immediate neighbourhood of the well and provides additional channels for the flow of oil.

(To be continued)

### References

1. *Sciences of Petroleum*, Vol. II. P. 891.
2. *Rec. Geol. Sur. Ind.*, 1930, 63, 379; E. J. Bradshaw, *Ibid*, 1937, 72, 141.



# A Mains Operated

## Stabilised High Tension Unit

FOR very many purposes in factories and laboratories it is desirable to have at one's disposal a source of voltage that is independent of the current, and as such, an accumulator battery has hitherto been most commonly used. There are, however, a number of drawbacks attached to the batteries, some of which are mentioned here :

- (1) Heavy weight, and consequently not easily portable.
- (2) Necessity of repeated recharging and maintenance.
- (3) Instability of the terminal voltage due to variation with time.
- (4) Rather high internal resistance, so that the voltage is dependent upon the current.
- (5) High initial cost and expense of maintenance.

With accumulator batteries the purpose aimed at is only approximately attained.

Neither can the purpose desired be reached with a simple mains voltage rectifier consisting of a transformer, rectifier and filter, because its internal resistance is so high that the output voltage is greatly affected by the current, whilst moreover, mains voltage fluctuations influence the output voltage.

Improvement is obtained by employing neon stabilising valves, but the internal resistance, which is then equal to the differentiated resistance of the stabilising valves, may still be anything between several score and several hundreds of ohms. If, however, one introduces in series with the output of a high tension supply unit, a resistance which automatically increases when through some cause or other, the output voltage rises, then a constant output voltage can be obtained. As such a variable resistance one may take, for example, a triode the resistance of which is varied by a suitable grid voltage, obtained, for instance, as the differential between the output voltage and a fixed voltage from a dry battery. A dry

battery is highly stable provided it does not have to supply any current.

The simplest solution, therefore, is a circuit in which an electron valve acts as a variable resistance in series with the output voltage. Small variations in the grid voltage are sufficient to cause large variations in the anode tension. If a still greater stability of the output voltage is required, then the small differences between the constant voltage of the dry battery and the output voltage are amplified with the aid of an amplifying valve. By conducting this amplified voltage to the grid of the valve first mentioned, which acts as a variable resistance in the high tension line, stability is considerably increased, approximately by the same factor as that of the amplification of the valve connected up as amplifier. By arranging a special circuit it can be also be ensured that the amplification of the amplifying valve remains as constant as possible.

Furthermore, a suitable circuiting provides for voltage correction whenever variations take place in the mains tension. Thus a much better independence of the mains tension is obtained. Furthermore provision is made for compensation of the remaining internal resistance, by employing in the supply apparatus a bridge circuit which reduce the internal resistance even to a level appreciably lower than that of an accumulator battery of the same voltage supplying an equal current.

Still other measures have been taken to increase voltage stability. The filament currents of the amplifying and regulating valves are kept constant by means of a ballast tube which is cut by a relay for bringing the valve filaments quickly to the required temperature and then automatically switched on by the relay, thus bringing also this stabilisation into action.

In this way a supply unit with a highly stabilised output voltage has been produced, viz. the *Philips GM 4560*. With mains variations of 5 per cent. the fluctuation in the output voltage is less than 0.004 per cent. which



means an *improvement* of at least a factor of 1,250 compared with a normal supply apparatus.

The internal resistance under normal working conditions is less than 1 ohm.

Some of the uses for which this voltage supply unit is especially suitable are—

- (1) Cable works : cable testing, instrument testing, etc.
- (2) Measuring instrument making : calibrations, testing, etc.
- (3) P.T.T. exchanges : testing cables, instruments, etc.
- (4) Laboratories, High Schools and Universities.
- (5) Radio transmitters : e.g. for powering line amplifiers.
- (6) Radio distributing stations: for supplying the distributing apparatus.
- (7) Radio laboratories, Aerodynamic laboratories, Pharmaceutic laboratories, etc.

The advantages of this supply unit are obvious. It is of small dimensions, 20 x 43 x 31 cm. and weighs only 18 kgm. The dimensions and weight of an accumulator battery of the same capacity are at least 3 times as great. Consequently the Philips unit is much easier to transport and, moreover, there is no spilling of acid.

The drawbacks of accumulators are many: they are not easily transportable, they are apt to be run down just when they are needed, so that a freshly charged one has to be fetched, and everything has to be set up anew; this

also implies that two sets of batteries have to be kept, one in use while the other is being charged; the voltage can only be adjusted in stages; maintenance costs time and money (charging, acid testing, renewing plates, etc.).

The stabilised supply unit only needs plugging into the light mains. It is suitable for all a.c. voltages between 110 and 245 V. Cost of maintenance is negligible.

The efficiency of the stabilised supply unit is about 20 percent viz. 150 W capacity from the light mains at a full load of 0.1 and 310 V. The efficiency of an accumulator battery calculated from the mains to the consuming apparatus is not appreciably higher.

Finally we give the electrical data of the Philips supply unit GM 4560 :

*Mains* : 110|125|145|200|245 V;  $\pm 10\%$ ;  
40—100 c/.

*Output* : Variable between 145 and 310 V in 10 stages of 15 V each and each stage continuously variable for 30 V.

Maximum current 100 milli amps. Momentary short-circuit current 400 milli amps.

*Stabilisation* : Better than 0.004% for mains voltage fluctuations of 5 per cent. Voltage variation less than 0.1 V between no load and full load of 0.1 amp.

*Internal* : At 150 V: with current dis. 30 milli amps.: 0.5 Ohm.

*Internal* : At 150 V: with current dis. 30 milli amps.: 2 Ohm.

*Resistance* : At 300 V: with current dis. 30 milli amps.: 1 Ohm.

*Resistance* : At 300 V: with current dis. 30 milli amps.: 4 Ohm.

*Hum voltage* : Less than 0.001% of the rectified voltage, independent of the load.

## REVIEWS

**X-rays in Research and Industry**, by H. Hirst, (Chapman and Hall Ltd. London) 1946, VII 124, Price 13 sh. 6d.

**D**URING recent years, X-rays are being employed in the investigation of a variety of industrial problems. They have proved particularly useful in metallurgical research. A book which deals with the fundamental principles underlying the applications of X-rays to industry in a manner intelligible even to the beginner, is indeed very welcome. This explains why a second edition of this small book by Prof. Hirst has become necessary within two years of the publication of its first edition.

The book contains six chapters. The first four are short ones dealing with the methods of production and properties of X-rays and the fundamental principles of X-ray crystallography. They serve as an introduction to the fifth chapter describing the applications of X-ray diffraction methods to the study of metals and alloys. In the last chapter are described different branches of industrial radiography. The author has been remarkably successful in making the presentation lucid and he deserves praise for the skill and thoroughness with which he has expounded the whole field of applications of X-rays to industry in such a short space.

K. BANERJEE.  
B. S. BASAK.

**Reports on Fuel Economy Since 1939.** British Report. (Central Office of the World Power Conference, London) 1946, Pp 52. Price 1 sh. 6d.

This contribution of the British National Committee of the World Power Conference to the series of reports on "Fuel Economy since 1939" constitutes preparatory material for the Fuel Economy Conference to be held at the Hague from 2nd to 9th September, 1947.

This comprehensive document covers the history of the fuel efficiency campaign conducted by the British Ministry of Fuel and Power during the war years. It deals with the restrictions controlling the supply position, the

conditions which gave rise to the need for the campaign, its organisation, the publicity used and the results achieved. The Fuel Efficiency Committee is convinced that the work carried out during the war years which yielded such splendid results, has but scratched the surface of the problems connected with fuel technology. Combined power and heating, utilising back pressure or pass-out steam, correct design and insulation of furnaces, recovery and utilisation of waste heat, the value of steam links between factories and the many possibilities in the development of new plant and appliances, both in industrial and domestic fields, are among the considerations for increasing the efficiency of fuel utilisation, which are discussed in the report. Examples are quoted in the report of what has been done in various industries which, if adopted universally would save several million tons of fuel a year.

### **Agricultural Economics in Canada**

Specialists who met in conference at Quebec on June 24, 1946 to discuss the current position and future trends in agricultural economics, have contributed a series of valuable papers which have been collected together and published by the Agricultural Institute of Canada in *Scientific Agriculture* (Oct. 1946).

In many countries of the world the State is wedded to a policy of deliberate economic planning with a view to maintaining full employment, and it is inevitable that in each country the State should formulate and execute an appropriate agricultural policy as part of the general economic policy. Research in the field of agricultural economics has, therefore, in the world of today ceased to be of mere theoretical interest; it is becoming more and more necessary preliminary to economic planning; and as J. B. Rutherford has emphasized in his paper on Research Trends in Agricultural Economics: "Future research in agricultural economics will be directed more to the solution of immediate problems and less to fact gathering and description of the problems as an end in itself. The research worker of the future must perforce not only describe the problem but proceed from this to propound a genuine and workable solution."

In Canada, as in India, the proper function of research in agricultural economics is to help in (a) the determination of the objectives of policy and (b) the choice of the policies most likely to achieve these objectives. As regards the former, the following objectives have been suggested by Prof. W. M. Drummond in his illuminating articles, *Agricultural Policy and Agricultural Economic Research: Protection of natural resources against wasteful exploitation and neglect; promotion of the most effective and economical use of these resources from the long run point of view; raising the general level of efficiency in agricultural production and marketing; a rise in the standard of living of those engaged in farming; and greater security to the farming population against natural and economic disasters.* This list of objectives would seem to be as suited to India as it is to Canada, though in India with her appalling malnutrition and inequitable systems of land tenure, greater emphasis may have to be placed on planned agricultural production suited to the nutritional needs of the population, the achievement of a proper balance between food and cash crops and the evolution of satisfactory systems of landholdings.

Obviously, sound policies designed to achieve these objectives can be formulated only on the basis of comprehensive economic research. As Professor Drummond has pointed out, among the main types of research in agricultural economics urgently needed today are: (a) research designed to bring production more in line with the principle of comparative advantage and the requirements of good nutrition; (b) research designed to indicate producer and consumer response to price changes; (c) research designed to indicate the kind of rural welfare policies required; (d) research aimed at achieving increasing efficiency in production and marketing, (e) research aimed at expanding demand; and (f) research designed to assist in transforming the need or desire for food into effective demand. If further research on these lines is deemed to be necessary for Canada three quarters of whose people are not farmers and whose main problem is not so much to make up a deficit in food supply as to find a market for the available surpluses, the urgent need for such research in India is obvious. Research workers in Indian agriculture, as well as those concerned with the framing of India's agricultural policy, would do well to study this issue of

*Scientific Agriculture* to get an idea both of the complexity of the problems involved and some of the ways in which these can be successfully tackled.

P. S. NARASIMHAN.

The Origin of Forces responsible for the Disruption of Continents, Mountain Building and Continental Drift: Origin and Permanence of Ocean Basins and Distribution of Land and Sea. By H. L. Chhibber, (*Bulletin No. 5, National Geographical Society of India, Benares*), 1947, pp. 6, Price As. 8.

In this bulletin Dr. Chhibber has dealt with the complicated subject of the origin of forces responsible for mountain formation and continental drift. In recent years there has been a considerable amount of discussion on this subject. Mountain building episodes in certain phases of geological history have been attributed to horizontal and vertical earth movements, and according to the present author, the loss of internal heat of the earth develops forces of compression which ultimately gives rise to mountain building by the buckling of geosynclinal sediments. In analysing the highly interesting theory of Wegener, Prof. Chhibber has taken the case of the disruption of Gondwanaland. He is of the view that forces of mountain building become the cause of continental drift, for, compression necessarily accounts for tension in adjoining region.

In the same *Bulletin*, the author has taken up the question of the origin of ocean basin and distribution of land and sea. He has dealt with this vast subject with precision, and has drawn conclusions which are, sometimes, rather assertive. To him the idea of the permanence of ocean basin is a "myth", while the hypothesis of the formation of the moon with the earth's sial is 'irrelevant'. The author has taken up the theories with a definite point of view and says, "the forces, which bring about the disruption of land-masses, building of mountains and the consequent drifting of continents are interconnected".

It is interesting to note that the author has also included "atomic energy" as a motive force required for the uprise of igneous intrusions.

The *Bulletin* will be of great help to advanced students of physical geography and geology.

SITANSHU MOOKERJI

# Textile Testing in Germany

THE subject of textile testing in Germany, during the years of world War II, was thoroughly investigated by a team of American Textile Scientists, serving as scientific consultants to the Technical Industrial Intelligence Committee. A report of the findings of this team is published in the *A.S.T.M. Bulletin*, 1947, No. 144, P. 17.

## Fibre Testing

The testing of individual fibres for breaking strength and extension was a routine procedure in Germany. The Schopper single fibre strength tester, was used most extensively. It is of the pendulum type and was skillfully developed for routine testing.

The pendulum is mounted on a knife-edge and when no auxiliary weight is placed on it to obtain a given load capacity, it is in equilibrium for any angular position. The inertia was kept so low that the precision of the indicated load is 0.5 per cent of the full scale value and 1 per cent. at any intermediate load. The two scales for indicating the load and the extension are linear. One division of the load scale is equal to  $\frac{1}{1000}$  of the auxiliary weight which is attached to the pendulum for a test. Seven such weights are provided to give load ranges from 0 to 1, 2.5, 10, 20, 50 and 100 gm., respectively. Extensions up to 25 mm., magnified four times, can be measured. An electrical trip mechanism is provided to disengage the extension indicator at the instant the specimen breaks.

Specimen lengths ranging from 10 to 100 mm. can be tested dry, and lengths ranging from 10 to 50 mm. can be tested wet. Provision is made for immersing the lower clamp and specimen in the liquid during the entire test. The load-extension curve can also be recorded throughout the test on a chart. Repeated loading and unloading curves can be recorded.

Extensions up to 5 mm. can be measured more precisely optically, with an uncertainty of only 0.05 mm. For this purpose a small microscope is attached to the lower clamp. A scale in the field of the microscope is adjusted so that the zero coincides with a reference mark of the upper clamp. The relative displacement of the two clamps, the extension of the specimen, can be read directly by looking through the microscope and noting the position of the reference mark on the scale. The instant when the specimen breaks can be observed from the sudden movement of the pendulum. This method of obtaining the extension required very careful attention, a technique which can, however, be readily mastered by a qualified operator.

About 50 to 60 fibres can be tested in an hour by an experienced operator. The fibres are inserted in the tester under an initial tension which is about equal to the weight of 500 m. of the fibre. Small

clamps of the required weight are suspended from one end of the fibre while the other end is fastened in the upper clamp. The speed of the lower clamp, which is usually driven hydraulically, is adjusted by means of a sensitive valve so that the duration of the test is  $20 \pm 2$  sec. This adjustment is made by making a few preliminary tests. A constant time interval for making a test has been adopted as standard for tension testing of fibres, yarns and fabrics in Germany. It is one of the specification requirements which differs from the standard practice used in textile testing in the United States.

The Kraiss single fiber tester, which was also used extensively in Germany, is of the constant rate of roading type and is generally known as "Deforden". It was constructed by H. Keyl at Dresden and is basically a balance. The load is applied to one end of the balance by water flowing from a bottle into a vessel at a constant rate. The rate is adjustable by the water head and 2 valves. The other end of the balance beam exerts the load on the fibre. The deflection of the beam is proportional to the extension of the fibre. This extension may be read directly from a scale or it may be greatly magnified optically and read on a scale in a darkened room. The extension can also be recorded against time. A chart is oscillated vertically at a uniform rate and a scribe fastened to the end of the balance beam records the extension against time. The zigzag record of high frequency indicates low extension, whereas a low frequency indicates high extension. A variable frequency is readily detected and indicates a variable time rate of extension. From the time-extension record and from the known uniform time rate of loading, a load-extension curve can be constructed. The latest model, known as "Deforsom", is provided with a valve operated electrically which shuts off the flow of water at the instant the specimen breaks and includes an optical extension indication of 100 magnification and an automatic device for recording the load-extension curve of the fibre. Provision is also made for immersing the fibre in water during a test to obtain the wet strength and extension.

## Yarn Testing

The Schopper yarn tester is of the pendulum type equipped with an automatic recorder of the load and extension. As in the case of the single fibre tester, the yarn tester is provided with a scale indicating the extension of the specimen. The older models were equipped with a gravity trip mechanism which disengaged the extension indicator when the specimen broke. This was not always satisfactory. The newest model is equipped with an electrical trip mechanism operated by the pawls of the pendulum. The pawls were attached to the end of a lever which was pivoted at the other end on the pendulum. When the specimen broke, the pendulum could swing back a small amount until the lever made contact with a stop on the pendulum. This slight motion

of the lever relative to the pendulum closed an electric circuit and caused a solenoid to disengage the extension indicator.

The breaking strength of yarns depends greatly upon the amount of twist, and to facilitate studying the effect of twist the Schopper Co. developed a special lower clamp for the yarn tester. With this clamp the twist in a yarn could be increased or decreased and in fact even reversed in direction after the yarn specimen was inserted in the tester. This permitted studying the effect of twist on the breaking strength and extension of yarn, even if only a small quantity of yarn was available.

### Continuous Yarn Testing

The Frenzel-Hann Universal Yarn Tester applies a constant extension to the yarn as it is passed through at constant speed, while the resulting variation in the yarn tension is recorded. The yarn is threaded first over a length-measuring device and then through the first feed roll assembly. From this point the yarn passes over a second length-measuring device and thence to the final take-up reel.

The instrument was so designed that the speed of yarn passage could be adjusted to any value between 5 and 60 m. per minute. Likewise, through changing the feed roll drives, any extension between 0 and 40 per cent. could be achieved. In the earlier instruments, the tension was measured by means of the conventional angular deflection of a pendulum. This system introduced numerous errors, however, due to the inertia of the assembly, and actually caused variations in elongation due to the movement of the tension measuring roller linked to the pendulum. To overcome these difficulties, a tension measuring system employing the Magnetic Strain-Gauge was developed. In this system the yarn passes over a roller similar to that used in the pendulum assembly; however, in this case the link is attached to a calibrated cantilever, the small deflection of which is a function of the tension. On either side of the cantilever, in the plane of bending, are mounted two inductances which in turn are made to serve as two legs of an inductance bridge. This bridge circuit, in conjunction with auxiliary electronic amplifiers, is connected with an indicating and recording galvanometer. In operation, the tension applied to the roller deflects the cantilever, changing the air gaps between the inductances on each side. This results in an unbalancing of the inductance bridge, which unbalance is amplified and recorded by galvanometer. The cantilever and circuit are carefully adjusted and calibrated so that the resulting autographic record is direct reading.

The general practice followed with this instrument was to test successive lengths of yarn with a definite increase in deformation of 2 per cent. until excessive breakage occurred. During each of these runs, the tension was recorded together with the number of breaks and permanent elongation as calculated from the length-measuring device before and after the feed rolls. Frequently the yarn is passed through the machine a second time to study fatigue and various other effects.

The Dietz Continuous Yarn Tester applied a constant tension to the yarn as it passed through at

constant speed, recording the variation in elongation at this constant tension. A length of 50 or 100 m. was passed through the instrument for a test. The Yarn from the take-up reel was weighed to determine the yarn number, the number of breaks observed, and the variation in elongation recorded. From these data the "ten-break tension," or tension which would cause the yarn to break 10 times in 1000 m. is calculated. It is interesting to note that the value of this "ten-break tension" varies from about 35 per cent. of the average breaking strength for linen to about 85 per cent. of the average strength for silk.

**Abrasion testing.**—The I.G. Farben circular edge tester, measures the resistance of fabrics to abrasion at localized edges such as cuffs and collars. The specimen is mounted with constant tension over a metal cylinder with specially shaped rims of hardened and polished steel. A separate set up is used to apply the tension in mounting the specimen. A similar metal cylinder rests on the specimen under a definite downward force. This cylinder is placed eccentrically with respect to the first cylinder so that the central angle subtended by the intersecting chord is 90°, which is position that produces maximum wear. The diameter of the two cylinder is 17 mm. They rotate in opposite directions with a speed of 3,000 rpm. The test is usually made with the second cylinder bare so that the end of the test is indicated by an electrical signal when a hole is worn through the specimen and contact is made between the two cylinders. The tension in mounting the specimen is adjusted to values between 500 and 2000 gm., and the downward force of cylinder is adjusted to values between 50 and 100 gm. so that the duration of a test is between 20 and 100 sec.

**Flexing-Endurance Testing.**—Closely related to abrasion resistance is resistance to flexing. The instrument built by Schopper, is similar in essentials to the M.I.T. fold tester. The specimen is gripped in jaws, rounded to a radius of 0.05 mm., which bend it back and forth 120 times a minute through 180° until it breaks. The load on falling rips an electric stop switch, and the number of bends is read from the counter. When this device was introduced data were presented showing that, for a given material there is a linear relationship between the logarithm of the number of folds and the tension. This can be extrapolated backwards to give theoretical flexing endurance at zero load. The standard procedure for comparative testing, however, is to use 10 per cent. of the breaking load.

### Spinning Tests

With the intense interest in the development of new fibres, mostly variations on viscose rayon, there was a need for small scale testing of spinning characteristics. This was met by a device, about the size of a small desk, called the "Spinn taxer". The device has two parts, a small card, requiring only 45 gm. (0.1, lb.) of staple, and a single ring spinning spindle. In use, the staple is given a preliminary carding and removed as a fleece, and a second carding, from which it is removed as a top. This is then drafted and spun to a yarn in the range from 6 to 10 English number (cotton count). The breaking strength of the yarn so produced is compared with the breaking strength of similar



yarn so produced from comparison materials. The "Spinn taxer" can be used to study the effect of twist, or of lubricants applied to the staple. The yarns produced were somewhat lower in utilization of the inherent strength of the individual fibers than standard large scale spinning, since standard production usually gives from 50 to 55 per cent. of the fibre strength, but the "Spinn taxer" gives only about 45 per cent. Nevertheless, the value of such a device for development and testing work is obvious.

### Measurements on Staple

A constant care of each of the rayon plants was the maintenance of uniformity of staple. A large fraction of the personnel and space of the rayon laboratories was taken up with this control work. As nearly as could be observed, however, the interpretation of production control appeared to be intuitive, rather than being systematized in the control chart methods which the A.S.T.M. has helped to introduce in America.

Every rayon laboratory was equipped with knives and torsion balances for determination of denier. The staple was combed and stretched across a steel anvil, for example, 2 cm. wide. Two knives operated by a lever cut out the staple at the two sides of the block. The operator would then count out bundles of 50 fibers for weighing to determine denier. The same method was often applied to continuous filament.

One of the few electronic instruments seen in German textile laboratories was an importation from Holland, namely, Gonsalves' device for the determination of denier on an individual filament. This used the basic law of the violine string, that the frequency (pitch) is dependent on the length, the mass of the fibre and the tension on it. Single fibres were attached to the element of the loud-speaker unit, which could be tuned until resonance was seen in the fibre. The tuning control was calibrated to read the frequency. From this the weight per unit length, that is the denier, could be found. The device was calibrated by cutting segments from continuous filament rayon, and an accuracy of 3 to 4 per cent. was claimed.

Alternatively, the methods developed for wool could be used. There are two methods: one, the projection of a large number of cross-sections on the screen the other, the Rapid Lanometer, measuring the area of a bundle of fibres in a square wedge under standard pressure.

Staple length was usually determined in a combing device developed by Johansen. The chief difference between this and the Suter-Webb Duplex Sorter used for cotton, or the Suter Wool Sorter, is in the hinging of the two banks of combs so that the upper bank can be pressed down into the lower, reducing the sorting interval to one half.

### Shrinkage and Creeping Measurements

There appeared to be an emphasis on shrinkage measurements in Germany at the yarn stage, as well as on the Fabric. A convenient device for multiple observation of yarn shrinkage consists of a rack from which ten yarns could be hung in front of a sheet of cross-section paper. Such a rack would be about 2 m. high with the yarns hung for initial

dry observation in the upper half. The lower half was filled by a frame carrying a corresponding number of glass tubes which could be raised to immerse the respective yarns. After a suitable period, the glass tubes could be racked down again and the yarn length observed after drying.

Another device was well adapted to observation of creeping tendency by determining the force of contraction. A battery of levers and indicating pointers is arranged to load yarns with a series of weights, increasing from one lever to the next. The creeping bath is then raised to immerse the yarns. Some of the weights stretch the yarn; some are less than the creeping tension, and from the plot of change of length against load the point at which there is no change of length indicates the creeping tension.

A third device, to study shrinkage from the point of view of yarn uniformity consists in winding the yarn under constant tension on to a cylinder where it is marked by a resistant ink in a strength line drawn on the length of the cylinder. The yarn is then wound off in a skein, treated with water, alkali or otherwise, dried, and wound on to the cylinder again. The markings now produce a spiral, because of shrinkage, and variability along the yarn is revealed by variations in the pitch of the spiral.

### Swelling

The methods to improve the strength and durability of rayon occupied a position only second to methods of production. The leading idea was that if the swelling of rayon in the wet state could be prevented or diminished, the damage in washing and abrasion under moist conditions would be reduced and the useful life extended. A standard method of measuring swelling was in terms of water retained after centrifuging. The essential points of this method were; wetting out for 30 min. in distilled water wetting agent at 20° C.; centrifuging in a basket centrifuge at a specified centrifugal acceleration (600,000 to 800,000 cm. per second per second) for 2 min. The decrease in swelling was calculated as the difference in retention between untreated and treated materials, divided by the water uptake of untreated material.

A technique of estimation of swelling by dyeing was also used which was carried out as follow. Sizing or soluble material is removed from the fabric by appropriate purification. It is then soaked in sodium hydroxide solution of 12° Baume, to 9 per cent. sod. hydroxide cold. The uniform wetting of the piece with alkali is very important and can be improved by addition of a wetting agent. The fabric is rinsed in warm and cold water, acidified in dilute formic acid, rinsed and dried. It is dyed with 0.2 to 0.5 per cent Sirius light blue B, in a ratio of goods to solution of 1:20 to 1:30 with 0.5 gm; Igepon per liter and 5 to 15 per cent salt, at a temperature starting at 40% C. and continuing to boiling. The time varies with the size of the piece from 30 to 60 min. After the dyeing the piece is rinsed and dried. The dyed piece is compared against standards dyed by the same procedure, which have had their degree of swelling determined by the other method. An eight-step scale from no swelling resistance to good resistance is used.

## NOTES and NEWS

### Isotopes as tracers

**W**HEN a small amount of a suitably chosen radioactive or stable isotope is added to one of the substances taking part in a physical or chemical process, it is possible to follow the atoms of that isotope right through the process, to detect their final resting place and to study their distribution by means of their radioactivity or difference in atomic weight. The isotope thus used as a tracer can be employed to furnish information about the nature and the progress of the process in question. This is especially true for process as in which there is an exchange of identical particles. Since such processes are very important in physiology and chemistry, and the tracer method is at present the only known method of demonstrating and investigating such processes, it has found extensive application in these fields. For numerous investigations which in principle could be carried out in other ways, the tracer method is found to be of great value, thanks partly to its extremely high sensitivity and partly to the great ease with which the questions presented can be answered. (Aten Jr. and Heyn, *Philips Technical Review*, 1946, 8, 296).

### Steel Production

One of the impurities present in crude iron is phosphorus, and has to be rendered harmless by adding a slag-forming substance or by lining the crucible with a material that reacts with phosphorus, in the manufacture of steels. A continuous check has to be kept of the amount of phosphorus still present in the molten metal. This can, of course, be done by chemical analysis of samples, but results are obtained much more quickly and easily when a small amount of radioactive phosphorus is added to the melt at the beginning of the process. This is rapidly distributed uniformly throughout the melt, so that the ratio between the natural phosphorus present and the radioactive phosphorus added is the same everywhere. If phosphorus disappears from the melt into the slag floating on the surface or into the lining of the crucible this takes place to an equal degree with the natural and with the radioactive element. The decrease in the percentage of phosphorus in the melt can thus be determined merely by ascertaining the decrease in the radioactive phosphorus. This is extremely simple since it is only necessary to measure the radioactivity of a sample of the melt, which can be done with an electrometer or an electron-counter.

### Detection of mercury vapours

In many factories the workers come into contact with mercury, and it is known how harmful the regular inhalation of mercury vapour can be; a concentration of more than 10-4 gm. of mercury per cm. meter of air is known to be injurious to health. It is difficult to detect the presence of mercury in such minute proportions, because chemical

analyses are unavailing in such cases. In a certain case which occurred in the manufacture of tubular luminescent lamps in an American factory, where the lamps were filled with mercury vapour at a low pressure, a glass side-tube containing a small drop of mercury had to be "blown" on to the lamp, and inevitably the glassblower inhaled a very small quantity of mercury vapour into his lungs. In order to determine how much was inhaled a number of tests were carried out with a volume of 2 liters of air that had been in contact with drops of mercury under exactly the same conditions as in the manufacturing process, this being drawn off by suction and passed over a metal plate kept at the temperature of liquid air. Practically all the mercury in the air condensed on the plate.

The mercury used for the experiments contained a known, small percentage of a radioactive mercury isotope. The radioactivity of the plate, which was quite simple to measure after the experiment, gave an indication of the amount of mercury contained in the air which had passed over the plate. In this way a concentration of  $5 \times 10^{-6}$  gm. per cm. meter of air could be detected. The average mercury concentration was about 10-5, and in one case about  $4 \times 10^{-5}$  gm. per cm. meter, from which it was concluded that in the manufacturing process in question the glassblower ran no danger of poisoning.

### Structure of metals and alloys

When a piece of metal is fused with a radioactive lead isotope in an atmosphere of hydrogen and then allowed to crystallize again, there are two possibilities. In some metals, such as thallium and magnesium, lead is soluble to a considerable percentage, the lead atoms being uniformly distributed in the grains and a polycrystalline metal obtained. In other metals such as bismuth, tin, antimony, silver, gold, copper and nickel, in which lead is practically insoluble, the radioactive lead is situated on the boundaries of the grains. When a microscopic preparation of the metal is made and laid on a photographic plate for several hours, the plate after development will be found to be blackened at those places where it lay against radioactive particles, i.e. where lead has been deposited. By means of such an "autoradiogram", it is possible, in the first place, to ascertain whether and to what degree the added lead is soluble in the metal; if the lead is entirely dissolved the entire surface of the photographic plate is uniformly blackened, if the lead does not dissolve, or dissolves only partially, the radiogram shows up the boundaries of the grains. Thus the isotopic method can furnish valuable information on the changes taking place in the structure of the metal in recrystallization and in rolling.

### Friction and Hardness of Surfaces

Friction between two metal surfaces is due partly to adhesion, the result being that when the surfaces

slide over each other, extremely small particles of metal are torn out of one and taken up in the other. This exchange of metal may take place to such an extent that two surfaces become, as it were, welded together (the familiar seizing). The quantity of material thus transferred from one metal to the other is a measure of the contribution of this effect to the total force of friction. In general the quantities involved are so small, that they can be hardly detected by chemical tests. If the surface of one of the metals is "activated", i.e., if it consists for a small part of atoms of a radioactive isotope of the metal, and this surface is made to slide over the second, non-activated metal surface, the latter also shows a certain amount of radioactivity. Amounts as small as 10-10 gm. of transferred metal can be detected in this way, and, what is more, by means of a radiogram the distribution of the transferred material on the surface can be studied. The influence of factors, such as pressure, hardness of the surface, etc. on the transfer of material can be studied, as also the effect of a lubricant, can be studied of this technique.

From these examples, the significance of the tracer method becomes at once evident. Processes in which there is an exchange of identical particles are common in nature not only in chemical and metallurgical processes, especially in the physiology of plants and animals. The tracer method, which offers the only method of approach, has been used on a large scale for the investigation of such processes. An important point in the employment of a radioactive isotope is that it can be localized easily when mixed with a different or a chemically identical substance, and its distribution can be determined.

## Petroleum Industries

The tracing of stoppages in an oil pipeline provides an important application of the use of radioactive indicator. In the periodical cleaning of the walls of the pipe a screw-shaped scraper is placed in the line and pushed along by the pressure of the oil itself. If the scraper gets jammed somewhere, it has to be located as quickly as possible, in order to open the line at that point and remove the accumulated deposit. With the help of a radioactive indicator this localisation is astonishingly simple. A scraper is used that contains a little radioactive material emitting  $\gamma$ -radiation, which easily penetrates through the walls of the pipe and can be detected with a somewhat modified "electron counter". Another similar application in the petroleum industry is for determining the setting depth of the cement that is pumped in behind the casing of an oil well. A radioactive mineral, carnotite, is mixed with the cement and when a counting instrument is lowered into the drill hole it indicates a strong radiation at the level of the cement.

## Quantitative Measurements

In order to determine the efficiency of a fog, smoke or dust filter, it is necessary to measure the very small quantities of fog-forming or other substances retained by the filter. As fog-forming substance, tricresyl phosphate containing radioactive phospho-

rus is used. When this is passed successively through several filters, their efficiency can be judged by comparing the intensity of their radioactivities.

Radioactive isotopes provide excellent means of measuring very small solubilities or very low vapour pressures. The vapour pressure of thorium acetyl acetate, for example, has been determined by preparing a sample of the compound with a strongly radioactive thorium isotope, saturating a given volume of nitrogen with the vapour of the compound and passing the gas through acidified alcohol, in which the compound is absorbed. The thorium concentration can then be calculated from the radioactivity of the liquid and from that the amount of vapour in the given volume of nitrogen.

Radioactive isotopes find many applications in microchemistry. Radioactivity has made possible the investigation of the chemical properties of the elements "43" and "85" and of several "trans-uranium", elements which are not found in nature but from which radioactive isotopes can be prepared artificially in imperceptibly small quantities.

Important perspectives are opened by the application of radioactive indicators in chemical analysis, so often constituting daily routine work in technology. As an illustration the determination of bromide in a mixture of a bromide and a chloride may be given. A complete separation of the two compounds is difficult. If, however, a little radioactive bromine (in the form of the compound in question) is added to the mixture, only a partial separation of the bromine is sufficient. Due to the homogeneous mixing it is known that the ratio between the bromine is equal to the ratio between the radioactivity separated out and the original radioactivity. Since the latter ratio is easily determined, it is possible to calculate the desired total content of bromine directly from the amount of bromine separated out.

An application in biology somewhat resembling the above is the determination of the total amount of blood in an animal. If a solution of some substance containing a known quantity of radioactive atoms, is injected into the test animal intravenously and given time to distribute itself homogeneously throughout the whole circulatory system, a small sample of the blood suffices for the estimation of the total amount of blood in the animal. The fact that it is practically unnecessary to interfere with the normal life of the test animal is of importance for many investigations, especially those of a pharmacodynamic nature.

Non-radioactive isotopes can also be employed as tracers. The atoms of such isotopes are recognizable (labelled) by their different atomic weight and the properties connected therewith, such as specific weight, velocity of diffusion, heat conductivity, etc. The most important tracers of this class are "heavy hydrogen" (deuterium) of atomic weight 2, the oxygen isotope of atomic weight 18, and the nitrogen isotope of atomic weight 15. With such stable isotopes are employed measurements of density, or the like are made to reveal their presence. These measurements are generally less simple than the measurement of radioactivity they are also less sensitive. They are employed in studying processes

of exchanging where no suitable radioactive isotopes can be found.

### Synthesis of Valine

There is no known method for isolating the natural form of valine from protein hydrolysates. The cost of the synthetic product is prohibitive. A systematic investigation was undertaken at the Department of Biochemistry, Laval University, Quebec, to synthesise *dl*-valine by the Strecker method using isobutyraldehyde as starting material. The potassium cyanide-ammonium chloride procedure, as modified by Gandry has been found most satisfactory for the large scale preparation of the amino acid, considering the yield purity and the time required (*Can. J. Res.*, 1946, BDB, CJ1).

A mixture of ammonium chloride (60 gms.), potassium cyanide (66 gms.) water (400 cc.) and conc. ammonium hydroxide (155 cc.) is placed in a 3-necked flask (1 litre) immersed in cold water. Freshly distilled isobutyraldehyde (91 cc.) is added drop by drop. In the course of this addition, which should take about 30 mins. the temperature reaches about 40° C. Ethyl ether (100 cc.) is then added, and the mixture extracted with ether is removed *in vacuo* at room temperature and the residual liquid treated with conc. hydrochloric acid (500 cc.). After standing overnight, the mixture is boiled under reflux for 24 hours, and then evaporated to dryness *in vacuo* on a water bath. Water (300 cc.) is added to the residue, and boiled under reflux with charcoal. It is filtered and the filtrate evaporated to dryness *in vacuo*. The solid residue is taken up with absolute alcohol (500 cc.) boiled under reflux for a few minutes, cooled and filtered. Pyridin (100 cc.) is added to the solution, and the mixture stored in an ice box overnight. The valine which separates out is filtered and washed with absolute alcohol until free from pyridin.

The study reveals that the experimental conditions best suited for the synthesis are: (1) freshly distilled isobutyraldehyde should be used to get maximum yields (2) the reaction between aldehyde, cyanide and ammonium salt is best carried out at room temperature; cooling is recommended while the aldehyde is added to the mixture of cyanide and ammonium salt to avoid overheating, but no further cooling is necessary (3). Excess of potassium cyanide or ammonium chloride does not increase the yield. The presence of an excess of ammonia in the reaction mixture significantly increases the yield; the presence of a little ether is also beneficial (4) Hydrolysis of a mononitrile with hydrochloric acid is recommended as it enables easier separation of the amino acid from the reaction mixture. (5) The amino acid can be isolated by glacial acid (Marvel, *Org Syntheses*, 1940 BJ, 105-108), but the isolation is more easily and rapidly effected by dissolving the *dl*-valine hydrochloride in absolute ethyl alcohol, filtering the insoluble salts and precipitating the valine with a slight excess of pyridine.

### Chemicals from Fatty acids

A series of new products of industrial value derived from fatty acids are described in the *Chemical Trade Journal* (1942, 120, 373).

The initial step in the preparation of fatty acid derivatives is to separate a fatty acid mixture into its component parts. This is accomplished by either continuously vacuum distillation or solvent crystallisation. Fatty acid derivatives can be roughly divided into two types. The first comprises those compounds prepared by a modification of the hydrocarbon chain and the second type are those prepared by the chemical reaction of the carboxyl group. The second method of attack can yield literally thousands of compounds, many of which have an important and far reaching industrial uses.

When fatty acids are treated either in the liquid or in the vapour phase with ammonia at elevated temperatures, they are first converted to amides which yield nitriles, possessing melting and boiling points appreciably below those of the parent acids. The presence of a triple-bonded nitrogen renders them chemically active. They serve as plasticisers for a variety of polymers, the nitrile group rendering them compatible with many substances. Certain of these compounds particularly, the lauronitrile, are highly repellent to insects. This property is also possessed by the higher homologues in spite of the fact that they are essentially odourless to human beings. A distinctive property of the long chain nitriles is that they can be thermally cracked without loss or modification of the polar group by processes similar to those employed in the petroleum industry for cracking hydrocarbons. The products consist of both saturated and unsaturated shorter-chain nitriles and hydrocarbons from which alcohols, alkylhalides, alkyl thiocyanates and a wide variety of aliphatic compounds can be easily prepared.

Hydrogenation of the nitriles yields amines or alkylammonias, the salts of which are important surface-active agents. The amine salts belong to a class known as cationic colloidal electrolytes.

The unusual behaviour of the amine salts is due to the fact that the long-chain colloidal ions possess a positive charge. They are strongly adsorbed upon silica and siliceous minerals, glass threads and fibres which contain an adsorbed film of amine possess many times the tensile strength of unlubricated fibres. A trace of amine adsorbed upon a siliceous aggregate prevents the aggregate from working out of asphalt, so that roads prepared with asphalt containing a small amount of amine or made with a treated aggregates, possess much longer lives than untreated roads.

The unusual adsorption characteristic of amine salts have led to their use as flotation agents. The amine salts generally reverse the usual flotation procedure in that it is the siliceous mineral or "gangue" which floats. It is because of this phenomenon that a major portion of the phosphate rock can be separated by flotation. Amines have also found important uses in the beneficiation of iron ores. They form complexes with salt of heavy metals which show marked insecticidal and fungicidal properties. Such compounds are indicated for use as disinfectants for a variety of agricultural purposes. When the tertiary amines are treated with alkyl halides or sulphates or certain other esters they are converted into quaternary ammonium compounds. They all possess extremely high bactericidal properties. Fish



packed in ice containing a trace of these compounds can be shipped over long distances without deterioration.

### Specimen films

A film of cellulose nitrate or similar material is laid on a water surface and transferred to a specimen holder. A thin layer of beryllium or aluminium is deposited on the film from the vapour phase, after which the cellulose nitrate is dissolved away. Only the metal film then remains, and its lack of visible structure can be checked by photographing in the electron microscope. The durability and resistance of the layer to chemical attack are high. A matter of great importance in the study of specimens in which completely opaque particles occur is that the metal film readily conducts away absorbed energy. With such particles, however, films of cellulose acetate or similar materials are easily broken. Beryllium and aluminium films of as little as  $20^{\circ}$  A. thickness (diameter 100), and aluminium films  $12^{\circ}$  A. in thickness have been prepared by this procedure. (*Nature*, 1947, 159, 370).

The absorption being slight in this film it is possible to make a closer study of minute structures, such as large molecules and their shapes. By taking a series of photographs it is possible to follow the reaction of one and the same particle of the substance when subjected to different chemical treatments. It is not difficult to number the holes on the preparation holder in such a way that any given hole can be easily located.

An experimental procedure for studying the structure of clay has been worked out (*Nature*, 1947, 159, 354), which can also be employed for the study of diatoms. A fresh fracture surface is prepared in the clay, and on this surface are spread immediately some drops of a liquid when surface tension is lower than that of water, for example, amyl acetate in which cellulose nitrate has been dissolved. The liquid then enters the moist surface of the clay entering even the slightest irregularities. The evaporation of the solvent leaves a coat of surface which can be peeled off. It is found that diatoms lying in the clay surface accompany the cellulose acetate film when it is drawn away. Since the cellulose nitrate encloses the diatoms within an envelope of sufficient strength, they can readily be removed from the clay. Even diatoms of extremely fine structure or of great fragility can be loosened and transferred to an aluminium or beryllium film for observation in the electron microscope. In cases where the diatoms consist of adjacent fragments, they also can be studied in the microscope with their original relative positions in the clay undisturbed, which has hitherto been impossible. The high resolving power of the electron microscope employed makes it possible to study diatoms of submicroscopic size. Diatoms have thus been seen which have hitherto been unobservable on account of their minute dimensions. Of interest is the detailed study of the structure of diatoms, especially the structure of shells which is sometimes so fine that magnifications of more than 100,000 are necessary to disclose it.

### Applications of Powder Metallurgy

Powder metallurgy is finding a large variety of applications in manufacturing operations according to a report from the British Commonwealth Scientific Office. Among the operations listed are: Tungsten filaments of incandescent lamps; Molybdenum wire and foil, for W/T valves; Iron, nickel and molybdenum for low hysteresis coil cores; Copper for commutator segments, carbon brushes and contacts; "Alnico" magnets; Metal-silica friction surfaces for breaks; Small low stress parts of intricate form such as are used in instruments; and powdered metal flake for paint pigmentation.

Powder metallurgy also finds important applications in aircraft manufacturing processes such as; carbide tipped tools for machining hard materials; Powdered metal and flux rods for welding; Pressing plastics and small brass parts like "Zip" fasteners in dies made from metal powder.

The American aircraft industry is extensively using, the so called "oilless" bearing of which a large lumber may employ as many as 4000.

The operations for making parts by powder metallurgy process are simple. The consist mainly of: Compacting, i.e. pressing together of the mixed powders in a die at room temperature at pressures varying between 2 to 60 tons per sq. inch; Sintering, i.e. heating of compacted powder in a controlled atmosphere, the heating temperature being less than that of the most fusible metal constituent.

The characteristic property of parts made by the powder metallurgy process is that the constituent particles remain discontinuous in the body of the material in the form of voids. In certain cases these voids are filled with a different metal to change the properties. In general it has been found that the powder process furnishes materials which when compared with the bulk metal have smaller density and less tensile strength.

The advantages of the process is that it makes it possible to produce materials in useful forms where the extremely high melting points make the usual methods impracticable. The fabrication of tungsten, (mp.  $3360^{\circ}$  C.) and tantalum (mp.  $2,800^{\circ}$  C) articles may be cited as an examples. Other advantages are: Parts can be made from aggregates or mixtures of metals which do not form satisfactory alloys such as copper and tungsten. It is possible to prepare with a very high degree of accuracy alloys which involve difficult machining processes. An example of this is the making of alloy known as "Alnico". With metal powders the individual properties of the constituents materials can, if required, be maintained. Thus is copper-tungsten mixtures, the high electrical conductivity of copper is combined with the wear resistance of tungsten; it is possible to compact non-metals with metals. The production of carbide tipped machine tools and copper carbon brushes are instances; by a suitable grading of powder and control of consequent compacting and sintering processes, it is possible to obtain a variety of useful products like porous bushes and metallic filters; powder metallurgy allows the economical production of parts where small sizes will make machining an uneconomic proposition.



Of the metals employed, aluminium powder is being commercially employed in the manufacture of small high permeability permanent magnets of the 'Alnico' class. Experimental work is in progress in America for the manufacture of porous aluminium bearings containing lead and tin. Bronze powders are finding considerable application in the manufacture of "oilless" bushes containing about 25 per cent. voids. Both brass and bronze bearings are extensively used for making small parts required to close tolerances. Chromium powder is finding use in making stainless steel parts and cobalt in the manufacture of sintered carbide tools. Electrolytically produced copper powders are extensively used by the electrical industry chiefly in the manufacture of commutator segments, resistance rings of A.C. motors, copper-carbon bushes and contacts. Copper is a constituent of a friction material containing copper, iron, lead, graphite and silica which has considerable promise in brake and clutch lining. Iron oxide, besides its use in friction materials, is being used for the manufacture of iron-graphite bearings and oil pump gear wheels. An experimental bearing is made from iron powder in which about 30 per cent. of the space is filled with habbitt metal. Iron powder has also been used in the manufacture of filters which, allow light petroleum fuels to pass but hold back water. Such filters are also used for filtering caustic liquors. Iron powders have been used for a long time now in the manufacture of low-loss cores for many high frequency electrical devices. Lead powders have been used in the manufacture of lead bronze bearings.

Molybdenum, is used in the manufacture of wireless transmitter valves. Nickel powders enter into the composition of permanent magnetic nickel alloy and low-loss high permeability cores. Powder metallurgy process has been suggested for making silver table wares. Such wares could be made with about 50 per cent voids and should thus be cheaper than solid silver articles.

### Industrial Applications of Luminescence

The application of luminescence to industry has been rendered possible by improvements in the performance of ultra violet lamps. The modern forms of mercury discharge lamps operate at a high efficiency (38 to 45 and more lumens per watt), have a long life (1,500 hrs.), a high overall power factor (0.75 to 0.98) and are electrically as well as mechanically well fitted for somewhat rough handling in workshops. *Electronic Engineering*, Dec. 1946).

The imperviousness of mercury vapour to ultra-violet light is utilised in the detection of traces of mercury in an ore in the ratio of 1: 10<sup>-5</sup>. An ore, suspected to be mercuriferous, is powdered and heated strongly. The suspected vapours are allowed to pass between a source of ultraviolet source and the screen. Vapours other than mercury cast no or only a very hazy shadow on the screen; mercury vapour even in as low a concentration as 10<sup>-5</sup>, casts a very dense shadow on the screen.

The methods of crack detection in materials have been enriched by the use of fluorescent substances. According to the shape of the test object some methods show advantages over others. Small plain

objects are tested by the "duochromatic" method. A 1 per cent. solution of 3:6 dihydroxy phthalimide in petroleum ether is colourless in day light or in incandescent light but shows a strong blue colour when irradiated with long wave ultraviolet (3650 A.U.). A drop of liquid is applied to the test object (or the object can be dipped in the fluorescent indicator). The volatile liquid vapourises within a few seconds, but is retained longer in curves, cracks, flaws, etc. When viewed in ultra violet light after the liquid has volatalised the dry surface will appear yellow, the cracks standing out in bright blue against the background. This method is often employed in grinding, polishing and honing workshops in order to detect cracks in crankshafts and other vital gear. Large flat surfaces of metallic or other smooth bodies can be inspected by the "brushing" method. An inorganic fluorescent powder is brushed over the surface to be inspected and then is wiped clean. A few grains in the crevices give away the position of the flaws.

The high pressure, high intensity mercury arc is an efficient source of bactericidal irradiation. This is used in the brewing industry to sterilize water.

### New alloys and jet propulsion

Britain's spectacular success in jet propulsion is mainly due to the metallurgical discoveries which made the gas-turbine engine a practical proposition. For years the efforts of scientists and engineers to make a working gas-turbine were defeated by the inability of any known material to stand up to the unprecedented combination of heat and stress to which the turbine blades are subjected.

The blades have to revolve at about 15,000 revolutions per minute, involving stresses of some 16 tons per square inch at temperatures over 700° C. These temperatures, at which the blades are literally red hot, combined with the stresses imposed, cause "Creep", which means that turbine blades begin to stretch and ultimately break. The restriction of "creep" to a minimum was one of the principal metallurgical problems to be overcome, and involved the use of elaborate testing apparatus, which can measure changes in the length to within one-millionth of an inch (0.000025 mm.).

Intensive research in the Mond Nickel Company's research Laboratories led to the development in 1940 of *Nimonic 75*, a new nickel-chromium alloy, which fully satisfied the specifications of the Air Ministry. It was not long, however, before progress with jet engine design led to the demand for a still stronger material and, as a result of further research, a greatly improved alloy, *Nimonic 80*, was made available late in 1941. Ever since, it has been the standard material for the blades of all British-built gas turbine engines. Meanwhile, research on *Nimonic 75* in the form of sheet has led to its adoption as the standard British material for flame tubes. It is also used for other jet engine components.

### Conduction of Electricity through Cellulose

The conductance of cellulose sheets containing more than 1 per cent of salt depends mainly on the

moisture content irrespective of the kind or the amount of salt present; the nature of the salt present in low concentration (Sullivan, *Shirley Inst. Memoirs.*, 1946, 20, 109-143).

When an electrical current is passed through salt impregnated cellulose sheets, it is found that the ions migrate along the sheet. The conduction of electricity through conditioned cellulose is essentially ionic.

Mobilities of hydrogen and hydroxyl ions in cellulose sheets have been studied by using pieces of metal impregnated with an alkali salt and a mixture of indicators, the potential gradient being kept as uniform as possible. Mobility is found to depend on the moisture content and varies a thousand fold between 35 and 29 per cent relative humidity. The relationship between mobility and moisture content is found to be similar to that existing between conductivity and moisture content, and this indicates that the electrical conductivity in conditioned cellulose is mainly ionic.

### Fireproofing of Fabrics

A report published by the Department of Scientific and Industrial Research, London, ("The Fireproofing of Fabrics, 1947) discusses fireproofing of fabrics by water soluble and water insoluble deposits. Fireproofing with water soluble deposits is cheap and easy, where the protected material is not subsequently exposed to the action of water. Some quite simple and readily obtainable inorganic salts deposited in relatively small amounts are shown to be effective in preventing flame and glow. Mixtures of substances are better than single substances and borax is an important constituent of them all. An effective mixture is made up of boric acid and sodium phosphate. Water insoluble deposits are generally obtained by double-decomposition and in a few cases impregnation in single solution followed by drying and immersion in boiling water. Two methods suitable for the flame glow proofing of fabric consist in the deposition in balanced amounts of the oxides of iron, tin and tungsten, followed by silica. Another successful procedure is the deposition of ferric oxide, stannic oxide and chloro-polyvinyl chloride.

The great fire hazard associated with Kapok is due to the rapidity with which the flame travels over the surface followed by flameless combustion which destroys the whole mass. Two methods are described which are used for fireproofing of Kapok. In one method, an insoluble deposit of stannic oxide, tungstic oxide and alumina is after-treated with boric acid. The other method employs a simple deposit of alumina after-treated with boric acid. Fireproof finishes for aeroplane fabric involve consideration of the nature of the tautening dope with which the fabric is treated. No satisfactory fireproofing treatment was found for fabric coated with nitre—or acetyl cellulose dopes, but with dopes made from chloropolyvinyl chloride previous treatment of the fabric with 20 per cent. by weight of ferric or stannic oxide gave a permanent fireproof effect.

### Pectin Fibres

Investigations at the Western Regional Labora-

tory. (U.S.A.) on the use of pectonic acid as a raw material for textile fibre production has led to promising results. The work is still at its early stages. By spinning a 1 per cent. solution of low methoxy! pectonic acid (half acid and half sodium salt) into a 1 per cent. solution of calcium or magnesium chloride, fibres have been obtained which have a dry tenacity of 40,000 to 50,000 lbs. per sq. in., i.e. comparable with nylon.

The normal procedure adopted for the isolation of pectins and related acids is to grind orange peel with 3 times its volume of water, acidified to pH 2 and boiling for 30-60 mins. to give a 1 per cent. solution of methoxy pectic acid which may then be treated in one or two ways. It may be concentrated five-fold under vacuum and precipitated with ethyl alcohol, or it may be precipitated with aluminium hydroxide at pH 4.2, the precipitated aluminium pectin complex ground and washed with alcohol hydrochloric acid (50 per cent. alcohol rising to 90 per cent.).

The enzymic de-esterification of pectin is smoothly accomplished by the esterase contained in the orange peel. The pectinic acid obtained has a molecular weight of 70-90 thousand and is very suitable for extrusion to fibre.

### Spectroscopy of micro quantities of tissue fluids

The spectroscopic examination of fluids and extracts from small animals is often difficult because the volume of material available is too small to give a liquid layer thick enough to show up characteristic bands when the ordinary tubes or cells are used. A simple device for the *New Zealand Journal of Science and Technology*, (1946. 28B, 186). A plunger type of apparatus with which a 5" light-path was obtained with 5cc. of solution was described by Harrison (*Biochem. J.*, 1938, 32, 933) but this does not entirely fulfil the required purpose. In the new device a 5" light-path can be obtained with 0.75 cc. solution, and a different principle, that of raising and lowering the liquid column by suction, is used to vary the thickness of the solution.

The observation cell consists of a piece of thick-walled glass tubing, a broken 1 cc graduated pipette with lumen of 3 mm. dia. is suitable, but tubing of smaller bore can be used if greater sensitivity is required. This is mounted vertically and cemented at the top into a metal head. The head is turned from a brass rod and contains a glass disc set between two rubber washers held in place by a screw-cap. The glass disc can be roughly cut from a microscope slide with scissors under water. A small side-tube enters the head just below the glass disc, and is connected by rubber and glass tubing to the pipette teat. The lower end of the vertical glass tube dips into the flat bottom vial (1 cm. dia) which acts as the solution container, and this is set directly above a hole in the metal lamp housing. A 36 watt, 12 volt automobile lamp is used as light source, but 25-watt mains operated frosted lamp is satisfactory.

The glass tube rests on the bottom of the solution container and also passes through holes in two

wooden supports so that it can be raised to remove the container. The spectroscope is usually held by hand over the lumen of the tube during an observation, but it may be supported in a wooden clamp, which can be swung aside to allow the tube to be raised. A wooden burette stand makes a suitable base and upright on which to mount the apparatus.

Examples of the application of this apparatus include the detection of phenothiazine derivatives in tears and aqueous humour of calves and sheep and differentiation between phylloerythrin and coproporphyrin in small samples of blood and urine.

### Weighing of Textile Fibres

A microbalance of the cantilever type for the weighing of single centimetre lengths of cotton, entailing the determination of weights of the order of  $1000 \times 10^{-8}$  gm; with an accuracy of about  $3 \times 10^{-8}$  gm. is described in the *Shirley Institute Memoirs* (1947, 21).

This new balance is entirely different in principle from the torsion type and depends upon the deflection of the end of a fine phosphor-bronze ribbon mounted horizontally and secured at one end. When a specimen is placed on the free end of the cantilever beam, its weight is indicated by the displacement, which is measured by means of a small microscope containing a scale in the eyepiece. Provision is made to bring the end of the unleaded cantilever into coincidence with the zero of the eyepiece scale, so enabling the displacement to be measured as a direct reading.

The balance consists of a phosphor-bronze ribbon mounted with picein wax on the end of a flat steel spring (about 0.25" wide) the other end of which is riveted to a brass rod. The second end of the brass rod is secured in the top of a slotted pillar. The free end of the flat steel spring presses down on the top of an adjustable stop passing through a bent lever. One end of the lever is pivoted to a second pillar. The other end of the lever rests on the top of a screw which passes through a brass collar in the base of the instrument and has a round flat head, the rotation of which raises or lowers the lever and produces a corresponding movement at the end of the phosphor-bronze ribbon. For sensitive balances, used for the weighing of single fibres, the free end of the phosphor-bronze ribbon is bent into a V-shaped hook. In the coarser models, the end of the ribbon is turned up for about an eighth of an inch, and a small hook is suspended by a piece of fine phosphor-bronze ribbon such as that used for galvanometer mountings. In both models the free end of the cantilever arm is observed through a small pocket microscope inside the eyepiece of which is a numbered scale divided into equal divisions. A small glass window is let into the back of the instrument to provide illumination for observation, and a piece of white card may be placed outside this window to reflect either daylight or artificial light through the aperture. At the side of the case and hinged to the top is a brass flap which is raised when the specimen is being placed on the hook and lowered to protect the instrument from draughts during weighing.

With balances designed for the weighing of single fibres, small wire riders carefully calibrated against N.P.L. standards are used. This method is not completely satisfactory because the wire weights are so small that they are very easily mislaid, or blown away by accidental draughts (The weights are of the order of 0.01 mgm.).

A method for the calibration of single-fibre balances makes use of fibres as standard weights. For this purpose 3-denier filament nylon is suitable. A bundle of about thirty 2-centimetre lengths of 3-denier nylon may be weighed together on a balance suitable for routine average fibre weight per centimetre determinations, with a total range of from 0 to about 0.3 or 0.4 mgm. The 2-centimetre lengths of nylon are then weighed separately on the single-fibre balance. The factor of the balance may then be determined from the total weight of the bundle and the sum of the separate deflections. If the weights of the separate 2-centimetre lengths are computed these fibres may be kept for use as standard riders at a later date.

The cantilever balance is being used in studies of the rate of growth development of the cotton fibre, and in the associated studies of fibre fineness. The balance may also find use in studies on the variation in the weights of short elements along yarns of continuous filaments. For such studies, balance having a sensitivity corresponding to a full-scale deflection of about 0.25 to 0.5 mgm. is suitable. As an illustration of this use, a length of 6' of a single filament was separated from some 60-denier, 15-filament acetate yarn; centimetre lengths were cut at 4" intervals along the filament and weighed in succession on a sensitive model of the balance.

### Temperature and Humidity of Skin and garments

A method for measuring temperature and relative humidity of skin and clothing of a human subject has been recently described (Ogden and Rees., *Shirley Inst. Memoirs*, 1946, 20, 163). The method is the result of a series of studies on the resistance of flow of heat and water vapour through a fabric.

Copper-constantan thermocouples (34 S.W.G.) are used for the measurement of temperature of clothed parts of the body and a surface pyrometer for the temperature measurement of exposed parts. A rectangular piece of thin grey cotton fabric is boiled in 1 per cent. lithium chloride, dried and mounted with foil having a narrow strip of the fabric  $1'' \times \frac{1}{8}''$ . All the weft threads in this strip are removed and this, then, constitutes the humidity sensitive cell. The principle of this cell is that the electrical resistance of the threads of the treated cotton fabric varies with their moisture regain and hence with the relative humidity of the atmosphere with which they are in equilibrium. The calibration of the cell is carried out by measuring its electrical resistance when suspended in an enclosure with controllable temperature and relative humidity. There is a logarithmic relation between the resistance of a typical cell and the relative humidities at temperatures between 27° and 40° C. The pre-

sence of an hysteresis effect in this relationship is admitted.

A test-garment, resembling a sleeveless vest, is made from British Army battledress fabric (14.1 oz. per sq. yard) in two halves capable of being laced together under the arms to give a tight fit and is used for determining temperature and relative humidity gradients in the clothing with the help of pairs of thermocouples and humidity sensitive cells stitched through the garments at the desired places. The standardisation of the test garment is made with Peirce and Rees' apparatus for measurement of heat flow through thick textile fabrics (*Shirley Institute Memoirs*, 1945, 19, 341).

In comfortable equilibrium with the surroundings,  $-10^{\circ}$  C air temperature and 60 per cent. R.H., the skin temperature of the trunk of a subject is stated to be  $33^{\circ}$  C., and the relative humidity at the skin surface, 30 to 40 per cent. From the measurements of thermal resistances of various garments worn by the subject and of the air-spaces between the garments, the rate of production of heat within the body of the subject, or "metabolism" as it is designed, is calculated. For a sitting resting subject, the value of metabolism is given as 58 watts per sq. metre (or 50 kilocalories per metre per hour).

The water vapour resistance of a fabric is a concept developed by Peirce, Rees and Ogden (*Shirley Inst. Memoirs*, 1944, 19, 51) on considerations analogous to the thermal resistance of a fabric referred to above; the value of this quantity for the fabric composing the test garment is 5.6 units. From this known value, the water vapour resistance of other garments and of the external air can be calculated. For a shirt the mean value for three positions is 2.9 units and for battledress 5.6 units compared with 5.6 units for the test garment.

S. RAJARAMAN

### Iso-Electric Point of Silk Fibroin

Studies on the cataphoresis of fibroin prepared from *Bombyx mori* hilk have been recently reported (*Shirley Institute Memoirs*, 1946, 20). The wide variation in the values of iso-electric point obtained by previous workers is attributed to the impurity of the protein employed and to the presence of protein degradation products. The cataphoretic mobilities of water-soluble fibroin are

measured in the presence of red gold sol by the moving boundary method, the range of pH, of the final solution being 2.0—8.5. When the values of cataphoretic mobilities are plotted against those of pH, the points lie approximately on a straight line. The iso-electric point interpolated from these points is 3.2. No significant change in mobility occurs with change in type or concentration of buffer.

C.N.

### New Antibacterials for Tuberculosis and Typhus

An extensive search among mushrooms and toadstools for antibacterial substances at the Institute of Medical and Veterinary Science, Adelaide (South Australia) has resulted in the isolation from an edible variety or mushroom (*Psalliota*) and a toadstool (*Cortinarino rotundisporus*), antibacterial substances having activity against a wide range of bacteria, including the one that causes typhus. The antibacterial substance from *Psalliota* killed tubercle bacilli in vitro.

The dried extract from the mushrooms is obtained by grinding the material, straining the thick brown fluid through muslin, and drying the extract by freezing in a vacuum at a temperature of  $-20^{\circ}$ C. The yield from 250 lbs. mushrooms is about 0.5 oz.

### Nitrogenous Fertilizers

In the Annual Report, for the year ended June 30, 1946, of the Council and Executive Committee of the British Sulphate of Ammonia Federation, Ltd. Particulars are given relating to world production and consumption of fertilizer nitrogen (in metric tons of nitrogen, *C.T.J.* 1947, 120, 95).

A noteworthy feature of wartime fertilizer practice in U.K. is the increase in the ratio of nitrogen to other plant foods consumed as fertilizers. The ratio of nitrogen to phosphate increased from 1:2.2 in 1938-39 to 1:1.9 in 1943-44. During this period the nitrogen content of compounds was raised from an average of about 3.5 per cent. to 5.25 per cent. There is a marked increase in the use of nitrogen fertilizers in the top dressing of cereals. In previous years nitrogen fertilizers were used mainly on tillage crops. Official statements in U.K. on post-war agricultural policy suggest a gradual switch over from the production of crops for human consumption to production of grasses for livestock.



# REPORTS FROM STATES & PROVINCES

## JAIPUR

### Industrial Progress

The last few years the Government of Jaipur have introduced many changes in industrial policy to encourage private enterprise and to strengthen existing industries. Grants of land for industrial purposes at concessional rates, the laying out of suitable sites for large and medium scale industries of Jaipur City and Sawai Madhopur, exemption from export and import duties and the provision of cheap electricity are some of the facilities offered to prospective industrialists.

The State is rich in minor products such as copper, mica, magnasite, marble, kaolin, red and yellow ochres, calcite and soapstone. The *Jaipur Mineral Development Syndicate* at Dausa produces powdered talc required in the manufacture of cosmetics for export to foreign countries. The *Hardwari Mineral Development Syndicate* exports graphite, felspar, flourspar, red and yellow ochres and lime-calcium in pulverised form. The *Jaipur Mineral Corporation* is exploiting the copper ore of Khetri. The Rajputana Corporation, Ltd., is engaged in mica mining.

The *Jaipur Metal Industries, Ltd.*, produces non-ferrous metals and alloys such as copper, brass, gun-metal, naval brass, leaded brass and aluminium bronze from mixed factory and domestic scrap metal. The manufacture of arsenical copper of the required specifications for railway locomotive boilers is one of its major achievements. There are 14 electroplating concerns, 5 automobile repair units and a gas-plant factory in the State. The *Man Industrial Corporation Ltd.*, has planned to manufacture door and window frames, scientific instruments and tools and implements on a large scale. The *Jaipur Glass and Potteries Works Ltd.*, manufacture glass jars, tumblers, bottles etc., worth Rs. 60,000 a month. A similar factory—*Hindustan Glass Works Ltd.*, is planning an early start at Sawai Madhopur with a capital of 12½ lakhs.

### Chemical and Allied Industries

(a) **HEAVY AND FINE CHEMICALS.**—The Kamani group of industries are considering a one-crore scheme for manufacturing alkali from the bitterns of Sambhar Lake.

(b) **PHARMACEUTICALS AND DRUGS.**—Ayurvedic medicines and drugs continue to be manufactured by the Dhanwantri Aushadhalaya and the Bharat Ayurvedic Pharmacy, Ltd. A new pharmaceutical concern has been started at Jhunjhunu.

(c) **STARCH MANUFACTURE.**—Since January 1945, The Jaipur Maize Products Co., with a capacity of 15 tons per day, has been manufacturing maize starch, glucose, dextrin, maize oil, bran and other products.

(d) **WATERPROOF PRODUCTS.**—A local firm has undertaken the manufacture of oil cloth, rain coats, tarpaulins, painted canvas, waxed paper, cardboard,

filter paper and writing and stamping inks. The market response has been so encouraging that the firm has opened new premises at Jhalana.

(e) **OIL AND SOAP.**—4 factories—*Navin Bharat Industries Ltd.*, Malpura Jaipur Oil Mills, *Mavandia Oil Mills*, Jaipur, and *Mabesh Ice and Oil Mills*, Phulera—have a yearly crushing capacity of 20,250 Mds. of seeds. The establishment of a dozen large crushing mills at Jaipur, Gangapur, Hindun, Sawai Madhopur and Phulera has received Governmental sanction.

### Textile Industries

(a) **COTTON MILLS AND HANDLOOM FACTORIES.** 69 handloom factories weave cotton fabrics from millspun yarn. A cotton spinning and weaving mill with 5,000 spindles has recently started functioning. Sufficient scope exists for small factories with power looms. The *Jaipur Textiles Ltd.*, has started working 7 power looms and three more are planned.

(b) **CARPET AND WOOLEN FACTORIES.**—Census figures for 1945 revealed that there were 34 lakhs of sheep in the State with an estimated annual output of 5 million lbs. of wool, the major portion of which is exported in its raw state. Within the last 2½ years the demand for army blankets and felts has been so great that 6 handloom factories have been established to supply the demand. 2 among them were engaged in executing war orders. The others are producing rugs, blankets, tweeds and felts for civilian use. Besides these, there are 50 carpet weaving factories in Jaipur city.

(c) **HOSIERY FACTORIES.**—8 hosiery factories have sprung up in Jaipur City during the last 2 years. Cotton vests, socks, stockings and woolen pullovers are some of the products produced.

(d) **DYING AND CALICO PRINTING.**—6 printing factories have recently come into existence to supplement the production from cottage industries.

### Miscellaneous Industries

(a) **LEATHER AND BONE BRUSHING INDUSTRIES.** In order to retain the large quantities of raw hides and skins now being exported to neighbouring Provinces for tanning into finished leather, the State has sanctioned the erection of a modern tannery within its own borders.

(b) **SAW MILLS AND BOBBIN FACTORIES.**—There are 6 saw mills operating at present.

(c) **LAPIDARY WORK.**—The cutting, faceting and polishing of precious, semi-precious and synthetic stones has long been one of the principal cottage industries of Jaipur City. Employing nearly 10,000 persons, 15 small factories using power have recently been established. Their number is likely to increase when more electricity becomes available.

(d) **ICE FACTORIES.**—Two ice factories, with a daily capacity of 18 tons, have started production and two are under construction.

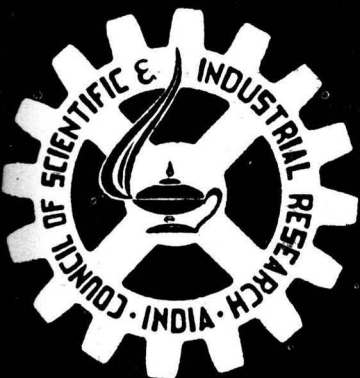


# INDIAN PATENTS

[The following is a List of Patent Applications notified as accepted in the Gazette of India, Part II, section I, for May-June, 1947.]

34609. ELECTRIC SWITCHING SELECTION CONTROL SYSTEMS: *After a selection has been made under control of marker circuit, a test is made to check the selection.*—Standard Telephones and Cables.
34632. ELECTRIC OSCILLATION GENERATORS: *Comprising thermionic valves and having positive and negative feedback paths, resistance capacity means included in the positive feedback path and directly heated thermistor connected in the negative feedback path.*—Standard Telephones and Cables.
34633. IMPROVEMENTS RELATING TO ELECTRICAL PULSE SIGNALLING SYSTEMS: *Includes a normally inoperative oscillation generator rendered operative by a repetitive wave of desired pulse frequency a characteristic of generated oscillations being varied by modulation signal.*—Standard Telephones and Cables.
34659. MICROWAVE ANTENNAS: *Comprising a pair of wave guides and a concave reflector facing the apertures of said guides.*—Western Electric Co. Incorporated.
34911. WELDING OF METAL STUDS AND THE LIKE TO METAL PLATES AND THE LIKE: *Chuck holding stud moved towards and away from plate by armature of an electromagnet; electrical means provided for controlling the magnet and welding circuit.*—Martin.
34917. DIRECTIONAL ANTENNAS: *Lobe sweeping action produced by varying the phase velocity of the waves delivered to or received from the antenna system.*—Western Electric Co. Incorporated.
34979. A COMBINED PORTABLE MIXER AND BARROW: *Barrel with one open end and inclined upwardly has a shaft projecting axially from closed end and mounted in bearing in a wheeled frame; means to rotate barrel provided.*—Lightburn.
35397. TELECOMMUNICATION SYSTEMS: *Comprises digit operable controller, locking and unlocking devices, digit responsive device and a source of current.*—Standard Telephones and Cables Ltd.
35399. BOTTLES AND PARTICULARLY HOT WATER BOTTLES: *Providing for the engagement of the stopper a re-entrant tubular element formed of an elastic material leaving about it a recess into which contents of the bottle may enter.*—Atkin.
35401. DIRECTIVE ANTENNA SYSTEMS: (16th August 1945): *Cylindrical parabolic reflectors with perpendicularly related focal lines.*—Western Electric Co. Incorporated.
35403. ELECTRON DISCHARGE DEVICES: *Cathode and anode mounted on respective support members with plane seating surfaces, and plane grid is aligned parallel by spacer means.*—Western Electric Co. Incorporated.
35405. WATER-ELEVATORS OF THE WHEEL AND BUCKET TYPE: *An open-topped tank is arranged close under the wheel over which is trained an endless conveyor with spaced buckets and the lower part of the wheel dips into the water in the said tank.*—Remfry.
35553. POWER UNITS FOR LOCOMOTIVES: *Internal combustion engine for driving supplied by air from a blower driven by auxiliary engine whose output is controlled to maintain power of main engine constant and its torque diminished with increased speed.*—Fell.
35561. TOTALIZER OR RECORDING AND COMPUTING MACHINES: *Totalizer having plurality of numeral wheels characterized by plurality of projecting fingers to control electrical circuits and mechanical movements.*—Ramington Rand Inc.
28148. RECOVERY OF CRUDE BENZOLE FROM BENZOLISED WASH-OIL: *Subjecting the benzole vapours produced in wash-oil distilling column to a rectification in an extension of the said distilling column.*—Woodall-Duckham (1920) Ltd.
28319. SEPARATION OF LIQUIDS OF DIFFERENT BOILING POINTS FROM MIXTURES THEREOF BY FRACTIONAL DISTILLATION: *Intermediate fractions are removed at the full optimum distillation rate of the still at certain period of distillation.*—Woodall-Duckham (1920) Ltd.
31438. HIGH EXPLOSIVE COMPOSITIONS OR CHARGES: *Mixing into a slurry of sensitive explosive compound and nitrocellulose, and insensitive to shock, liquid solvent for nitrocellulose.*—I.C.I. Ltd.
31672. ELECTRICAL CONTRACT BRUSHES: *Brush comprises predominantly carbon or mixtures of carbon and minor proportions of non-deliquescent salts or oxides of alkaline earth metals.*—National Carbon Co. Inc.
32550. CIGARETTES: *Combustible tip mounted on combustible support.*—Brown.
32656. MULTI-CHANNEL ELECTRICAL PULSE COMMUNICATION SYSTEMS: *Utilises a distributor with delay net work to which regularly repeated waveform is applied. There are means for compensating or eliminating effects due to distortion.*—Standard Telephones and Cables Ltd.
32868. PRODUCTION OF PLASTIC COMPOSITIONS: *Incorporating hydro-carbons extracted from mineral oil fraction and air blown with polycynylhalide.*—Anglo-Iranian Oil Co. Ltd.
33067. PENS AND LIKE IMPLEMENTS FOR APPLYING INK AND OTHERS FLUIDS: *Ball applicator in the tip having swage fit, and a flexible sac in a barrel.*—Fagle Pencil Co.
33219. HEAT EXCHANGE DEVICES: *Part of elements composed of copper plated with nickel and brazed together with high temperature solder*

- metal or alloy.—I.C.I. Ltd.
33884. REPAIRING WATER PIPES OR PERFORMING OTHER OPERATIONS ON LIQUID-CONTAINING PIPES: *The device comprises a two-part clip having a groove with rubber lining, a hollow piercer and a drip cock.*—Freez-Seal Equipment Co. Ltd.
33914. THRUST PRODUCING ROTOR DEVICE FOR AIRCRAFT: *Blades inclinable about longitudinal axes, control for changing inclination of blades, inclinable rotating inertia means (e.g. a fly wheel) and linkage inter-relating inclination of inertia means, blades and control.*—Bell Aircraft Corp.
34128. PURE HYDROCARBONS FROM PETROLEUM NAPHTHA AND OTHER HYDROCARBON FEEDSTOCKS: *Fraction distilling the range of 66-69% C to 72° C is passed to a zone of catalytic isomerisation.*—Anglo-Iranian Oil Co. Ltd.
34259. SHOCK-PROOF ELECTRIC WATER HEATER: *Perforated metallic grid spaced from and enclosing central line electrode.*—Acharya.
34352. STABLE AQUEOUS DISPERSIONS OF SECONDARY AROMATIC AMINES: *Incorporating lecithin in the aqueous dispersion of secondary aromatic amine.* Du pont de Nemours and Co.
34901. X-RAY DIFFRACTION APPARATUS: *Comprises X-ray tube housing, are shaped track for rotary displacement and a Geiger-Mueller tube.*—Philips Lamps Ltd.
34902. ELECTRIC DISCHARGE DEVICES: *A metallic electrostatic shielding embedded in the glass envelope.*—Philips Lamps Ltd.
35028. AUTOMATIC TELECOMMUNICATION EXCHANGE SYSTEMS: *Means for connecting one electrode of a cold cathode discharge tube to a test terminal for testing busy of idle conditions of the line.*—Telephones and Cables Ltd.
35063. SPINNING OF YARNS FROM SLIVERS OF JUTE, FLAX, HEMP, CUT ARTIFICIAL SILK AND OTHER FIBRES: *Sliver being drawn from rolls mounted for rotation in a creel wherein rotation of the rolls is stopped on breakage of the yarn.*—James Mackie and Sons Ltd.
35372. PHOTO-ELECTRIC CELLS: *Fluorescent screen applied on outer surface, screen facing the cathode.*—A. Graves and Alltools Ltd.
35582. FACILITATING THE TOPPING-UP OF ELECTRIC ACCUMULATORS: *Topping-up or watering orifice inside wall below level of electrolyte.*—The Chloride Electrical Storage Co. Ltd.
35626. TYRE-SPREADING DEVICE: *The shorter arm of one of two levers pivoted together is fashioned to engage the inner circumferential edge of a tyre and means are provided whereby the shorter arm of the lever can be held at any desired distance apart.*—Woodhouse & Mitchell Ltd.
35708. REPEATEDLY IGNITABLE COMPOSITION ROD: *A core containing an igniting substance and a hydrated inorganic salt, having an outer layer of combustible substance such as metaldehyde and or nitrocellulose of pyroxyline type.*—Ellenbogen.
35758. A LOCK FOR A CYCLE OR THE LIKE: *Locking mechanism comprises a casing with parts clamped together to embrace the steering head and a key-actuated lock secured therein.*—Milwain, Hitchcox and Hitchcox.
35765. LIQUID-FUEL COMBUSTION CHAMBER: *The burning mixture emerges through a sharp-edged of the burner chamber, in which injected fuel and primary air are mixed, into a space. Whence it is returned externally to preheat the burner chamber.*—Armstrong Siddeley Motors Ltd.
35766. INTERNAL-COMBUSTION TURBINE PLANT: *Controllable valve means allow about 2 per cent. of the burning mixture being abstracted from the combustion chamber and injected into the air at the compressor inlet.*—Armstrong Siddeley Motors Ltd.
35845. ELECTRIC FANS: *A reduction gear of toothed spurwheel type includes a reaction gear wheel, an output gear wheel, an input members and a gyratory gear cluster.*—Spencer and The Patent Lighting Co. Ltd.
35854. FIRE EXTINGUISHING INSTALLATIONS: *Comprises a delivery device and a supply pipe for the foam, between which is a moveable section which completes the foam passage.*—The Pyrene Co. Ltd.
35861. ELECTRIC DEVICE TO PREVENT THE FORMATION OF CALCAREOUS INCRUSTATIONS AND DISLodge THOSE ALREADY EXISTING: *Liquids passed into a magnetic field created by an electrically connected solenoid.*—Vermeiren.
35874. PLASTIC COMPOSITIONS: *Containing polyvinyl chloride or polyvinyl chloride co-polymers and hydrocarbon materials.*—Anglo-Iranian Oil Co. Ltd.
35970. COCKS: *An internally screw threaded nozzle unit engaging an external thread on a tubular member with an annular seat on the end of the member.*—Brock.
35983. A LIQUID ATOMISER: *Piston rod having a non-return valve at one end incorporates as a unitary structure therewith an atomizer nozzle.*—Drugs. Ltd.
35984. PUMP FITTINGS FOR LIQUID ATOMISERS: *A pump fitting having a piston rod, piston, a piston sleeve, a guide sleeve and a return spring capable of being screwed to a pressure conduit.*—Drugs Ltd.
36102. REFRIGERATING ENGINES: *Refrigerating engine is coupled with a hot-gas motor.*—N. V. Philips' Gloeilampenfabrieken.
36107. IMPROVEMENTS IN OR RELATING TO AIR CONDITIONING OR CIRCULATING APPARATUS: *Consisting of a trough, a fan, electric heating elements and an endless belt depending from a pulley.*—Lipton.
36189. A POLYPHASE DOUBLE SYNCHRONOUS SPEED SYNCHRONOUS MOTOR: *Phase sequences of alternating currents in stator and rotor are provided in opposite directions.*—Dr. Sukumaran.
36226. ABSORPTION TYPE REFRIGERATORS: *Comprises a U-shaped pre-cooling chamber.*—Charles Industries Pty, Ltd.
36283. TUBULAR HEAT EXCHANGERS: *Imparting to the tubular elements an aerofoil or substantially aerofoil section.*—Thermalair Ltd.
36316. PURIFICATION OF SYNTHETIC LOWER ALIPHATIC ALCOHOLS: *Treating aliphatic alcohol with hydrogen in presence of a nickel catalyst.*—Les Usines De Melle.



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[No. 8

## Electrolytic Reduction of Nitro-compounds

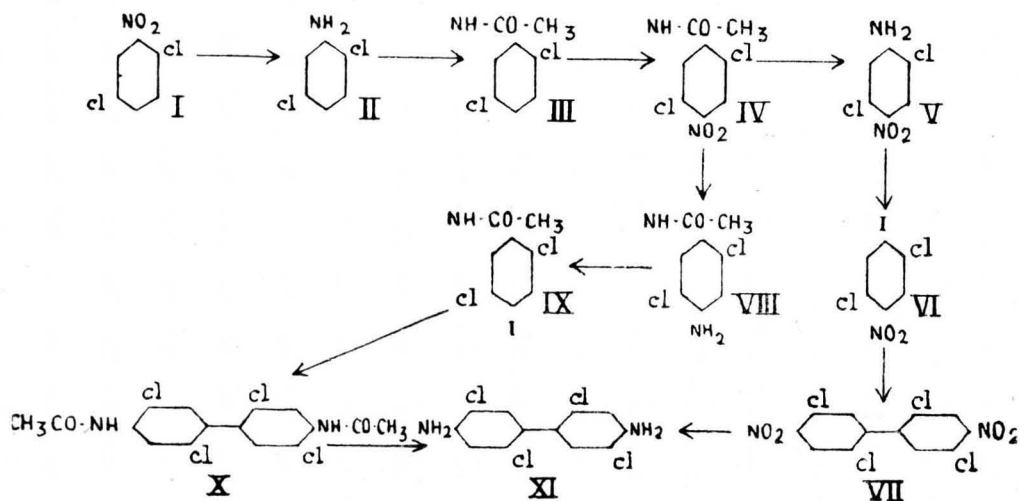
PART IX—SYNTHESIS OF 2, 5, 2', 5'—TETRACHLORO-BENZIDINE

By B. B. DEY, T. R. GOVINDACHARI, S. C. RAJAGOPALAN and  
H. VENKATAKRISHNA UDUPA

(Presidency College, Madras)

THE electrolytic reduction of 1, nitro-2,5, dichloro-benzene in alkaline emulsion to 2,5,2',5'-tetrachloro-hydrazobenzene, and the conversion of the latter into 2,5,2',5'-tetrachloro-benzidine by prolonged boiling with concentrated hydrochloric acid, has been previously described in a communication from this laboratory.<sup>1</sup> No reference is found in literature to the physical constants and other details regarding this compound, and

even such simple derivatives as the acetyl and benzoyl compounds are not mentioned. A synthesis of the product of electrosynthesis by a simple and unambiguous method appeared therefore to be necessary in order to place its constitution beyond doubt. Attempts were first made to obtain the compound by the application of the usual Ullmann reaction to 2,5-dichloro-4-iodo-nitrobenzene (VI), and to 2,5-dichloro-4-iodo-acetanilide (IX), on the following lines:—





Both 2,5-dichloro-4-iodo-nitrobenzene and 2,5-dichloro-4-iodo-acetanilide were obtained in satisfactory yields by the diazo-replacement of the corresponding amines by iodine provided certain precautions were adopted, the nature of which forms the subject of a separate communication. For some unexplained reasons, however, these iodo-compounds could not be converted by Ullmann's method into the desired diphenyl derivatives.

On heating either of the iodo-compounds with activated copper bronze in dry nitrobenzene, the main product of the reaction was an unpurifiable resinous tar and attempts to carry out the reaction in the absence of a solvent were equally unsuccessful. In the case of the iodo-acetanilide, a small amount of a crystalline substance (m.p. 127°C.; anhydrous, m.p. 180°C.) volatile in steam and containing nitrogen and chlorine but free from iodine, was obtained as a by-product. It was found to contain the acetamino-group and was hydrolysed by dilute alkali to a base, m.p. 48°C. It has not been possible to investigate the compound further on account of the very small quantity available, but it is hoped to take up this work shortly.

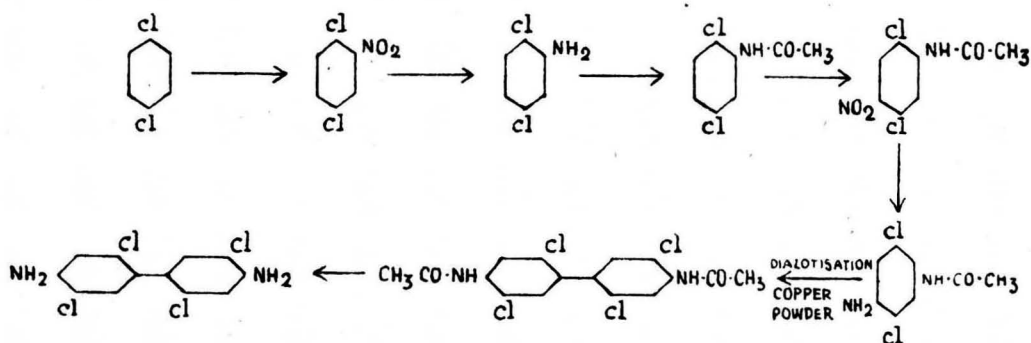
Having failed to obtain the desired compound by the method described above, the synthesis was next attempted on the following lines according to Gattermann's procedure:

admixture with the tetrachloro-benzidine prepared by the electrolytic method. The diacetyl (m.p. 297°C.) and the dibenzoyl (m.p. 252°C.) derivatives prepared from both the samples were also found to be identical. These experiments, therefore, furnish definite proofs of the constitution assigned to the tetrachloro-benzidine prepared by electrolytic reduction.

### Experimental

**2,5-dichloro-1-nitrobenzene:** This was prepared by the nitration of *p*-dichlorobenzene according to the procedure recommended by Ganopolski<sup>2</sup> in a yield of 90 per cent.

**2,5-dichloro-aniline:** No satisfactory method for the preparation of this compound has been described in literature. The procedure described here is based on that adopted by Adams<sup>3</sup> and has been found to give good results. In a 1-litre, bolt-head flask fitted with a mercury seal stirrer reaching to the bottom, dropping funnel and condenser, 100 gms. of 2,5-dichloro-nitrobenzene dissolved in 500 cc. of 50 per cent. alcohol and 80 gms. of specially pre-treated iron powder (see note 1) are stirred together and heated to boiling under gentle reflux. 5 cc. of conc. hydrochloric acid in 50 cc. of 50 per cent. alcohol are added very slowly in the course of about half an hour when there is a vigorous reaction at the beginning. The stirring is continued under gentle reflux for another 5



A pure tetrachloro-benzidine has been obtained by this method in a yield of nearly 50 per cent. It melted sharply at 135°C., and this melting point was not lowered by

hours, and a solution of 3 gms. of caustic soda in 10 cc. of water is added. The solution is filtered hot and the residue worked with three 50 cc. portions of boiling 95 per cent. alcohol.

The filtrate is shaken with 50 cc. of conc. hydrochloric acid and left in a flask in the ice chest overnight. The crystals of 2,5, dichloroaniline hydrochloride are filtered and a second crop of crystals is obtained by concentrating the filtrate. The combined hydrochloride crystals are basified and steam distilled when 74 gms. of the pure base, m.p. 50°C., were obtained (88 per cent. of the theory). On repeating the experiment on a slightly larger scale using 200 gms. of the dichloro-nitrobenzene in a 2-litre, bolt-head flask, the same yield was reproduced (150 gms. of the base, m.p. 50°C.).

*2,5, dichloroacetanilide:* 150 gms. of 2,5, dichloroaniline, acetylated in the usual manner, gave 181 gms. of the pure acetanilide crystallised from acetic acid, m. p. 132°C. (96 per cent. of theory).

*2,5, dichloro-4, nitro-acetanilide:* The nitration of the acetanilide was carried out according to the directions given by Whiston<sup>4</sup> 150 gms. of 2,5, dichloroacetanilide are dissolved in 580 cc. of 96 per cent. sulphuric acid at 40°C., and then cooled to below 5°C. Well cooled mixed acid consisting of 450 gms. of concentrated sulphuric acid and 90 gms. of 66 per cent. nitric acid (sp. gr. 1.405) is gradually added, the temperature not being allowed to rise above 10°C. When the addition is over, a period of about an hour and a quarter, the contents are allowed to attain room temperature during the course of about an hour and a half, and then stirred mechanically for 15 minutes and poured into ice with stirring. The pale yellow nitro-compound is filtered, washed first with water, then with sodium carbonate and finally with water till free from alkali, and then dried in a steam oven. The crude product weighed 179 gms. and melted at 141°-142°C. A small quantity was crystallised from alcohol (m.p. 148°C.), and the rest was hydrolysed to 2,5, dichloro-4, nitro-aniline (m.p. 152°-153°C.) according to the method of Whiston.

*2,5, dichloro-4, amino-acetanilide:* 28 gms. of 2,5, dichloro-4, nitro-acetanilide in 150 cc. of 50 per cent. alcohol and 20 gms. of pre-treated iron powder (90 mesh) were taken in a 1-litre, bolt-head flask provided with mercury seal stirrer, dropping funnel and reflux condenser. 5 cc. of conc. hydrochloric acid in 30 cc. of 50 per cent. alcohol were added slowly to the gently refluxing contents during the course of an hour. The stirring was con-

tinued for another 3 hours at the end of which 3 gms. of sodium hydroxide in 20 c.c. of 50 per cent. alcohol was added. The reaction mass was filtered hot and the residue washed with two 50 cc. portions of boiling 95 per cent. alcohol. The alcohol was finally distilled off and the residue treated with water and filtered. The crude material, dried at 100°C., weighed 22 gms., m. p. 127°-128°C. (90 per cent. of theory). A portion crystallised from alcohol melted at 131°-132°C. In an experiment on a slightly larger scale, 76 gms. of 2,5, dichloro-4, amino-acetanilide were obtained from 89 gms. of the nitro-acetanilide. Found: Nitrogen, 12.87 per cent;  $C_8H_8N_2Cl$  requires, nitrogen, 12.78 per cent.

*2,5, dichloro-4, iodo-acetanilide:* The amino-acetanilide (5 gms.) in 25 gms. crushed ice and 10 cc. conc. hydrochloric acid was treated drop by drop with a solution of 1.6 gms. of sodium nitrite in 3 cc. of water and allowed to stand at 0°C. for an hour with occasional stirring. The clear solution was then treated with 3.75 gms. of potassium iodide dissolved in 10 cc. of water with vigorous stirring. The mixture was heated on the water bath 70°C., filtered and the residue ground in a mortar with 1 per cent. alkali and again filtered and washed with water. Crystallisation from alcohol gave thick prisms, m. p. 180°C. (Yield, 4.8, gms) Found : Nitrogen, 4.51 per cent;  $C_8H_6NCl_2I$  requires, nitrogen, 4.23 per cent.

*2,5, dichloro-4, iodo-nitrobenzene:* When 2,5, dichloro-4, nitroaniline was diazotised in the presence of hydrochloric acid in the usual manner, the nitro-group was found to be eliminated and replaced by chlorine. This interesting observation forms the subject of a separate communication. In the absence of hydrochloric acid, however, the process of diazotisation was normal. The following procedure was adopted: 5 gms. of 2,5, dichloro-4, nitro-aniline suspended in a mixture of glacial acetic acid (75 cc.), concentrated sulphuric acid (7.5 cc.) and water (10 cc.) were treated slowly at 0°C., with a solution of 1.7 gms. of sodium nitrite in 5 cc. of water. The diazonium solution which soon became clear was treated in the cold with stirring with a solution of 4 gms. of potassium iodide in 10 cc. of water, and the mixture warmed on the water bath to 70°C., for half an hour. After dilution with water (100 cc.), the solid was filtered, ground with dilute alkali, washed, filtered and crystallised

from alcohol. Thick rods, m.p. 84°C., weighing over 6 gms. Found: Nitrogen, 4.17 per cent;  $C_6H_2O_2NCl_2I$  requires, nitrogen, 4.4 per cent.

*Synthesis of 2,5,2',5'-tetrachloro-benzidine:* 10 gms. of 2,5, dichloro-4-amino-acetanilide were dissolved in a mixture of 20 cc. of conc. hydrochloric acid and 50 cc. of water by warming to 70°C., and rapidly cooled with vigorous stirring. Fine crystals of the hydrochloride were immediately thrown down. The mixture was cooled to below 5°C., a solution of 3.5 gms. of sodium nitrite in 15 cc. of water added drop by drop with vigorous stirring and the completion of the process tested with starchiodide paper. 6 gms. of freshly prepared dry copper powder (see, note 2) were gradually added to the diazotised solution, and the mixture stirred mechanically for 3 hours in the cold, two hours at room temperature and finally for an hour in a boiling water bath. The dark coppery solid was filtered on cooling and dried in a desiccator under reduced pressure. It weighed 15.2 gms. It was placed in a Soxhlet thimble and extracted with boiling dry benzene (200 cc.) until the extract was colourless (nearly 5 hours). The residue left on distilling off the benzene weighed 6.5 gms. This residue was taken in a flask, 50 cc. of 40 per cent. sulphuric acid was added and the whole boiled gently under reflux for 3 hours. A colourless solid separated which increased in quantity as the reaction (hydrolysis) progressed. This was filtered, dried and extracted again with benzene to remove a portion soluble in benzene to which it imparted a deep red colour. The colourless solid insoluble in benzene weighed 4 gms. while the benzene solution, on distillation, left a red solid, m.p. 186°C., weighing 2.2 gms. The colourless solid was suspended in water and treated with sodium carbonate (5 gms) and boiled for half an hour. On cooling, the residue was filtered, ground with dilute caustic soda, filtered, washed and dried in a vacuum desiccator. It weighed 3 gms. and melted at 125-126°C. It was boiled with 50 cc. of methanol with the addition of norit and filtered. On cooling, light orange red crystals separated out (1.5 gms). The crystals melted sharply at 135°C. and the m.p. was not lowered by admixture with 2, 5, 2', 5'-tetrachloro-benzidine prepared before by electrolytic reduction of 2, 5, dichloro-nitro-

-benzene. The methanol mother liquor, on standing, deposited another crop of crystals melting at 133°C. (1.1 gms).

The diacetyl derivative prepared readily in theoretical yield by rubbing with acetic anhydride, melted at 297°C., not depressed by admixture with 2,5,2',5'-tetrachloro-1,1'-diacetamino-diphenyl obtained previously from the electro-reduction product.

The dibenzoyl derivative melted at 251°-252°C., and its mixture with the dibenzoyl derivative of the electrolytic product had the same melting point.

Our thanks are due to the *Council of Scientific & Industrial Research* under whose auspices this work was carried out.

*Note 1.* The iron powder (20 gms.) is kept stirred in 200 cc. of water and 3 cc. conc. hydrochloric acid added. The iron is allowed to settle and quickly filtered off and a current of air is drawn through the mass for a short time. The iron powder becomes quite warm when it is removed, ground in a mortar and added to the contents of the reaction vessel. Such activated iron functions very efficiently and gives excellent yields of the reduction products.

*Note 2.* 34 gms. of copper sulphate are dissolved in 200 cc. of water, cooled to 0°C., and 9 gms. of fine zinc powder are added. The mass is stirred mechanically for half an hour, filtered, washed with 50 cc. of water containing 2 cc. of conc. hydrochloric acid and then again with water and finally with acetone and dried in vacuum. This dry copper powder was used in the Gattermann reaction.

#### References.

1. Dey, Govindachari and Rajagopalan, *J. Sci. & Ind. Res.*, 1946, 6 B., 75.
2. Ganopolski, *Org. Chem. Ind.*, 1936, 1, 342.
3. Adams, *Org. Synthesis*, Vol. 11, p. 32.
4. Whiston, *J. Soc. Chem. Ind.*, 1924, 43, 370 T.

# Electrolytic Reduction of Nitro-compounds

PART X—ELIMINATION OF THE NITRO-GROUP IN THE PROCESS OF DIAZOTISATION OF 2, 5, DICHLORO-4, NITRO-ANILINE

By B. B. DEY, T. R. GOVINDACHARI AND S. C. RAJAGOPALAN  
( Presidency College, Madras )

THE formation of a tetrachloro-benzidine by the electrolytic reduction of 2,5-dichloro-nitrobenzene in alkaline emulsion has been described in part VII of this series.<sup>1</sup> A synthesis of the benzidine so as to confirm its constitution has also been completed and the results communicated in part IX of the series. In the course of the latter investigation, certain interesting observations were made which form the subject of the present communication.

The synthesis of 2, 5, 2', 5', tetrachloro-benzidine was first attempted by the application of Ullmann's reaction, using 2, 5, dichloro-4, nitro-1, iodo-benzene as the starting material. The simplest and the most obvious method of preparing the latter was considered to be the diazo-replacement by iodine of the amino-group in 2, 5, dichloro-4, nitro-aniline. The preparation of the latter starting from *p*-dichloro-benzene has been described in detail in (part IX). When a cold solution of the nitraniline in a mixture of acetic and hydrochloric acids was diazotised in the usual manner and treated with potassium iodide, a product was obtained which was free from nitrogen and which, on closer examination, proved to be identical with 2, 4, 5,-trichloro-1, iodo-benzene,<sup>2</sup> m. p. 106°C. In other words, the nitro-group had been eliminated during the process of diazotisation and its place taken by chlorine. The correctness of this view, and incidently of the structure of the compound, m. p. 106°C., mentioned above, was further proved in the following way: The clear diazonium solution prepared from the nitraniline was treated not with potassium iodide but with an alkaline solution of  $\beta$ -Naphthol, and the deep red azo-dye (m.p. 197°C.) was reduced with stannous chloride and hydrochloric acid. The mixed bases (1, amino-2, naphthol and the new chloraniline) were separated by extraction with benzene in strong alkali, and the iden-

tity of the chloraniline as 2, 4, 5, trichloraniline 3, 4, 5 was established in the usual manner (m.p. and mixed m.p., 96°C.; acetyl derivative, m.p. and mixed m.p., 190°C.).

The elimination of a nitro-group and its replacement by chlorine in the course of diazotisation of a nitro-amine in hydrochloric acid was first observed by Meldola and Eyre<sup>6</sup> in the case of 2, 3, dinitro-4, methoxy-aniline and an extensive study of such cases has been made by Meldola and his pupils<sup>7, 8</sup>. On the basis of the results of these investigations they have laid down certain general conditions for such elimination to occur, *viz.*, that (a) the nitrogroup should be either in the *ortho*- or in the *para*- position to the diazo-group, and (b) there should be a second nitro-group adjacent to the one which is knocked off. Meldola and his co-workers confined their researches mostly to the polynitro-anilines and the fact that such displacements of nitro-groups can occur even in the absence of a second nitro-group but in the presence of only a chlorine atom adjacent to the nitro-group appears to have escaped their observation. In the present instance the problem is complicated to some extent by the occurrence of a second chlorine atom in the *meta*-position to the nitro-group which is eliminated. That an *ortho-para*-orienting group like chlorine has the power to labilise groups in the *meta*-position with respect to itself has followed from the observations made by Kenner,<sup>9</sup> and the probability, therefore, that this second chlorine atom, by labilising then nitro-group, may have also had some influence in bringing about its displacement, is not entirely ruled out. While the relative shares of influence exerted by the two chlorine atoms and also perhaps by other groups on such elimination can be determined only by further investigations in this field, the observations made so far justify an extension of Meldola's

postulate by stating at this stage that the group adjacent to the nitrogroup displaced need not necessarily be a second nitro-group but may also be a chlorine atom.

The presence of methoxy- and ethoxy-groups may also produce a similar effect and it is proposed therefore to study the behaviours of several nitro-anisidine and nitro-phenetidine derivatives on diazotisation so as to arrive at more general conclusions regarding the influence of different groups on this peculiar effect.

It has been found that there is no disturbance of the nitro-group if the diazotisation of 2, 5, dichloro-4, nitro-aniline is carried out in the absence of hydrochloric acid, by dissolving the amine in a mixture of acetic and sulphuric acids. Under these circumstances the desired compound-2,5, dichloro-4, nitro-1, iodo-benzene<sup>10</sup>, m.p. 84°C. - could be prepared in a yield of nearly 80 per cent. That the reaction proceeded in these conditions in the normal manner without the nitro-group being eliminated, has been further confirmed by coupling the diazo-compound with  $\beta$ -naphthol in alkaline medium and reducing the resulting azo-dye (m.p. 236°C.) with stannous chloride and hydrochloric acid when 2, 5, dichloro-1, 4, diamino-benzene<sup>11</sup> (m.p. 168°C.; acetyl derivative, m.p. 296°C.) was obtained, besides 1, amino-2, naphthol.

It is worthy of note that a similar observation has been made<sup>12</sup> in the case of 1, nitro-2, amino-naphthalene. The nitro-group is removed and its place occupied by chlorine if diazotisation is carried out in hydrochloric acid, whereas if sulphuric acid is used no such replacement occurs.

### Experimental.

**2, 4, 5, trichloro-1, iodo-benzene.** The preparation of 2, 5, dichloro-4, nitro-aniline has been described in detail elsewhere. 15 gms. of the nitraniline were dissolved in a mixture of 15 c.c. of hydrochloric acid and 50 cc. of glacial acetic acids and treated at 10°C. with a solution of sodium nitrite (5 gms.) in 10 c.c. of water added drop by drop with vigorous stirring. The clear diazonium salt solution was treated with stirring with 12 gms. of potassium iodide dissolved in 15 c.c. of water and the mixture slowly heated upto 80°C. After cooling, the solid was filtered, ground with dilute alkali, filtered again, washed and

crystallised from alcohol. 12.5 gms. of the pure compound melting at 106°C. were obtained. Istrati<sup>2</sup> who prepared the same compound by heating 1, 2, 4, trichlorobenzene with iodine and sulphuric acid in a closed vessel records 107°C. as the melting point. Found: Iodine, 40.6 per cent.;  $C_6H_2Cl_3I$  requires Iodine, 41.3 per cent.

**2, 4, 5, trichloro-benzene-azo- $\beta$ -naphthol.** 1 gm. of 2,5, dichloro-4, nitro-aniline in 2 c.c. of conc. hydrochloric acid and 12 c.c. of glacial acetic acid was treated at 10°-20°C., with a solution of 0.35 gm. of sodium nitrite in 10 c.c. of water and shaken till a clear solution was obtained. It was then poured into a cold solution prepared by dissolving 0.65 gm. of  $\beta$ -naphthol in 50 c.c. of water containing 15 gms. of sodium hydroxide. The deep red dye was filtered, ground with water containing hydrochloric acid, washed and dried. Yield, 1.5 gms. Crystallisation from ethyl acetate gave dark red sheaves of needles, m.p. 197°C. Found: Nitrogen, 7.6 per cent.;  $C_{16}H_9ON_2Cl_3$  requires Nitrogen, 7.98 per cent.

**Reduction of the azo-dye to 2, 4, 5, trichloro-aniline.** The crude dye prepared from 1 gm. of the dichloro-nitraniline was taken in a small flask fitted with an air condenser and boiled under gentle reflux with 1 gm. of tin, 6 gms. of stannous chloride and 20 cc. of conc. hydrochloric acid. When reduction was complete as shown by the liquid becoming colourless, it was basified with excess of 25 per cent. sodium hydroxide solution and extracted with three 25 cc. portions of benzene and the benzene extract dried over anhydrous potassium carbonate. The benzene was distilled off and the crude base (0.6 gm.) was crystallised from ligroin. It melted at 95°C., and formed an acetyl derivative, m.p. 190°C. These are identical with the meeting points recorded in literature 3, 4, 5 for 2, 4, 5, trichloroaniline and 2, 4, 5, trichloroacetanilide.

**2, 5, dichloro-4, nitrobenzene azo- $\beta$ -naphthol.** 1 gm. of 2, 5, dichloro-4, nitro-aniline was taken in 1.5 c.c. of conc. sulphuric acid and 4 cc. of water, 15 cc. of glacial acetic acid added and the mixture treated at 10°C., with a solution of 0.35 gm. of sodium nitrite in 2 c.c. of water with stirring. The clear solution was added to 0.65 gm. of  $\beta$ -naphthol dissolved in a solution of 15 gms. of sodium hydrochloride in 50 cc. of water. The crude



azo-dye (1.6 gms.), purified in the usual manner, crystallised from ethyl acetate in purple needles, m.p. 238°C. Found: Nitrogen, 11.92 per cent.;  $C_{10}H_9O_3N_3Cl_2$  requires Nitrogen, 11.6 per cent.

*Reduction of the azo-dye to 2, 5, dichloro-1, 4, diamino-benzene.* The uncrystallised azo-dye prepared from 1 gm. of dichloro-nitraniline was boiled under reflux with 6 gms. of stannous chloride containing a little metallic tin and 20 cc. of conc. hydrochloric acid until it became colourless. The solution was basified on cooling with excess of conc. alkali and extracted thrice with benzene. The dried benzene extract, on evaporation, left 0.7 gm. of a crude base, m.p. 164°C. Crystallisation from 50 per cent. alcohol gave slightly coloured glistening prisms, m.p. 169°C. The diacetyl derivative, prepared readily by rubbing with a little acetic anhydride, crystallised from acetic acid in colourless thin plates melting at 296°-97°C. The melting points of 2, 5, dichloro-*para*-phenylene diamine and of its diacetyl derivative recorded in literature, are 170° (164°) C., and 301° (294-96°) C. respectively.

Our thanks are due to the Council of Scientific & Industrial Research under whose auspices this work has been carried out, for

permission to publish the results.

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## Some preliminary experiments on the use of colloidal fuels for Diesel Engines

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**I**N a previous communication<sup>1</sup>, it has been shown that most of the Indian vegetable oils could be successfully employed as Diesel fuels. In comparison with mineral oils, vegetable oils, in general, gave approximately the same power output, a slightly high fuel consumption, better brake thermal efficiency and slightly more but not excessive carbon deposits in the combustion chamber. Cotton seed oil, in particular, gave exceptional performance in that its consumption was definitely less than that of the minerals.

Despite good performance, vegetable oils

have not been able to compete with mineral fuels, because of their higher price. In case the vegetable oil could be used as a vehicle for powdered solid fuel, the resulting fuel will be cheaper than vegetable oil itself and may be able to compete with mineral fuel. Powdered fuels as such are being tried out in other countries as Diesel fuels<sup>2</sup>, but they require engines of altogether different design. Colloidal fuels made from mineral oil and powdered coal for boilers, furnaces, etc. are well known<sup>3</sup> but their use as Diesel fuels is little known. No work appears to have been

carried out so far with vegetable oil colloidal fuels. A few experiments were carried out to study the performance of a conventional Diesel engine on such fuels. The results obtained cannot be considered as being positively successful, but do indicate the possibilities and the lines along which future work may be carried out.

The equipment consisted of a Lister Diesel engine of  $4\frac{1}{2}$ " bore,  $4\frac{3}{8}$ " stroke and rated to give 8 H.P. at 1200 r.p.m.<sup>1</sup> The compression ratio was 15:1 for normal operation and 19:1 for starting and at light loads.

Charcoal of particularly low ash content (3.49 per cent.) chosen for these experiments was finely powdered (particle size, about 20  $\mu$ ) in a ball mill and dispersed in groundnut oil by grinding for 12 hours in an edge runner mill. Of the various stabilizers investigated in 25 different experiments, calcium soap of groundnut oil fatty acids used in the proportion of 3 per cent. by weight on oil was found to give the best stability to these dispersions over prolonged periods of storage, (a few months). The characteristics of some of the fuels used in the tests and those of the charcoal are given in Table I.

satisfactory fuel flow upto 45 per cent concentration could be maintained by impressing a steady air pressure of about 12 of water over the surface of the fuel in the tank, but engine behaviour became erratic indicating defective injection. A similar observation was made by Kustov and Khotuntsev<sup>4</sup> while studying the viscosities of mazut coal suspensions. Bulk quantities of 35 per cent. charcoal fuel were accordingly prepared for longer engine trials.

Before proceeding further, a study of the injection characteristics of the 35 per cent. charcoal colloidal fuel as prepared and after de-aeration in vacuum, was made and compared with similar studies on groundnut oil and mineral oil. The technique involved the collection of injector spray on a blotting paper sheet placed at a definite distance from the injector and measuring the area of the impression caused by the spray with a planimeter. The results are given in Table I. These studies at different injection pressures could not be completed because at one stage the injection system failed..

Closer examination revealed that the fuel pump plunger was worn out and that the

TABLE I. — Properties of Fuels

	Specific Gravity at 30°C.	Moisture content, %	Ash, % of dry sample.	Volatile matter, %	Fixed carbon, %	Calorific Value on moisture and ash free basis, B. Th. U. lb.	Calorific value as used, B. Th. U. lb.	Calorific value as used, B. Th. U. gallon.	Area of injection impression at 18" from injector at 2,100 lbs. per injection pressure, Sq. in.	Angle of injected fuel, Deg. Min.
Charcoal powder.	1.292*	4.67	3.49	22.91	73.60	13,800	17,500	159,100	25.80	18 — 6
Groundnut oil.	0.908	0.38	0.03	—	—	17,570	12,700	—	—	—
35% charcoal colloidal fuel as prepared.	1.054	3.07	2.42	—	—	—	15,810†	166,700	1.37	4 — 12
35% charcoal colloidal fuel after deaeration.	1.055	—	2.42	—	—	—	15,810†	166,800	1.04	3 — 40
Mineral oil.	0.840	—	—	—	—	—	19,400	163,000	43.84	23 — 24

A 12½ per cent. (by weight) charcoal colloidal fuel was tried in the engine for half an hour. The only noticeable difference observed was a slight reduction in maximum load. Fuels, containing increasing concentrations of charcoal were then tried out in order to find the maximum concentration of charcoal, which the pumping, piping and injection system could handle. Fuels containing upto 35 per cent. charcoal were found to flow freely through the fuel system. However,

injection hole was slightly enlarged. The wearing out of the plunger could not be definitely ascribed to the use of colloidal fuel, since the detailed earlier history of the engine and the injector was unknown. The immediate programme was directed to find out the cause of wear in the injection system.

The engine at this stage was decarbonized, valves ground, all wearing parts accurately measured, weighed and the engine re-assembled with a new injector and fuel pump. A

Series of runs were carried out in the following order:-

- (1). *Mineral high speed Diesel oil:* Injection pressure versus maximum load at 1,200 r. p. m. for choosing best injection pressure.
- (2). *Mineral high speed Diesel oil:* B. H. P. versus fuel consumption at rated speed and best injection pressure.
- (3). *35 per cent charcoal colloidal fuel:* Same as (1) above for choosing best injection pressure with colloidal fuel.
- (4). *35 per cent charcoal colloidal fuel:* Same as (2) above.
- (5) *Mineral high speed Diesel oil:* Same as (1) above to determine best injection pressure with this fuel after colloidal fuel run.
- (6). *Mineral high speed Diesel oil:* Same as (2) above.
- (7). *Groundnut oil:* Same as (1) above.
- (8). *Groundnut oil:* Same as (2) above.

All runs were carried out under the following standard conditions:

Speed	... ..	= 1,200 r.p.m.
Air intake temperature		= 37.5 + 0.5°C.
Radiator water outlet temperature		= 80.0 + 0.5°C.
Compression ratio 2. H. P. to full load		= 15 : 1 .
Compression ratio 2 H. P. load and less		= 19 : 1
Room temperature		= 17.5—19.5°C.
Relative humidity		= 47—64 per cent

Tests (1) and (2) were uneventful. During test (3), the first sign of trouble occurred after 20 minutes' run at full load, from when onwards the injector pin gave no indication of movement. However, as the engine continued to run without misfire or drop in speed, the tests were continued. Test No. (3) was finished 55 minutes from start and was followed immediately by test (4). When this test had been in progress for 35 minutes, there was a drop in maximum load and some sound presumably from inside the fuel pump. This sound persisted from then onwards. After a further run of 30 minutes, i. e. 2 hours from the start of colloidal fuel run, when the compression change-over-lever was thrown into high compression for test at low load, the compression change-over-valve started leaking. Thereafter the engine started misfiring and increase of load, change of compression, etc. had no effect. At 2 hours

and 14 minutes from the start of colloidal fuel run, the engine stopped by itself. Attempts to revive it by turning on mineral oil failed. Even after the fuel system was cleaned of colloidal fuel and primed with mineral fuel, the engine could not be started.

The injector valve was found to have been struck. This was released and the fuel pump cleaned. On re-assembling, the fuel spray appeared to be normal but the engine did not start. On opening out the engine, the inlet valve face and seat were found to be clean but some muddy coloured deposit was found on the exhaust valve seat and in the main and the auxiliary combustion chambers. There was some carbon deposit on the cylinder head piston top and also on the compression ratio change-over-valve seats. After cleaning and re-assembling, the engine worked satisfactorily. Tests (5) to (8) were then completed on another day.

The engine exhaust gave black smoke with mineral oil and trace of black smoke with groundnut oil at maximum load. Exhaust was clear in both cases at other loads. With colloidal fuel at full load, exhaust emitted less black smoke than mineral oil, but a trace of black smoke persisted even at reduced loads. During full load runs on colloidal fuel cooling water had to be fed into the radiator at a slightly increased rate to maintain standard temperature, showing thereby that the engine ran hotter with this fuel.

Results of the various tests have been plotted in Figs. 1, 2 and 3. Exhaust gas analysis is given in Table II. The pump plunger lost 2.2 mgs. in weight and 0.00005" in diameter, when measured by a light-wave

TABLE II. — Exhaust Gas Analysis

Fuel	B.H.P. at the time of gas sampling.	% by volume				Ratio of air supplied to air burnt.
		CO <sub>2</sub>	O <sub>2</sub>	CO	N <sub>2</sub>	
1. Charcoal colloidal fuel, 35%	4.8	9.3	5.4	2.0	83.3	1.32
2. Mineral oil.	6.8	10.2	3.3	0.0	86.5	1.17
3. Groundnut oil.	6.8	10.3	2.4	0.8	86.5	1.11

micrometer correct to 0.00001". The injector hole was enlarged in diameter by 0.0008". This wear has to be attributed to the colloidal fuel because short runs on mineral or groundnut oil cannot be held responsible

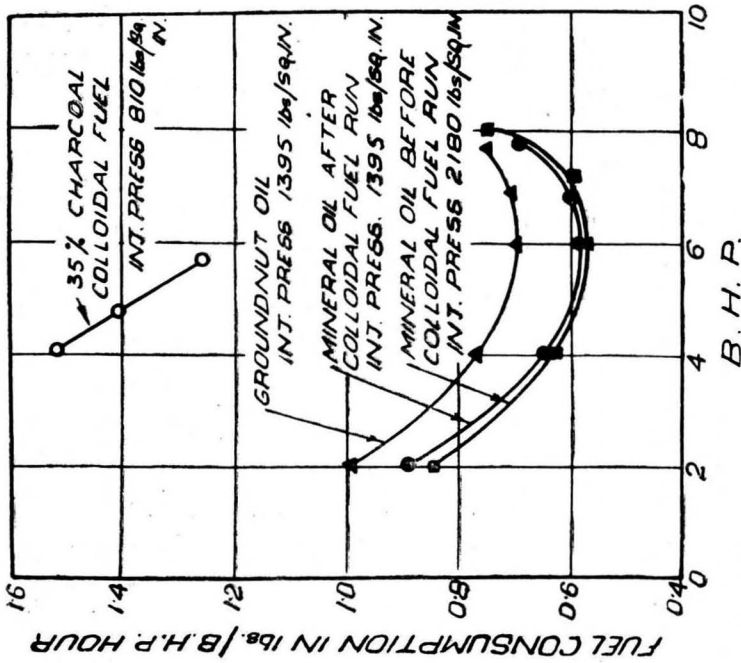


Fig. 2. B.H.P. Vs Fuel Consumption.

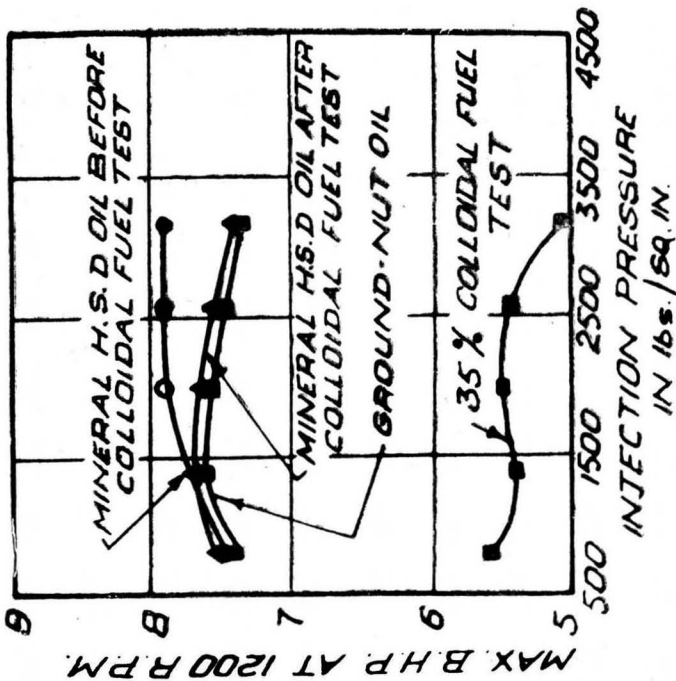


Fig. 1. Injection pressure Vs Maximum B.H.P.

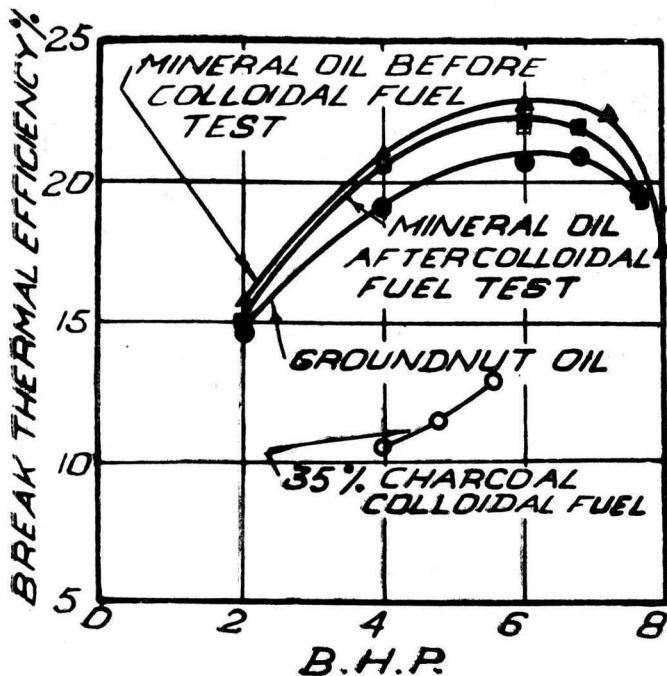


Fig. 3. B.H.P. Vs Break Thermal Efficiency.

for it. Performance figures obtained after colloidal fuel run show a slightly lower maximum B.H.P. and higher fuel consumption, thereby showing the effect of wear. The immediate damage to the injection system has not been much but the rate of wear is too high for continued operation.

The maximum B.H.P. developed with groundnut oil was 98.7 per cent of that with mineral oil while with colloidal fuel it was only 72.8 per cent. The best brake thermal efficiency with groundnut oil was 20.8 per cent as against 12.8 per cent. with colloidal fuel. Excess air calculated from exhaust gas analysis was 11 per cent. with groundnut oil and 32 per cent. with colloidal fuel. These figures and exhaust smoke go to prove that with colloidal fuel the mixing of air and fuel inside the engine was not efficient. However, in an earlier short run with colloidal fuel, starting with clean injection system, a thermal efficiency of 16.8 per cent. was obtained. In the present case it is likely that the performance of the injection system

deteriorated prior to fuel consumption tests which were begun after the engine had already run on the colloidal fuel for one hour. These figures are, therefore, not a true guide to the efficiency with which the fuel itself could be burned inside the engine if properly injected.

Studies of injection characteristics of the fuel showed that the colloidal fuel, due to its high specific gravity and viscosity, formed only a narrow cone as compared with that of mineral oil. It is most likely that the colloidal fuel inside the engine penetrates in the form of a narrow pencil and strikes the walls of the combustion chamber instead of dispersing uniformly as a spray in the compressed air. Had the fuel been injected in a wide-angled cone by the use of a different nozzle, e. g. of the pintle type, better fuel atomisation and higher thermal efficiencies might have been obtained.

It is concluded that the injection system cannot stand up to the abrasive action of the colloidal fuel as prepared and used. The



idea of using colloidal fuel itself, however, seems to hold out promise for the following reasons:

(1) With the use of a stabilizer prepared out of the oil itself, colloidal fuels containing as high as 35 per cent. by weight of charcoal can be prepared, handled like liquid fuel and stored for lengthy periods without the charcoal settling down.

(2) Provided the injection problems are solved, chances appear to be that thermal efficiencies approaching that of groundnut oil can be achieved with colloidal fuel so that the charcoal part of the fuel can be burned as effectively as the oil itself.

Further work towards reducing the wear of the injection system by using charcoal

particles as small as  $2 \mu$  and redesigning of the injection system is proposed to be carried out.

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## Retting of Coconut Husks

### PART I—PROXIMATE ANALYSIS OF COCONUT HUSK AT VARIOUS STAGES OF RETTING

By N. S. VARIER AND K. L. MOUDGILL

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THE production of coir fibre from coconut husk is the most important cottage industry on the West Coast of India, especially in Travancore. Whereas in the other coconut producing countries of the world, coir is prepared in small quantities from coconut husk by mechanical means, it is produced in this area almost entirely, by retting.

India produces about 15 per cent. of the world's output of coconut, out of which Travancore's share is about 35 per cent. Patel<sup>2</sup> estimates that about a quarter million people are employed in this industry on the West Coast, and that about 750 million husks are retted annually.

The importance of coir in the export trade of Travancore will be apparent from Table I.

TABLE I

Year	Total trade Crores of Rs.	Quantity Cwts.	Trade in coir Value Rs.
1936-37	9.23	5,94,362	1,76,99,647
1937-38	9.43	6,91,569	1,64,10,624
1938-39	9.58	7,08,102	1,78,46,019
1939-40	11.17	6,68,686	2,12,30,134
1940-41	9.96	5,36,483	1,28,19,098
1941-42	15.38	6,80,248	1,37,87,419
1942-43	16.54	5,41,254	1,07,55,020
1943-44	25.52	—	2,57,37,000
1944-45	31.90	—	4,03,00,000

The process of retting is briefly as follows: Fresh ripe husks obtained after the removal of nuts are bundled into lots of about 10,000 in coir nets called *malis*. The mass is loaded with stones and mud, and kept immersed in the saline backwaters for 6-10 months. During this process changes, mainly bioche-

mical, take place and the binding materials connecting the fibre with the pith in the husk are removed, the tidal action of the backwaters, helping to wash away the unwanted products.<sup>4</sup> The material is taken out and the fibre separated from the pith by beating with wooden mallets. Alternately, and to a lesser extent, the soaking is done in small shallow pools, where tidal action is not possible. This yields "beach yarn", which is of poor quality. In a third type of retting, called "pit-retting", husks are buried in pits dug on the banks of the backwaters, and the ebb and flow of water at the top and the free percolation of sub-soil water below help to wash away the products of disintegration. These pits may contain as many as 20,000-30,000 husks.

The fibre so obtained is better than "beach yarn" and is sometimes of as good a quality as that obtained by direct soaking.

Coir is made from coconut husks in almost every village on the banks of the lakes and backwaters of the West Coast by one or the other of the above processes. Conditions of soaking, the nature of soil, and the extent of tidal flow differ from place to place and various grades of fibre, (e.g. *Anjengo*, *Aratturai*, *Astamudy*, *Vaikom*, etc.) are obtained.

Though considerable scientific work has been done on the retting of jute and flax, with the exception of the work published by Fowler and Marsden very little seems to have been done on the retting of coconut husks. On a scheme financed (annual endowment, Rs. 1,000/-) by Mr. S. Krishna Iyer, the Central Research Institute of the Travancore University started work in 1939 on the retting of coconut husks and utilisation of by-products. The objects of the study were:

- (1) to study the chemical changes during retting,
- (2) to shorten the duration of retting,
- (3) to improve the quality of fibre, and
- (4) to find uses for the by-products.

Besides the laboratory work at Trivandrum, three field stations were opened in Thottapally, Kayamkulam and Perumkuzhy, to study retting under different conditions. These stations are representative of the three main areas of coir production and of the varying conditions of salinity of water which

plays an important role in retting. At Thottapally the water is saline only for a limited period of about 2 months in the year, while at Kayamkulam this period extends to about 6 months. At Perumkuzhy, which is close to the bar, excepting for a short period, the water is more or less saline throughout the year. The extent of salinity varies in each place but seasonal periodicity was observed during a weekly survey covering a period of 4 years.

To study the chemical changes during the course of retting, methods for the analysis of coconut husk in terms of (a) moisture, (b) ash, (c) fats and resins, (d) cellulose, (e) lignin, (f) pentosans and (g) pectin were first standardised. Coir fibre has been analysed in the chemical laboratory of the Dacca University<sup>5</sup> and by S. R. K. Menon<sup>6</sup>. The proximate methods used in the present study are mostly similar to those recommended by Doree<sup>7</sup> with slight modifications.

## Experimental

20 husks from the same bunch of a coconut tree (of the quality used for retting) were divided into 2 lots. One lot was soaked at Perumkuzhy (one of the field stations) and the other lot was kept submerged under running tap water in the laboratory. Samples were taken from both the lots at periodic intervals and analysed. The results obtained are given in Tables II-V.

TABLE II

Sample No.	Before	2A	3A	4A	5A	6A
	retting					
Duration of retting, months	0	I	4	7	10	12
Moisture, %	10.50	15.16	9.39	8.07	10.12	9.12
Ash, %	6.23	6.10	5.68	3.18	3.06	3.10
Fats & Resins, %	5.46	5.09	3.60	2.61	2.01	1.52
Lignin, %	47.60	45.91	39.96	42.44	42.46	41.20
Cellulose, %	44.00	45.28	47.21	46.22	45.10	43.40
Pentosans, %	16.21	15.23	15.10	14.14	14.02	13.90
Pectins, %	0.91	0.81	0.60	0.48	0.36	0.28

TABLE III

Sample No.	I	2B	3B	4B	5B	6B
Duration of retting, months	0	I	4	7	10	12
Moisture, %	10.50	14.20	11.70	8.02	10.84	9.44
Ash, %	6.23	5.12	3.59	3.09	3.07	2.96
Fats & Resins, %	5.46	5.34	1.91	1.86	1.21	1.20
Lignin, %	47.06	44.97	44.31	44.85	45.41	44.82
Cellulose, %	44.00	46.42	45.82	44.50	44.82	46.20
Pentosans, %	16.21	14.51	13.87	13.82	13.68	13.42
Pectins, %	0.91	0.62	0.21	0.18	0.12	Traces.

TABLE IV

Sample No.	1	2A	3A	4A	5A	6A
Ash, %	6.23	6.10	5.68	3.18	3.06	3.10
Fats & resins, %	5.46	5.09	3.60	2.61	2.01	1.52
Lignin, %	38.40	38.06	35.20	38.70	39.53	41.73
Cellulose, %	35.94	37.52	41.59	42.15	41.99	41.23
Pentosans, %	13.23	12.63	13.30	12.90	13.05	12.50
Pectins, %	0.74	0.67	0.53	0.44	0.34	0.11

TABLE V

Sample No.	1	2B	3B	4B	5B	6B
Ash, %	6.23	5.12	3.59	3.09	3.07	2.96
Fats & Resins, %	5.46	5.34	1.91	1.86	1.21	1.20
Lignin, %	38.40	37.77	40.19	41.26	41.78	41.68
Cellulose, %	35.94	38.99	41.56	40.94	41.23	42.87
Pentosans, %	13.23	12.19	12.58	12.71	12.59	12.25
Pectins, %	0.74	0.52	0.19	0.16	0.11	Traces.

Table II gives the results of analyses of the laboratory samples, (numbers 1, 2A, 3A, 4A, 5A and 6A.) and Table III those of field samples, (1, 2B, 3B, 4B, 5B and 6B.). Sample 1 was taken before soaking.

The different components are not mutually exclusive, and omitting the moisture content which is not significant, the total of the other percentages does not sum up to 100. As some of the components get reduced progressively, the total also varies. To get

comparable figures, the analytical data have been recalculated on the basis of a total index of 100. These recalculated results given in Tables IV and V are useful for comparison.

It is seen from the Tables that during retting, while there is a fall in pectin, pentosan and fat and resin contents, there is comparatively little change in lignin and cellulose. The change in pentosan is rapid. The ligno-cellulosic complexes present in coconut husk are attacked but little by the micro-organisms, but the pectins and pectocelluloses which probably form the binding material are effectively attacked.

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