

Journal of Scientific & Industrial Research

V. 9, No. 7, JULY 1950



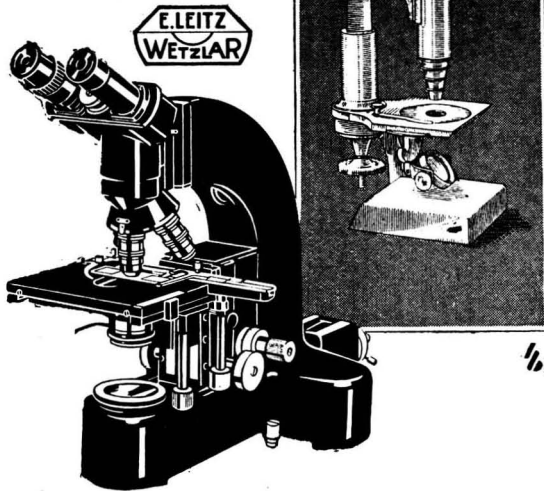
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COVER PICTURE

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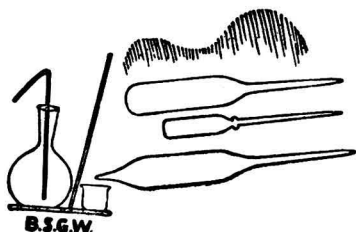
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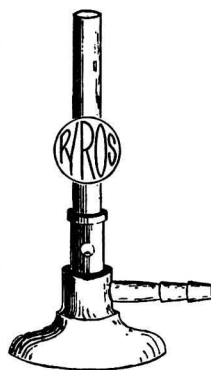


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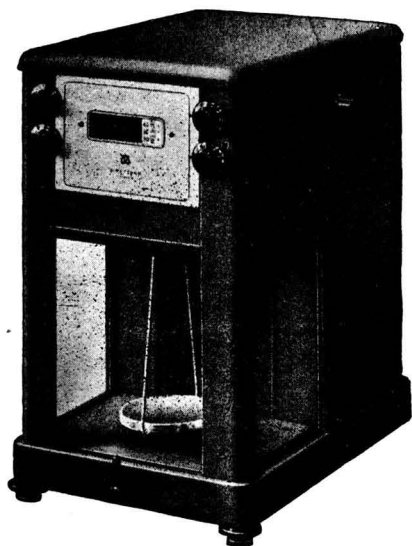
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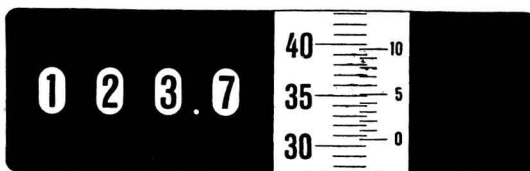
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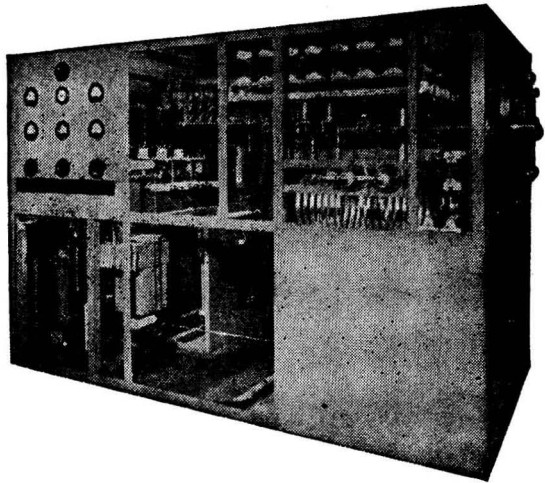
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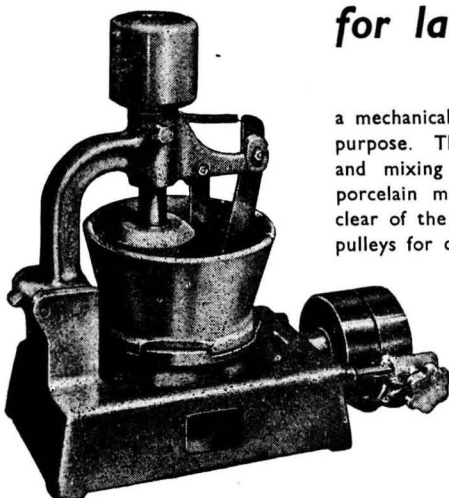
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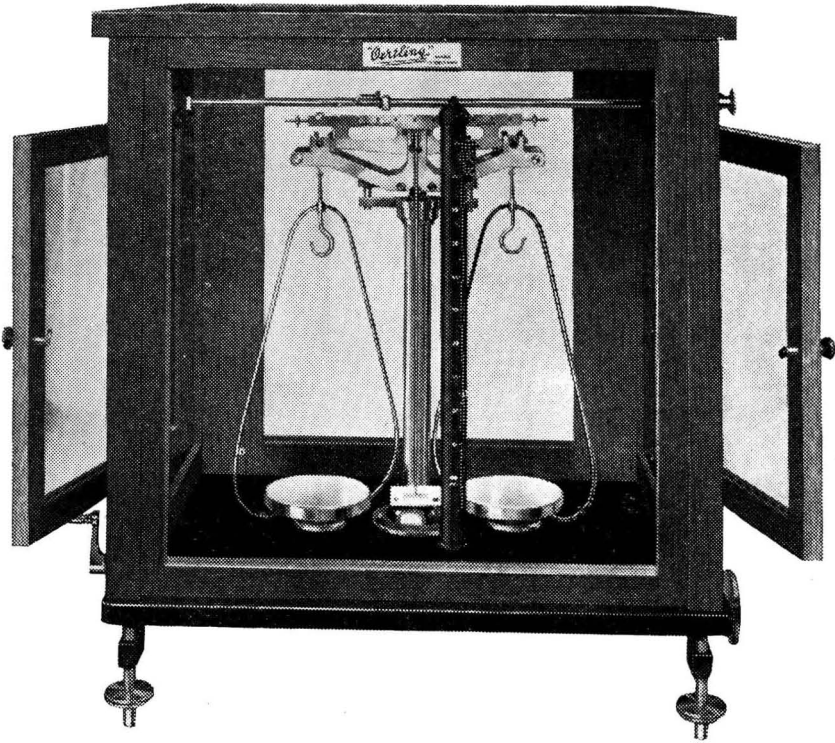
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THE setting up of a well-equipped Ore-dressing and Mineral Beneficiation Division of the National Metallurgical Laboratory, Jamshedpur, will be warmly welcomed by industries interested in the production and utilization of minerals. It is probably the best equipped laboratory in India, competent to undertake a wide range of standard beneficiation tests on ores.

There was little or no research in India on ore-dressing problems before World War II; there was hardly any institution with even the basic equipment required for systematic ore testing. The *Indian Copper Corporation Ltd.* had established a testing laboratory at their Moubhandhar Works, as an adjunct to the flotation plant, for assessing the changing characteristics of the ores. The Utilization Branch of the Geological Survey of India, which was started in 1942 for exploiting the deposits of strategic minerals, was faced with the problem of developing several low grade ores. At the request of the Geological Survey, the *Indian Copper Corporation Ltd.* placed their ore-testing laboratory at the disposal of the Government, and several investigations on beneficiation of low grade ores were undertaken. Towards the end of 1945, the Utilization Branch of the Geological Survey was wound up and all ore-testing work had to be stopped.

The Indian Bureau of Mines, set up by the Government of India in 1947, concerned as it is with the conservation and planned utilization of mineral resources of the country, is faced with the problem of utilizing the large quantities of low grade ores accumulating at mine sites during the last few decades. The utilization of these ores involves the testing of a large variety of materials from different parts of the country for their amenability to beneficiation

processes and the determination of optimum working conditions for recoveries.

The National Metallurgical Laboratory at Jamshedpur, which is one of the eleven National Laboratories established by the Council of Scientific & Industrial Research, has now set up a well-equipped ore-dressing laboratory* which would be in a position to undertake such investigations.

Scope for Ore Testing

The value of the total annual production of minerals in India is estimated to be 60 crores of rupees. India, however, is far behind other countries in the application of ore-dressing techniques to the development of her immense mineral wealth. Most of the manganese ore, magnesite, chromite,

* The Division is housed in one of the five bays (each measuring 100'×40') in the Technological Block. The crushing section is adjacent to the ore-dressing bay and is equipped with the following machinery: (1) one Denver laboratory forced feed jaw crusher, 5"×6", with 5 h.p. motor; (2) one Denver laboratory forced feed jaw crusher, 3½"×2¼", with 1 h.p. motor; (3) one Denver laboratory crushing roll (type M), 10"×6", with 2 h.p. motor; (4) one Denver McCool laboratory pulverizer, No. 9½K., 5 h.p. motor; and (5) one Denver-Dillon double-deck vibrating screen, 12"×24", with ½ h.p. motor.

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Batch grinding can be effected in a 12"×5" Denver (open) batch mill. Where a uniform feed without much fines is required, the Denver laboratory rod mill (7"×14") will prove serviceable.

ilmenite, zircon, monazite, kyanite, etc., produced in India is exported for processing abroad. Minerals intended for export should conform to certain grades and specifications, and to secure exportable qualities, selective mining and crude dressing method, e.g. hand-picking, have been adopted. These practices have led to the accumulation of appreciable quantities of unmarketable grades *in situ* or in dumps.

Taking the case of the manganese mining industry, it is reported that about 40 million tons of high grade ore have been exported from India during the past few decades. On a conservative estimate, about half of this quantity should have accumulated during all these years in the mining centres as low grade ore. It should be possible to recover at least half of this quantity as high grade concentrate through suitable process-

ing. This means that India's manganese ore reserves can be increased by 10 million tons from the old workings alone. The present average annual production of marketable manganese ore is estimated to be 6,00,000 tons. This results in an equal tonnage of low grade ore which goes into dumps, from which about 1½ lakh tons of marketable ore can be recovered. India possesses a minimum reserve of 15-20 million tons of metallurgical and chemical grade manganese ores, but the reserves of low grade ores are several times this quantity.

India has large reserves of iron ore containing over 60 per cent iron, sufficient to last for a few hundred years at the present rate of consumption. The fines produced during mining operations, which are not suitable for use in blast furnaces, are rejected at the mine site and only the coarse sizes are utilized.

For agitation of cyanide pulps, amalgamation tests and grinding with pebbles, a Denver bottle type agitator with 4 numbers of 24" rollers, which can take at a time 4 numbers of trojan type porcelain jars (capacity 1.4 gal.) is employed. The Denver super-agitator can be used for conditioning and agitating cyanide pulps at varying speeds and for the determination of settling rates. Three spherical bottom pyrex jars of capacities 6.6, 12.7 and 19 litres, are available for use with the agitator.

Four laboratory flotation machines (2 of 500 gm., 1 of 1,000 gm. and 1 of 2,000 gm. capacity) are installed for batch testing. Filtration of the test products is carried out in Denver vacuum filters (12"×3"), or counter-balanced pressure filters (12"), or filter flasks with Buchner funnels, whichever is convenient. The products are dried in a Denver laboratory oven No. 12.

A sieve shaker, which can hold 7 Nos. of 8"×2" sieves, is employed for screen analysis. Tyler standard sieves are in use for all sizing work and a complete set of these is kept in stock from 1.05" opening to 325 mesh, the openings decreasing in the ratio of $\sqrt{2}$ or 1.189.

The products are sampled with a Jones laboratory sample splitter (10"×18"). Conventional methods can be adopted for smaller quantities, using chilled iron bucking boards and mortars and pestles for grinding purposes.

A laboratory electromagnetic separator (type D, No. O), manufactured by *Stearns Manufacturing Co.*, is erected for magnetic separation of minerals, and one electrostatic separator (model B-1-12, of *Messrs Sutton, Steele & Steele, Texas*) will soon be installed for the separation of minerals with different electrical properties.

The Humphrey's spiral concentrator (No. 24A, single, 5 turns), which is to be installed shortly, would be useful for the separation of heavy minerals in batch or continuous tests.

Continuous Testing—The batch testing units and the working benches for sampling and processing occupy about one half of the bay. The conti-

nuous testing plants are to be erected in the other half. The following pilot plants will be shortly installed.

Dorr Company's counter-current decantation plant, consisting of 3"×10" dia.×10" deep agitator, 3"×18" dia.×12" deep thickeners and 3 diaphragm pumps. All parts coming into contact with solutions are made of stainless steel. This plant is useful for cyanidation of gold ores and leaching and other operations employing counter-current decantation.

A laboratory experimental sink-and-float plant (*Huntington, Heberlein & Co. Ltd.*, London) with a capacity of 200 lb. of ore per hour and employing galena as the medium. The preparation of the medium from raw materials, and the reclamation of galena will be carried out in the Denver continuous plant.

Denver Equipment Company's continuous pilot plant to treat 200-500 lb. of ore per hour. This comprises an ore-feeder, a ball mill, a cross-flow classifier, a hydro-classifier, a mineral jig, a unit flotation cell, 2 conditioners, 12 Nos. of No. 7 flotation cells, a spiral thickener, a drum filter, vacuum equipment, 4 wet reagent feeders, a dry reagent feeder, a pulp sampler, 2 sand pumps and 2 Wilfley tables (50"×24"). The crushing unit is not included in this plant as the ore will be reduced to the size suitable for the ball mill in the crushing section situated in the adjoining room. This continuous plant can be used for combined gravity (jigging and tabling) and flotation treatments and for selective flotation of one or more minerals.

The Ore-dressing Division is equipped with V-41 mineral light for the identification of minerals using ultra-violet light, a D.F.C. electric assay furnace with automatic temperature control for gold and silver fire assays, a Bausch and Lomb stereoscopic wide field binocular microscope, a Denver-Havard double beam trip scale, a solution and pulp scale, hand amalgamation pans and vanning plaques.

Many million tons of accumulated fines could be washed to produce high grade concentrates low in objectionable impurities. These and the blue dust containing over 68 per cent iron, available in large quantities, can be sintered and utilized in blast furnaces.

The magnesite and chrome ore mining industries in India are also operating under similar conditions. Only the hand-picked concentrates are utilized for export or local consumption. Low grade ores are rejected.

Indian beryl is obtained by hand-picking the crystals from the field. Sometimes hand-cobbing is adopted to separate the crystals from the matrix. It is reported that there are large reserves of beryl-containing rock in the country which can be mined and the beryl separated by flotation processes.

The occurrence of fluor spar has been reported from a few places in Central Provinces and adjoining States. At present India's requirements of this mineral are met almost wholly by imports. If the reported deposits are sufficiently large, though of low grade, it should be possible to process them for the production of metallurgical fluor spar. Acid grade fluor spar required for the manufacture of hydrofluoric acid can be, likewise, obtained.

The lead-zinc ores of Zawar (Rajputana), the apatite deposits of Singhbhum, the monazite-ilmenite sands of Travancore are among the other minerals that can be enriched by ore-dressing processes. Glass sands can be purified, and cryolite can be recovered from carbon dust of the aluminium reduction furnaces at Asansol and Alway by suitable

methods. These instances are only indicative of the range of materials that are amenable to treatment by beneficiation processes.

Service Facilities

The Ore-dressing Division of the National Metallurgical Laboratory is equipped for batch as well as continuous tests on minerals.

Batch tests provide all the requisite information on the amenability of minerals and rocks to different treatment methods and the optimum conditions for attaining efficiency with any of the processes. Although a commercial size plant designed and based on the results of batch tests can be reasonably expected to duplicate the results obtained in the laboratory, it is advisable that a continuous test on a pilot-plant scale should be performed. The results of such tests provide more reliable information than those obtained from batch tests, especially regarding the behaviour of middling products, the use of reclaimed water in the mill and the classification of flotation feeds.

The primary aim of this Division is to render every possible assistance to industries interested in mining and minerals utilization. The laboratory will undertake the testing of samples from deposits and provide specifications for machinery required for processing. The laboratory will also advise on the erection and operation of ore-dressing plants in the country.

G. P. CONTRACTOR
P. I. A. NARAYANAN

Census of Trained Personnel

THE INITIAL VOLUME OF THE NATIONAL Register of Scientific and Technical Personnel in India, published by the *Council of Scientific & Industrial Research*, catalogues the names of engineers under the following heads: civil, electrical, mechanical, chemical, marine and mining. The information furnished is classified further to give information on the academic and professional qualifications, practical experience—its nature and scope—,

addresses, present occupation and nature of employment held. Apart from being an effort towards the stock-taking of scientific personnel in the country, the National Register provides a continuous census of scientific manpower and will assist in the effective utilization of scientific talent, assessing gaps in the growth of science and technology and supplying service personnel to industry and to educational and research institutions.

Sir Edward Mellanby, Director, Central Drug Research Institute

SIXTY-SIX-YEAR old Sir Edward Mellanby, who has been appointed Director of the Central Drug Research Institute, Lucknow, is one of Britain's foremost medical scientists. Three years ago he was awarded the Buchanan Medal of the Royal Society "for his services to medical science and in recognition of his outstanding researches on dietary factors".

He has been a pioneer in the study of diet and nutrition, and for the last 16 years he has carried on his work concurrently with directing medical research throughout Britain, for he is the Secretary of the Medical Research Council.

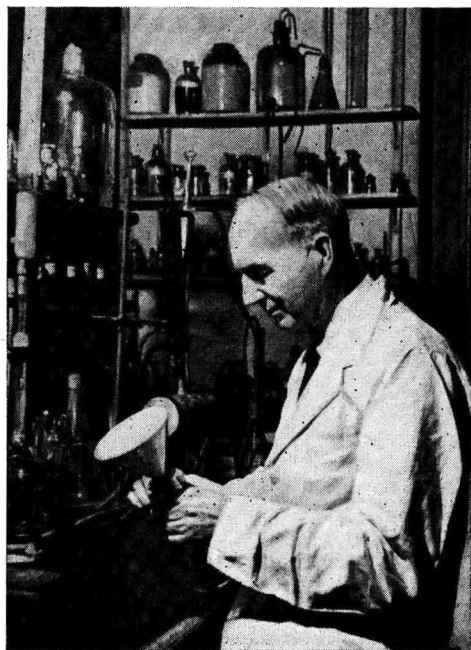
Born at West Hartlepool, County Durham, in 1884, he was educated at Barnard Castle School and Emmanuel College, Cambridge, where he was a scholar, research student

from 1905 to 1907 and later an honorary fellow. He took his M.A. and M.D., doing his hospital course at St. Thomas', London, where he afterwards began his teaching career as Demonstrator in Physiology. From 1913 to 1920 he was Lecturer and later Professor of Physiology in London University (King's College for Women); he then went to Sheffield as Professor of Pharmacology until 1933, when he received his appointment as Secretary to the National Medical Research Council. He had been a member of the Council for two years. In 1935 he was elected Fullerian Professor of Physiology to the Royal Institution.

Research on Rickets

One of Dr. Mellanby's early pieces of research concerned the physiological effects of alcohol, an investigation which he undertook for the Government during the first World War. Around this time the late Sir Frederick Gowland Hopkins was carrying on his research which led to the determination of the vitamins, and Dr. Mellanby also did pioneer work in this field. By dietetic experiments on puppies he found that rickets was caused by an absence from their diet of a fat-soluble substance which controlled the deposition of calcium in the bones. This anti-rachitic substance later received the name of vitamin D.

In 1925 the Medical Research Council published a report of further experiments by Dr. Mellanby on these lines, establishing the fact that bread and cereals tended in themselves to cause rickets; later he identified the rachitic substance in a cereal diet as phytic acid, and found that it could be counteracted by adding vitamin D or extra calcium. A later series of publications demonstrated that actual bone changes in shape could be caused by faulty diet; following up this line, Sir Edward — he received the K.C.B. in 1937 — proved that lack of vitamin A causes abnormal bone growth and neurological disorders due to pressure of the overgrown bone.



SIR EDWARD MELLANBY

His investigations have included a study of the effects of lack of iodine ; and one of his researches demonstrated that bread made from flour bleached chemically by the Agene process (NCl_3) can cause the condition in dogs known as canine hysteria.

In 1925 Dr. Mellanby was elected a Fellow of the Royal Society. He had become a recognized authority on nutrition, and was member of successive committees appointed to advise the Ministry of Health. He worked for the League of Nations, was Chairman of two International Conferences for the Standardization of Vitamins and of the International Technical Commission on Nutrition.

Sir Edward Mellanby was Honorary Physician to the King from 1937 to 1941. He held many other important positions during World War II. He was chairman of a committee which investigated and advised on the health of flying and naval personnel

and has been, since 1940, a member of the Scientific Advisory Committee to the Cabinet. In 1941 he was appointed a member of the Colonial Products Research Council, and in 1945, chairman of a medical research committee to advise on colonial problems.

Sir Edward Mellanby's wife, whom as May Tweedy he married in 1914, is like himself a distinguished scientist.

Lady Mellanby has specialized in research on the effect of diet on dental health ; the Medical Research Council published a three-volume report by her on this subject between 1931 and 1934. Her work on the teeth of London school children has been outstanding.

She is an honorary member of the British Dental Association and an investigator for the Medical Research Council ; in 1935 she and her husband were jointly awarded the Charles Mickle Fellowship of the University of Toronto.

Characteristics of the Ionosphere over Calcutta (April 1950)

S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY,
R. B. BANERJEE & A. P. MITRA

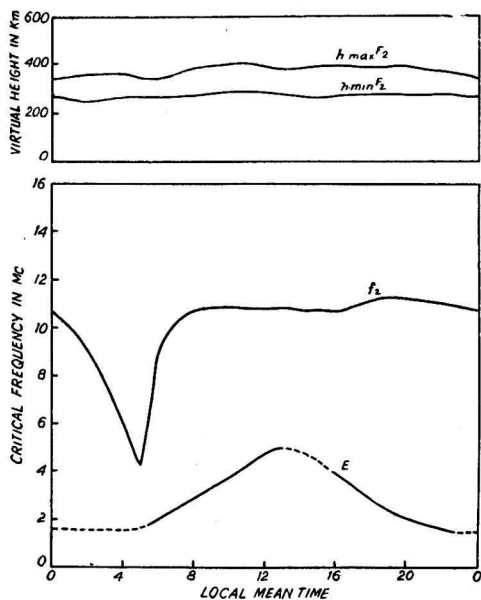
Ionosphere Laboratory, University College of Science, Calcutta

THE following are the ionospheric data observed at Calcutta for the month of April 1950.

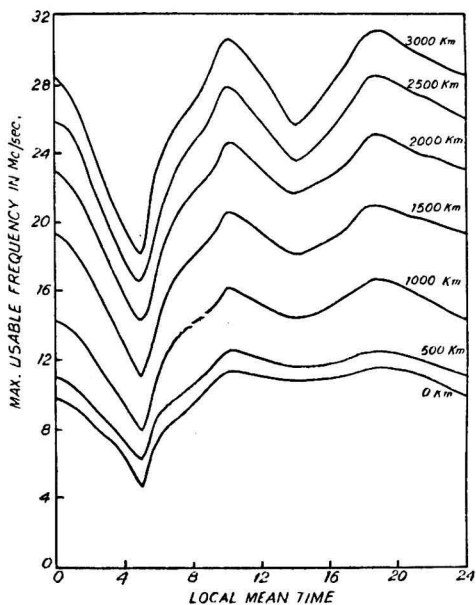
Fig. 1 represents the mean hourly values of the penetration frequency of region E and the penetration frequencies and virtual heights of region F_2 in graphical form. The figures are obtained from average values of the data taken for each hour of the day for 5 days a week. Fig. 2 gives the prediction of the maximum usable frequencies which can be used for different distances of transmission by reflection at the F region over Calcutta for the month of July 1950. Table I gives the different occasions during routine observations when sporadic E ioniza-

tion was observed and the values of the corresponding penetration frequencies and heights.

Sporadic E ionization was found to occur frequently during evening and night with infrequent occurrences in the mornings. The frequency of occurrence was found to be less than that at the same time last year. It is of interest to note that the weather was less turbulent this year than last year. This suggests a relation between weather conditions and occurrence of sporadic E. Normal E echoes were frequently absorbed during mid-day and early afternoon region F_2 ionization was found to remain steady up to midnight with a slight rise late in the evening.



(5 HR. 54 MIN. AHEAD OF G.M.T.)
FIG. 1 — APRIL 1950.



AT POINT OF REFLECTION.
FIG. 2 — PREDICTED M.U.F. VIA F_2 LAYER DURING THE MONTH OF JULY 1950.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	h_{E_s} Km.
April 1950	4	00.00	5.00	140
		20.00	3.75	125
	6	18.00	2.45	100
		22.00	2.20	90
		23.00	2.25	105
	11	09.00	3.00	125
		19.00	4.15	135
		17.00	3.35	130
	15	22.00	3.00	130
	18	23.00	2.25	105
	19	07.00	5.00	140
	20	18.00	3.00	130
		19.00	2.00	105
		20.00	2.50	105
21.00		2.10	105	

TABLE I—contd

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	h_{E_s} Km.
April 1950	21	10.00	6.20	135
		18.00	3.50	130
		21.00	4.25	140
		22.00	3.00	135
	22	23.00	3.00	130
		00.00	3.00	130
	27	08.00	10.00	140
		17.00	4.85	120
		21.00	1.80	105
		22.00	1.70	105
	28	06.00	3.00	120
		18.00	4.00	125
		20.00	3.00	120
		21.00	2.80	120
22.00		2.65	120	
29	23.00	5.00	125	
	00.00	3.00	130	
	08.00	4.00	135	

Detonation & Molecular Structure

G. BROERSMA

Djakarta, Indonesia

SOME years ago the author published a report¹ on detonation measurements in the so-called Duchene apparatus (to be compared with a single stroke engine), which consisted of flame pictures, pressure and ionization diagrams. As a result of this and other researches, two mechanisms of detonation were indicated, viz. (1) the mechanism defined as physical detonation, in which by physical processes of aerodynamic flow, mixing and diffusion a critical stage is reached at which the speed of the flame front changes from the normal subsonic rate to a supersonic rate; and (2) the mechanism defined as chemical detonation, in which by chemical processes of chain reactions in the end-gas a critical stage is reached when the end-gas ignites at one or more points. From these points combustion proceeds at normal subsonic speeds. Each one of the two mechanisms has its own time constants, depending upon thermodynamic, chemical and geometric conditions of the vessel in which the combustion takes place. They may, under certain circumstances, overlap; that is to say, when chemical detonation has started physical detonation may follow, but not the reverse.

Another attempt to combine "auto-ignition" and the detonation wave theories into a single theory has since been made by Miller². According to Miller, detonation of a comparatively low pitch may be caused by ignition of the end-gas itself at a rate too slow to produce audible gas vibrations. Detonation involving both low-pitched and high-pitched tones may be caused by ignition of the end-gas followed by the development of a detonation wave in the end-gas. Detonation of high pitch may be caused by a detonation wave in after-burning gases behind the flame front. This wave may also pass through the un-ignited end-gas. The finding of Miller on the explosive detonation reaction is that it is a self-propagating disturbance travelling through the last parts of the gas burning at a

speed ranging about the speed of sound to approximately twice that speed.

Chemical Detonation

In considering the mechanism of chemical detonation, one may speculate on the relation between the structure of the hydrocarbon molecules and this type of detonation. (*Mutatis mutandis*: on the influence of detonation inhibitors or of detonation accelerators.) What happens in the burning gas and also in the end-gas may lead to chemical detonation. As has been shown³⁻⁵ time has a great influence on the occurrence of detonation, i.e. the faster the combustion proceeds, the less time there is for reaching the critical stage of "auto-ignition" of the end-gas. From this point of view, if the structure of the hydrocarbon in any way influences the detonation, it can manifest in two ways: firstly by influencing the speed of combustion of the original flame front; secondly by influencing the reactions in the end-gas.

Reynolds and Ebersole⁶ published measurements on the effect of hydrocarbon type and chain length on uniform flame movement in quiescent fuel-air mixtures. They found that the peak-flame velocity increased with increased unsaturation in a molecule of a given size. That is to say, *cet. par.*, that as reactions in the end-gas are allowed a decreasing amount of time to develop towards a critical stage, the chance of detonation decreases. For the unsaturated compounds, the peak-flame velocity decreases as the chain length increases, i.e. *cet. par.*, that as events in the end-gas are allowed an increasing amount of time to develop towards a critical stage, the chance of detonation increases. The peak-flame velocity of the saturated straight-chain compounds was essentially the same.

Detonation & Molecular Structure

The experiments of Lovell⁷ give an insight into detonation and the molecular structure.

The outstanding characteristic of hydrocarbons is the quite regular and consistent relationship between their tendencies to detonate and their molecular structure.

From the data given for all classes mentioned, it appears that (1) the critical compression ratio — before detonation starts — decreases when successive carbon atoms are added to a straight chain; (2) the critical compression ratio increases when the atom is added so as to form a branch; and (3) the critical compression ratio increases when the molecule becomes more centralized.

The results presented in the report of the American Institute of Petroleum Research, No. 45 (A.P.I. 45), are overall results and do not definitely show where the quality of a fuel becomes manifest, whether in the speed with which the gases burn during combustion — that is its influence on the time factor — or in the way events happen in the end-gas (or — before this — during the intake and/or compression in the engine, or in both).

As for these "events" in intake, during compression and in the end-gas during combustion, one is accustomed to speak of (1) the incubation period; (2) the pre-reaction period; and (3) the combustion period.

To start the so-called pre-reactions, some dissociation must be present. It is supposed that such dissociation is present under the usual conditions in the intake; moreover, it increases with increasing temperature when the mixture proceeds along the intake, comes into the cylinder, and finally is compressed. The initial dissociation will be caused by the impact of the various molecules. As the speed of the molecules increases with increasing temperature, dissociation will also increase.

Increasing temperature will increase the initial number of reacting molecules and the reactions will be accelerated.

During the pre-reaction stage, all kinds of compounds may be formed as, for instance, amines, amides, alcohols, aldehydes, peroxides, ozonides and acids. During the pre-reaction period, one compound may be transformed into another. With normal combustion, if the flame front starts and passes through the mixtures, everything breaks down again into CO_2 or CO , H_2O_2 and N_2 . In chemical detonation, however, there first will be a building up of these compounds, then a

critical stage will be reached at which they will break down faster than they are being built up due to temperature conditions. This faster breakdown causes the mixture to combust internally, i.e. before the original flame front has passed or, in extreme cases, even before it has started. In Lovell's report indications are available of the influence of the pre-reaction period when the use of super-fuels is referred to. In the above-mentioned compounds the occurrence of oxygen compounds is conspicuous. There are definite indications that these compounds play a considerable part in the course of the pre-reaction stage^{4,5,8}.

It is proposed to study the above "events" more carefully, especially from the point of view of molecular structure, detonation inhibitors and detonation accelerators. The case of paraffins, olefines, di-olefines, naphthenes and aromatics will also be considered.

The Molecular Structure of Hydrocarbons

The molecular structure of a hydrocarbon has to be taken spatially, although, for the sake of simplicity, one is usually satisfied with the chemical structural formula as written on paper. Even if the plane of symmetry were to contain the centre of gravity of the nuclei electrons, even then the molecule will have another dimension, be it infinitely small as compared with other dimensions.

Paraffins — If the molecules are looked upon as fixed moving objects, it is clear that with a straight-chain hydrocarbon the longer it is, the more chance there is for it to collide with another object and, if this is a small one, to "catch" it (FIG. 1). Also, if such a collision occurs between two big objects,

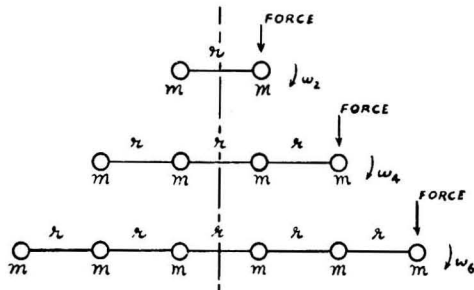


FIG. 1 — COMPARISON OF STRAIGHT-CHAIN MOLECULES OF DIFFERENT LENGTHS (FORCE AND W DO NOT OCCUR AT THE SAME TIME).

the longer the chain the greater the chance of the chain breaking down.

Comparing inertia forces of chains with 2, 4 and 6 carbon atoms, it follows that the longer molecules offer higher resistance to movement than the shorter ones. If the force is applied with a certain speed, a certain amount of energy must be transmitted. A molecule will transmit part of the energy of its rotation and another part into its inner construction, that is to say, that the bonds will have to absorb that energy. The less is absorbed by the rotational movement, the more will have to be taken up by the bonds. So the longer molecule will have a larger load applied to its bonds than has the smaller one, and stands a higher chance of breakdown. Therefore, the longer a straight-chain hydrocarbon the greater the chance for it to attach to itself another atom such as oxygen and/or nitrogen; but if the temperature increases and with it all the speeds of the molecule, the greater is also the chance that it will break down again.

The movement of the molecules is proportional to the temperature, and the energy of the molecules is equally divided over the possible degrees of freedom: translational, rotational and vibrational. If the temperature increases, then these energies increase.

The translational speeds of the molecules are not equal but vary according to a distribution first used by Maxwell. We shall, however, confine ourselves to average speeds (and energies) in this paper.

If hydrocarbons have the same temperature, they will have the same amount of energy if a calculation involving rotational energy shows, in the case of the three molecules, that the centrifugal force on the bonds is larger as the molecule is shorter, under the same temperature conditions. But as this

force is opposed by attractive forces of a kind inversely proportional to the radius, a smaller molecule will shorten and, one may say, stabilize itself somewhat more than a longer one. No actual change in the volume of the molecule need occur; only the attractive fields on force which surround it diminish in strength and it seems, therefore, as if the molecule has decreased in volume. The shorter molecule, therefore, will less easily attract other atoms, e.g. dissociated oxygen and/or nitrogen atoms than a longer molecule⁹.

In order to appreciate the difference between a straight-chain molecule and a centralized one, one might, for instance, compare a straight chain of 5 atoms (or groups) with a compact molecule wherein the bonds extend radially from a central atom to 4 other atoms (FIG. 2). The figure shows the projections of both molecules on the plane of symmetry, i.e. the plane perpendicular to the vector of the maximum moment of inertia. The straight-chain molecule extends farther into space in one direction; on the other hand, the compact molecule extends into space more evenly into two main directions. From a geometrical point of view, the chances of catching dissociated atoms of oxygen and/or nitrogen appear to be equal for both types of molecules. The difference in the chances that they may break down would be the straight-chain molecules of different lengths. From considerations of inertia forces it can be argued that the bonds of the straight-chain molecule will be more heavily loaded than those of the compact molecule at a collision; the straight-chain molecule will have a greater chance of breakdown.

Comparing what happens when both molecules are subjected to the same tem-

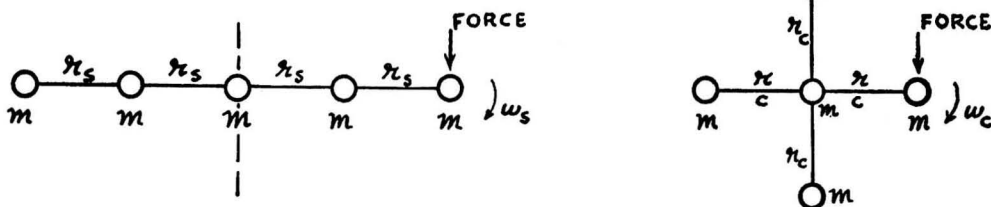


FIG. 2—COMPARISON OF A STRAIGHT-CHAIN MOLECULE WITH A COMPACT MOLECULE (FORCE AND ω DO NOT OCCUR AT THE SAME TIME).

perature, it is found that the centrifugal force on the bonds is the average for the compact molecule again involving calculations of rotational energies. By applying the same reasoning as applied before to the straight-chain molecules, it now appears that the compact molecule will attract less easily other atoms as, for instance, dissociated oxygen and/or nitrogen atoms than a straight-chain molecule of the same number of atoms, mass and bond dimensions. From this it follows that with increasing chain length, i.e. with increasing number of carbon atoms, the quantity of pre-reaction products will increase. These products will also be less stable as the chain is longer. Therefore, with an increasing number of carbon atoms, the straight-chain paraffins will have an increasing inclination to detonate, *cet. par.*

Furthermore, the more compact the molecule, the fewer and more stable will be the pre-reaction products. Therefore, with increasing compactness the paraffins will have a decreasing inclination to detonate, *cet. par.*

Both these findings agree with A.P.I. 45, from which it may be seen that the critical compression ratio decreases with the increasing number of carbon atoms and increases with increasing compactness.

The difference shown in A.P.I. 45 as to the influence of the centralization of the molecule, or of adding methyl groups is a difference in the compactness of the molecule. Centralization, which indicates the greatest benefit with respect to the allowable compression ratio, is actually the best way to make a molecule stable and compact. Adding methyl groups instead of hydrogen atoms, however, has a stabilizing influence with a lower benefit as to allowable compression ratio than has centralization.

Olefines — The olefines have the same general formula as the naphthenes C_nH_{2n} ,

but are of open structure, whereas the naphthenes are of cyclic construction. Compared with the paraffins, the olefines have two hydrogen atoms less; they fail at adjacent carbon atoms (FIG. 3).

A kindred consideration as for the paraffins holds good also for the olefines. Typical differences are due to the double bond in the molecules of the olefines so that the pre-reaction products of the olefines are formed much more easily than those of the paraffins. It appears that the olefines have a strong inclination to add atoms or groups to carbon atoms with double bonds. There will be more pre-reaction products with olefines than with paraffins under similar conditions. On the other hand, the resulting molecules are more stable than the original ones. That is to say, compared with the intermediate products of the paraffins originated during the pre-reaction stage, those of the olefines are more stable. Under similar conditions, therefore, there would be less chance for a sudden breakdown of the pre-reaction products of the olefines than those of the paraffins. Thus, if it is known which of the two properties dominates in a certain olefine, it can be determined whether an olefine is more or less inclined to detonate than the paraffin having the same number of carbon atoms. It may be stated, however, that the addition of oxygen or nitrogen makes itself felt more if there are fewer carbon groups, for then the influence of the double bond is greater.

Comparing olefines and paraffins, the difference between pre-reaction products should be greatest between the molecules with the least number of carbon atoms, and should decline with the increasing number of carbon atoms. Also the difference between the inclination to detonate would be largest between the molecules C_2H_6 and C_2H_4 , and this would decrease with the increasing

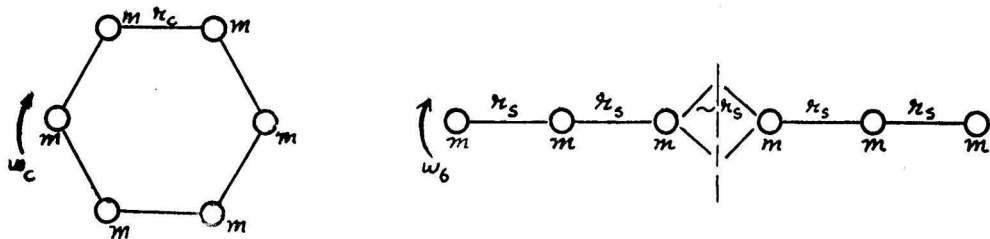


FIG. 3 — ELECTRON BOND DIAGRAMS FOR DIFFERENT MOLECULES.

number of carbon atoms. This is confirmed by A.P.I. 45.

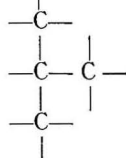
Di-olefines — The di-olefines have two double bonds as compared to the olefines. The general formula is C_nH_{2n+2} . It is to be expected that in their behaviour the di-olefines will show properties which are similar to those of the olefines, though in a more pronounced degree.

Naphthenes — The naphthenes are saturated hydrocarbons of cyclic structure. The molecules seem to be rather flattened. The general formula is C_nH_{2n} . The cyclic construction causes compact molecules to "catch" stray oxygen or nitrogen atoms with some difficulty. Their stability, considering their symmetrical cyclic construction, is favourable. Comparison shows that their bonds are taxed higher than straight-chain molecules with the same number of carbon atoms under similar temperature conditions (FIG. 4).

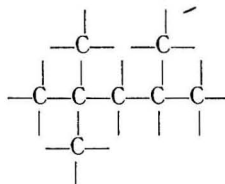
It may be shown that the centrifugal force on the bonds of cyclohexane, for instance, is higher, being 50 per cent above the average, than any of the forces on the bonds of the olefine. The field of force, therefore, is smaller around cyclohexane than around the corresponding straight-chain molecule C_6H_{12} (see FIG. 4). Cyclohexane will, therefore, less easily attach stray atoms to itself than C_6H_{12} . Cyclohexane is an example of a structure where the connecting bonds of the carbon atoms nearly lie in the direction which is natural to them¹⁰. This also causes it to be very stable. This is even more the case with cyclopentane. In accordance with the foregoing, it can be easily deduced from A.P.I. 45 that cyclohexane is superior to straight chain C_6H_{12} , and cyclopentane is superior to cyclohexane.

Aromatics — The basic aromatic is benzene, C_6H_6 , whence all the others are derived. Benzene has hexagonal cyclic structure, with three double bonds (it is possible that the three bonds move around the molecule). Often a structure is assumed with six bonds directed centrally. This last representation forcibly accounts for the good anti-knock properties of benzene and its derivatives, as may be inferred from A.P.I. 45.

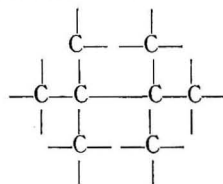
The Structure — From A.P.I. 45 it is conspicuous that the group



has very favourable anti-knock properties. One can see it in *iso*-octane



and still more in the octane



which is twice as resistant to detonation as the former.

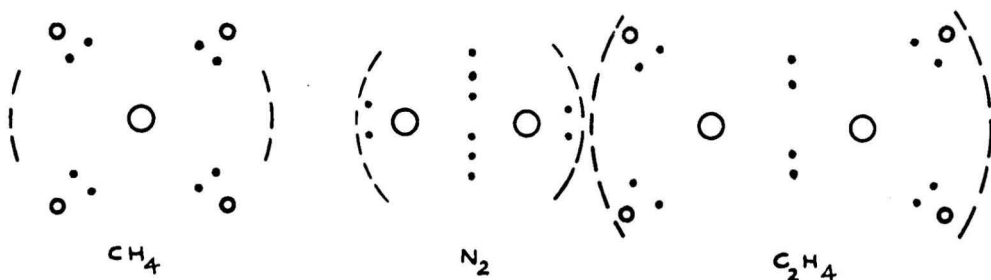
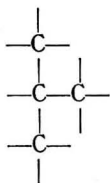


FIG. 4 — COMPARISON OF A NAPHTHENE MOLECULE WITH AN OLEFINE MOLECULE WITH THE SAME NUMBER OF CARBON ATOMS (A DOUBLE BOND IS OPEN; see FIG. 3).

Furthermore, in all hydrocarbons where this group is added, the anti-knock properties increase many folds. This is very likely due to the following. The group



will have the connecting bonds of the carbon atoms in their natural direction and will be of very stable construction. When added to a hydrocarbon, it either dominates the rest, or else may stabilize the rest by calling for an opposite and also stable construction by its field of force and mass distribution.

Sensitivity — The characteristics mostly used for the designation of hydrocarbons are the anti-knock value (octane number) and the permissible compression ratio. These are measured on standard engines under standardized conditions. If, however, the engine or the conditions are changed, these characteristics generally attain another value. This property of a hydrocarbon might be called its sensitivity.

A.P.I. 45 gives measurements where the critical ratio, under one set of conditions (C.F.R.-F-1, research method), has been plotted against the critical compression ratio in the same engine under another set of conditions (C.F.R.-F-2, motor method) for paraffins and aromatic hydrocarbons. Paraffins appear to be the least sensitive. Under severe conditions, however, their sensitiveness increases appreciably (the only exception being *iso*-octane). Some aromatics are very sensitive, others less or not at all. Cyclo-paraffins are more sensitive than paraffins.

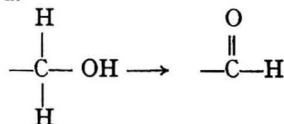
It may not be far-fetched to connect sensitiveness with the pre-reaction stage of the combustion. An aromatic like benzene, for instance, may under certain conditions form an ozonide, which is very unstable and has "explosive" properties¹⁰, i.e. it breaks down suddenly and causes auto-ignition of the end-gas, and therewith detonation. The sensitiveness of benzene is great for these particular conditions. If another aromatic with side chains, under any conditions in the intake and the compression stages, does not go beyond some aldehyde or alcohol

formation, then that aromatic shows little sensitivity.

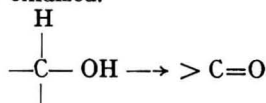
Pre-reactions — To fit in our problem more or less in the general picture of combustion, it may be considered in three stages: (1) the incubation period, in which first contact (collisions) occur between the hydrocarbon(s) and the oxygen and nitrogen of the combustion gases; (2) pre-reaction period, in which oxygen and nitrogen of the air are "reacting" with the hydrocarbon(s), i.e. all kinds of oxygen and nitrogen compounds are being formed; and (3) complete combustion in which the hydrocarbons are broken down completely to CO₂, CO and H₂O.

Of these three stage, we may discuss the second stage in chemical formulae, viz. formation of alcohols, aldehydes, peroxides, ozonides, amines and amides. In all cases, we shall consider compounds having the least number of oxygen and/or nitrogen atoms attached to the hydrocarbon. If the temperature conditions in the intake, the carburettor or the cylinder (during compression) allow the formation of the compounds mentioned in a certain engine, and if some of the formations are preparatory to detonation, then a hydrocarbon is regarded as "sensitive". The reactions mentioned will be examined for paraffins, olefines, diolefines, naphthenes and aromatics.

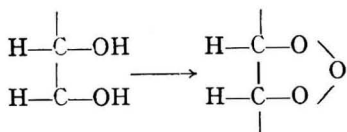
Paraffins — Methane and ethane only produce primary alcohols, propane may produce a secondary alcohol (in its isomers). Butane may produce a tertiary alcohol. From the primary alcohol an aldehyde may be derived.



In the aldehyde occurs a group $\begin{array}{c} || \\ -\text{C}- \end{array}$ and a hydrogen atom which is so easily oxidized that an aldehyde withdraws oxygen from the somewhat unstable oxides. Once aldehydes have been formed from peroxides, for instance, further breakdown of peroxides is accelerated. From a secondary alcohol a ketone may be derived. But ketones are not easily oxidized.

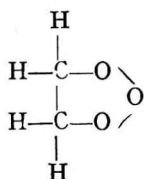


From a tertiary alcohol, an ozonide may be derived.



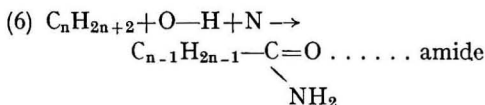
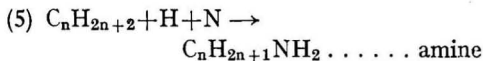
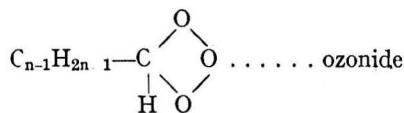
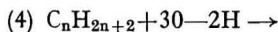
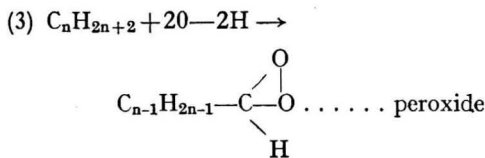
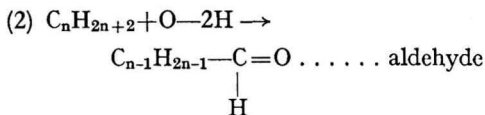
Higher alcohols occur with ozonide isomers. The secondary, whence ketones may be derived, are difficult to oxidize. From the tertiary alcohols ozonides are formed with difficulty. That means that with isomers the oxidation is far more difficult than with normal compounds. If peroxides and/or ozonides occur with the isomers, these compounds will be more stable being built more centrally than those of the normal molecule.

The formation of an ozonide from CH_4 needs the coupling of two CH_4 molecules.



That is to say, CH_4 and C_2H_6 will have the same ozonide. The formation of this ozonide from CH_4 is less probable than that from C_2H_6 , since in the former it requires the meeting of two molecules of the hydrocarbon, apart from the attachment of the oxygen atoms. Since there is a chance for the ozonides to detonate, under identical conditions CH_4 will show less tendency to detonate than C_2H_6 .

The Formations



The above formations indicate the possible reactions. They cover the least amounts of oxygen and nitrogen.

These products may occur in the intake in gaseous form or they may occur in the cylinder during the compression stroke.

It will be seen that these reactions take in dissociation products. They are called chain-absorbing or chain-forming reactions. They may occur together in a definite way and where the pre-reactions involve condensation, it indicates that during compression also the pre-reactions will be accelerated according to the principle of Van't Hoff-Le Chatelier. So, if the pressure is increased, that reaction which affects the least number of molecules will start.

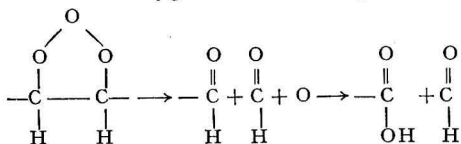
It is not difficult to see that with an increasing number of carbon molecules in the hydrocarbon, the quantities of oxygen or nitrogen required decrease for all reactions. That is to say, a higher hydrocarbon the easier it is to form these products. As these products may initiate the final detonation, it also means that the inclination to detonate increases with the higher degree of a hydrocarbon.

Breakdown of Products — During the compression stroke in the cylinder, and also during the compression of the end-gas by the burning gases, pressure and temperature are constantly increasing, and this means increase in translatory rotation and vibratory energy (in equal parts).

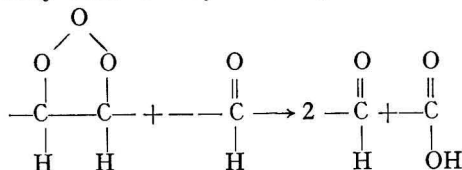
The increase in translatory energy means faster-moving molecules and more collisions, more chance of the formation of new compounds but also of a breakdown. The increased rotational energy means first of all more compact molecules (fields of force) until a critical point is passed when the molecule breaks down⁹. The increase in

vibrational energy means a continuous loosening and fastening of the bonds, and this causes the molecules to become increasingly unstable.

The demolition of the products considered above can be envisaged chemically in the following way: an ozonide releases an oxygen atom and then breaks down into two aldehydes, whilst one of the aldehydes absorbs the oxygen atom, forming an acid:

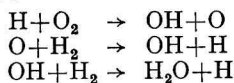


An ozonide together with an aldehyde may form two aldehydes together with an acid. The H atom next to the carbonyl group is easily oxidized into an aldehyde. The aldehyde has strong reducing tendencies and the oxygen of an unstable oxygen compound, even as an ozonide, is, therefore, easily withdrawn by an aldehyde.



In the two last reactions one sees that during the demolition of the products formed in the first stage of the pre-reactions the number of molecules again increases. Apart from the piston compression and/or that of the burnt gases in a further stage, this also causes a rise in pressure.

This might happen locally, influenced by certain catalysers, and cause a local rise in pressure and total combustion¹¹. Since H and O atoms are present in the breakdown stage, a branched-chain reaction may start as follows:



In the first reaction of this link, for every disappearing active part two new ones reappear. This constitutes the branching of the chain reaction. This is to say, even if only a few active parts occur at first, the number rapidly increases, as does the speed of the reaction with the branched-chain reactions simultaneously.

If the supply of the active oxygen parts, for instance, increases rapidly, the branched-chain reaction may attain such a speed that it leads to total combustion and detonation. As an example we may draw attention to the positive influence of ozone¹⁰. Here we have at work two opposite influences — the increasing pressure would produce reactions that decrease the number of molecules, while the increasing temperature breaks down the ozonides and induces reactions that increase again the number of molecules.

It is to be expected that aldehydes, peroxides and ozonides, in this order, will, in increasing measure, enhance the chance of detonation. In the same order they are less stable with reference to the oxygen, and will speed up the chain reactions. Similarly, if the formation of the oxygen compounds mentioned can be retarded directly or indirectly, the chance of detonation will decrease. This can be done, for instance, by choosing suitable intake conditions ("mild engine") and also by lowering all temperatures when using a rich mixture. With such a mixture, combustion is not complete. In addition to CO₂, CO is also formed. As CO has an appreciably lower combustion value than CO₂, all combustion temperatures will go down.

Another effect of an increase in mixture strength is that there are increased quantities of hydrocarbons for the same quantity of combustion air. This means that under the same conditions the temperature will be lower — in reality the same number of dissociation products of the combustion have to be used to produce an increased number of hydrocarbons. Then there will be retardation in forming, for instance, higher oxygen compounds as there will be less oxygen present per hydrocarbon molecule. This also increases the chance of attaining a critical period in the pre-reaction stage.

Lower combustion temperatures may also be obtained by the injection of water.

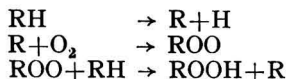
Quite recently Wheeler, Downs and Walsh¹¹ announced their finding that of all the intermediate products of combustion, the peroxides are the only ones which possess an appreciable pro-knock effect. Wheeler further found that a rise in pre-flame peroxide takes place prior to top dead centre, followed by a much higher peak value just before the flame. With the increase of compression ratio, there is an earlier rise in

pre-flame peroxide concentrations, and much higher peak value. The curves of peroxide concentrations vs. crank angle are two stage in nature. For compression ratios from 7 to 9.9, they find peroxide concentrations of about $15-30 \times 10^{-6}$. The effect of tetra-ethyl-lead is to lower the concentration of the peroxides. The effect confirms the two-stage nature of the curves¹².

Aldehyde curves run parallel to the peroxide curves. The maxima are about 30 times greater. Tetra-ethyl-lead uniformly decreases the aldehyde concentrations at all points.

As regards the nature of the aldehydes produced, repeated tests with dimedone gave only the formaldehyde derivatives¹³.

Walsh¹² gives the following chain reaction for peroxide formation :



and so on.

These degradations are complicated by the decomposition of alkyl and peroxide radicals, the former to olefines.

Thermal explosion may occur at high temperature because of greatly increased radical decomposition. Decomposition of ROOH, but no ROO, is assumed to be catalysed by aldehydes, thus explaining the apparently greater stability of ROO.

Olefines — The double bond, which means two pairs of electrons, is slightly open as the bonds will try to assume their natural direction (tetrahedron). They may be opened still further with no great effort to form linkages with dissociated oxygen, with nitrogen and also with hydrogen, that is to say, the olefines first saturate the double bond.

Two atoms of oxygen, hydrogen and/or nitrogen are taken in when the double linkage opens symmetrically as compared with the corresponding paraffins, the olefines absorb more dissociated products (oxygen, hydrogen, and/or nitrogen) at less cost of energy of the molecule, because no other atoms (hydrogen) have to be removed. Karrer¹⁰ has drawn attention to the easy addition of ozone to olefines. The ozonide formed is "explosive", forming 2 aldehydes or ketones together with water.

The number of atoms that may start a branched-chain reaction is thus lessened considerably.

The effect of "detonation inhibitors" — iron carbonyl and nickel carbonyl — may be explained by assuming that they accelerate the binding of hydrogen atoms by unsaturated hydrocarbons.

Generally it would appear that, as regards detonation, olefines will be better than paraffins.

Di-olefines — What applies to the olefines, applies in an increasing measure to the di-olefines, due to their triple bond, as compared with the double bond of the former.

Naphthenes — As observed earlier they are chemically very stable; in fact, more stable than the paraffins.

Aromatics — The basic aromatic, benzene, may take on hydrogen and change to cyclohexane, thus reducing the number of hydrogen atoms that may start a branched-chain reaction. If, however, ozone has been formed during the pre-reaction stage, an ozonide of benzene may appear. This ozonide is very unstable and has an "explosive" character¹⁰. It might induce combustion of the end-gas.

The behaviour of benzene depends a great deal on conditions of reaction. It may have a high anti-knock value or a low anti-knock value. Its sensitivity is high. Toluene and other derivatives favour the formation of oxygen and other compounds more readily than does benzene. They approach paraffins in their behaviour with increase in the number and length of branches. The anti-knock behaviour also varies accordingly.

Heron and Beatty¹⁴ draw attention to the fact that the aromatics have a higher latent heat of evaporation than the paraffins.

In the intake, the incubation stage of the aromatics will proceed slower than that of the paraffins. In general, there will be more delay. Thus aromatics might benefit in this way more or less, and have better anti-knock properties than paraffins. Leary, Taylor and Jovellanos¹⁵, however, think that the excellent anti-detonation properties of benzene appear to be due to the slowness of the pressure rise in the combustion of the end-gas, rather than to an exceptionally long delay period. These two views, apparently in contradiction, are reconcilable in view of the fact that the former speaks of the delay period in the intake, while the latter considers the subsequent delay period during

compression (due to piston, or moving flame front).

Conclusions

The distinction made between physical and chemical detonation is justified by the possibility of dealing with all detonative phenomena from one point of view. Miller's work strengthens this belief.

Thus fundamental concepts of mechanics, physics and chemistry may be combined with experimental evidence on detonation to further elucidate the nature of detonation. Good agreement can be shown to exist between theory and experiment.

In some respects theory lags behind experiment. Jost's theory on the effect of lead, if elaborated, might close the gap to a certain extent. The same may be said of further research in the borderland of chemical and physical detonation. Here reference has been made especially to the explosive reactions of ozonides. These reactions may start from physical detonation.

Generally speaking, mild pre-reaction may initiate chemical detonation, and severe pre-reactions may cause physical detonation.

To reach the critical stage of physical detonation, physical processes of flow, mixing and diffusion must be applied to ensure the necessary formation of an explosive kernel in which ozonides have been formed. This also shows how the two phenomena may overlap. In recent literature a survey is given of Muffing's theory on explosive reactions¹⁶. Elsewhere Karpov¹⁷ has made combustion calculations, where the pre-

reaction stage had to be taken into consideration in order to explain the thermal ignition process.

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Engineering Exhibition 1951

THE CENTRAL BOARD OF IRRIGATION, SIMLA, has issued the prospectus of the Indian International Engineering Exhibition which will open in New Delhi in January 1951. The Exhibition is being held during the meetings of four international organizations also to be held in New Delhi. The Organizations are : the World Power Conference, the Inter-

national Commission on Large Dams, the International Commission on Irrigation and Canals and the International Association of Hydraulic Structures. Working and still models depicting the latest developments in the science and techniques covered by the above international organizations will be on show during the exhibition which will last for a month.

Microwave Spectroscopy & Its Applications

A. PANDE*

All India Radio, Lucknow, India

MICROWAVE spectroscopy is the cross-road of infra-red spectroscopists, nuclear physicists and electronics engineers. It is also one of the few cases where development of technique in electronics have opened a new field in pure physics. It is similar to optical spectroscopy in principle but differs widely in technique, range of electromagnetic spectrum, resolving power, etc. In optical spectra, especially in line spectra, electronic structure of the atom is studied whereas microwave spectroscopy is used to investigate the molecules in their vibrational and rotational states as is the case in the band spectroscopy. Microwave spectroscopy can be used to study heavy molecules which cannot be studied very effectively with optical techniques and it is very effective for investigating effects of the nucleus upon the molecular spectra. Its resolving power is about 1,00,000 times that of the best infra-red grating spectrometer due to which it promises to be one of the most powerful methods yet developed for the study of nuclear spins and nuclear quadruple moments. Microwave spectroscopy region can be specified as the region between the long wavelengths of infra-red and the longest wavelengths of microwaves where cavity oscillators and resonators and wave-guide components become effective, i.e. 3 mm. to about 20 cm., though millimeter range is of greater interest.

Experimental Techniques & Equipment

In microwave spectroscopy there is no instrument for dispersion. Instead an electronically controlled oscillator is used as a source of radiation which makes possible the use of electrically tuned detector. The frequency of oscillator is measured very accurately up to seven figures by the beat method. The indicator is an oscillograph whose sweep is synchronized with the frequency modulation of the source, thus

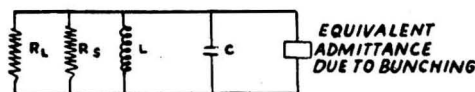
making a visual study of the absorption pattern under study. The whole set up usually consists of:

- (a) A source of radiation, i.e. a frequency controlled oscillator.
- (b) Absorption cell with vacuum arrangements and modulating electrodes.
- (c) Wave-guide and associated components.
- (d) Detecting systems.

A block diagram of the arrangement is given as under (Fig. 1).

In literature^{1,2} can be found detailed description of microwave theory, components and techniques and, therefore, only brief description of the various equipment and circuits used will be discussed here.

(a) *Source of Radiation* — The most commonly used oscillator is the reflex klystron which operates on the principle of velocity modulation and "electronic bunching". An equivalent circuit diagram illustrating the principles of reflex klystron is given as below:



EQUIVALENT CIRCUIT OF A REFLEX KLYSTRON.

The cavity resonator and its load are represented by the parallel resistance capacitance circuit. The losses are represented by R_s and the loading by R_e . The bunching action produces a radio frequency current which depends on the beam current and the bunching parameter. The equivalent capacitance C represents the capacity of the resonator gap, the value of which can be estimated with fair degree of accuracy.

Reflex klystron has only one cavity resonator which acts both as input and output circuit. Also the reflector electrode is operated at a potential negative with respect to cathode. Electrons which have passed

* Now at the National Physical Laboratory, New Delhi.

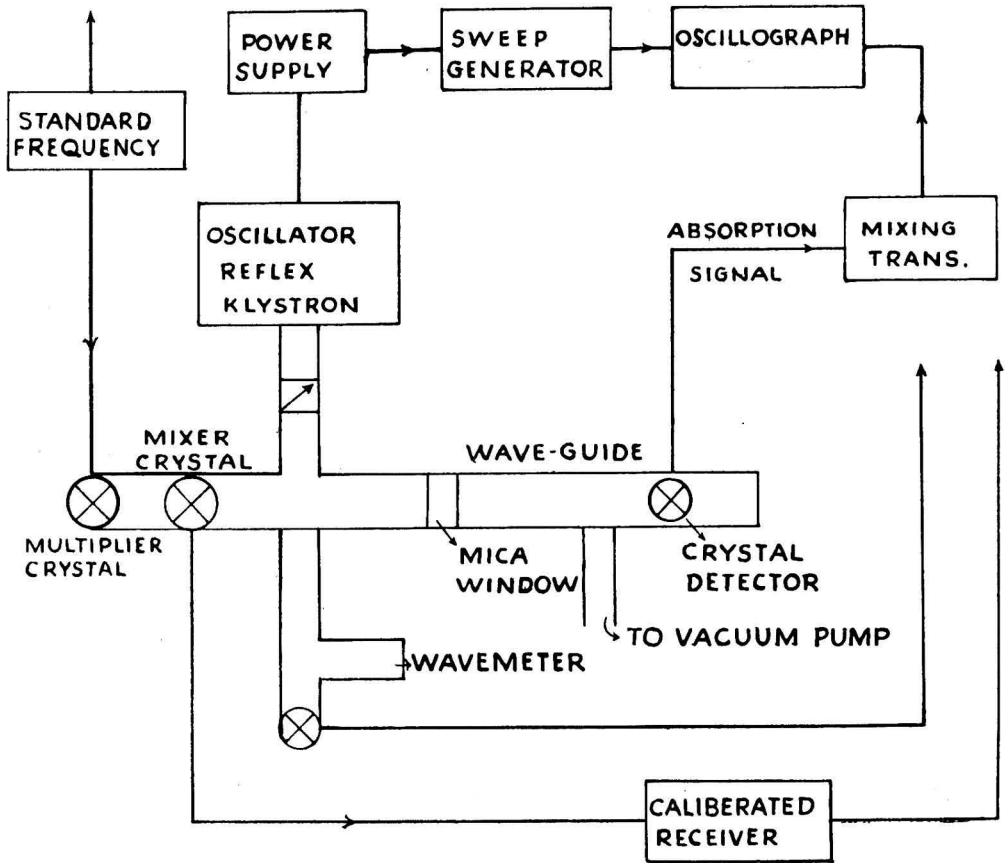


FIG. 1 — BLOCK DIAGRAM OF A MICROWAVE SPECTROMETER.

through the r.f. gap are, therefore, subjected to the retarding electric field which reverses their motion before they reach the reflector and returns them through r.f. gap in such a phase as to give up energy to the oscillating field at the cavity. Frequency characteristic of the reflex oscillator is given as

$$\tan \theta = 2Q \cdot \frac{(f_0 - f)}{f_0} \dots \dots \dots (1)$$

where θ is the transit angle
 Q includes the effect of loading
 f = frequency of oscillation
 f_0 = its value at the centre of the node.

Another great advantage in using a reflex klystron is the ease with which simple types of modulation, e.g. square wave amplitude modulation or saw-tooth frequency modulation, can be obtained at any desired rate

and over any frequency range from a few kilocycles to 40 megacycles by simply modulating the reflector voltage of the klystron. Such oscillators can be obtained to cover a range of 5 mm. to 20 cm. Small output, varying from 150 microwatts to 20 milliwatts, from these oscillators is an advantage rather than a disadvantage, as powers of higher order produce "saturation effect" in the absorbing gases as well as detecting crystals. Hence magnetron oscillators are not used now though the first microwave absorption work was done using split magnetron by Cleton and Williams³. For regions below 5 mm., crystal harmonic generators have been used at Duke University by Gordy⁴ and his group. Electronically controlled power supplies are used in all these circuits.

Frequency of the oscillator is stabilized by the Pound⁵ method with an external cavity resonator at high Q and some other means for frequency conversion. A block diagram is given below.

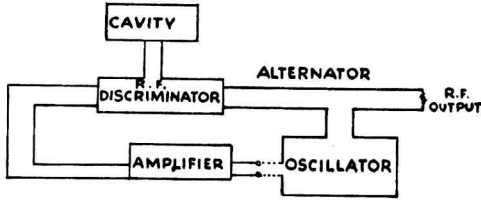


DIAGRAM OF A FREQUENCY STABILIZATION.

A standard signal applied to this micro-wave circuit produces an output voltage which, with the resonant frequency of the cavity, produces an "error voltage" which is then amplified and super-imposed upon the supply voltage of an element of the oscillator, the potential of which controls the oscillator frequency. For the stabilization circuit, it is essential that the output voltage from the amplifier have the inverse phase to that impressed upon the control element of the oscillator.

Absorption Cell—Absorption cells are most frequently made from sections of rectangular wave-guides with thin mica window to seal the cells. Some workers have used S-band guide (3" x 1½") very effectively for absorption cells in the region from 3 to 5 mm. The absorption of gas in wave-guide cell is greater than that in a free space cell of equal length by a factor

$$\frac{\lambda_g}{\lambda}$$

where λ is the wavelength in vacuum and λ_g the wavelength in the guide given as

$$\lambda_g = \frac{\lambda}{\left[1 - \left(\frac{\lambda}{\lambda_c}\right)^2\right]^{\frac{1}{2}}} \dots\dots\dots (2)$$

λ_c is the cut off wavelength and is given as

$$\lambda_c = \frac{2}{\sqrt{\left(\frac{m}{a}\right)^2 + \left(\frac{n}{b}\right)^2}} \dots\dots\dots (3)$$

where a and b are dimensions of the guide and m and n are the integers denoting the mode. Hershberger⁶ uses a similar rectangular guide 300 ft. section made vacuum tight with mica windows.

The attenuation for the dominant TE₀₁ mode of a rectangular silver guide in terms of its smaller and larger dimensions "a" and "b" and wavelength λ is given as

$$\alpha_c = 0.142b^{-3} \left[\left(\frac{b}{2a}\right) \left(\frac{2b}{\lambda}\right)^3 + 1 \right] \times \left[\left(\frac{2b}{\lambda^3}\right) - \left(\frac{2b}{\lambda}\right)^{-\frac{1}{2}} \right] \text{ db. per meter (4)}$$

Auxiliary components include a standing wave detector, tuners to eliminate a standing wave, pressure gauges, a liner amplifier and an output voltmeter. Resonant cavities are also used and effective absorbing path in a resonant cavity is of the order of

$$\frac{\lambda^2}{\pi\lambda_g} Q_c$$

These cavities are specially suited for studies of Zeeman effect because the magnetic field can be applied easily.

Detecting Devices—Before describing a few detecting devices, it is worth while to discuss the minimum detectable absorption. According to Hershberger⁷ minimum detectable power is given as

$$\Delta P = \left[\frac{4K T.F. \Delta f}{P_0/e} \right] \alpha_c \dots\dots\dots (5)$$

- where α_c = attenuation coefficient ;
 K = Boltzman constant ;
 T = temperature in absolute ;
 F = noise figure of the receiver ;
 Δf = band width of the receiver ;
 and
 P / e = available power at the receiver.

Hershberger has taken only Johnson noise into consideration and has ignored other types of noise considerations, e.g. shot noise and partition noise. Gordy has suggested that in order to obtain the optimum cell length and the minimum detectable absorption coefficient of gas with a super-heterodyne receiver, one must maximize the detected power (ΔP^1) rather than the absorbed power P. He gives

$$\Delta P^1 = \left\{ P_i \exp. \left(-\frac{\alpha_c \rho}{4} \right) \left(\alpha_g \cdot \rho \right)^2 \right\} \dots\dots\dots (6)$$

- where α_g = absorption coefficient of the gas ;
 α_c = attenuation coefficient of the cell ; and
 α = length of the cell.

With values of $\alpha_c = 5 \times 10^{-4}$ nepers/cm.
 $P_i = 10^{-5}$ watts
 $f = 300$ c.p.s.
 $F = 40$

Value of length $l = 40$ meters.

In order to improve the sensitivity of the detector Hughes and Wilson⁸ have employed a stark modulation method in a relatively short section with very good results. In this method the position of the absorption line is itself varied sinusoidally by means of low radio frequency. In the case of ammonia (3, 3) line, the shift is approximately 15 mc./s. for a field of 1,400 volts per cm. and the shift follows the square law. Expression for the wave-field component H is given as

$$H = H_0 (1 + k \cos. 2 \phi t) \cos. wt \dots\dots\dots (7)$$

where k is the modulation factor.

In addition to increasing sensitivity by avoiding low frequency noise, this ingenious method also completely eliminates the troublesome problem of spurious signals arising from mismatch in the microwave line. Hence the problem of measuring the absorption coefficient of gas involves simply the simultaneous measurement of the average received power and modulation factor k . A similar system has been used by Gordy and Kessler⁹. In their method the klystron frequency is swept 20 mc./s. at 60 cycles rate and simultaneously swept perhaps 40 kc./s. at 100 c./s. rate, a 100 kc./s. receiver follows the microwave detector. In this case the problem of detecting and measuring an absorption coefficient involves in noting the change in power as a function of frequency fed through a microwave transmission path containing gas as the spectrum line which acts as a discriminator and converts frequency modulation into intensity modulation.

Super-heterodyne is found not to be superior in sensitivity to simple Video receiver operating with a square law detector, because in microwave spectroscopy small changes of power are to be detected. Hence a rather more sensitive microwave spectrograph can be made by employing the frequency sweep method with a crystal Video receiver. Very narrow band audio amplifiers are used. Sometimes a double-crystal balanced Video receiver is used for detection.

The most direct method of determining the overall sensitivity of a microwave spectroscope is the measurement of the signal to

noise ratio obtainable for weak spectral lines of known absorption coefficient. Thermocouples and bolometers are also used sometimes, but they are not so sensitive for detecting sub-microvolt power in the microwave region.

Frequency Measurements — One of the advantages of microwave spectroscopy over optical spectroscopy is the degree of accuracy attainable in the measurement of the frequency of the oscillator. For regions above 1 cm. wavelength, a number of cavity wave-meters have been designed with accuracies of about ± 3 mc./s. The co-axial type is self-calibrating and can be made to tune over a very wide frequency range. The frequencies of different modes of a cylindrical cavity are given by the formula :

$$f = \frac{c}{2} \left[\left(\frac{2 \times l_m}{\pi D} \right)^2 + \left(\frac{n}{L} \right)^2 \right]^{\frac{1}{2}} \dots\dots(8)$$

where c is the velocity of light, D and L are diameter and length of the cylinder.

For more accurate measurements, a standard signal of 10 mc./s. is taken from W.W.V.; this 10 mc./s. multiplied to 270 mc./s. and then to 2,970 mc./s., which is again multiplied by crystals to the desired 5 mm. range. The crystal is also modulated by 270 mc./s. power and by some of 90 mc./s. power which is made to bypass the klystron, the side band frequencies thus produced provide markers which cover the microwave region at 90 mc./s. intervals. Using a communication receiver and applying frequency sweep method, it is easy by mixing the two signals to super-impose the "pip" from the receiver upon the absorption line appearing on the oscilloscope. Cavity wave-meters are used to check the markers. Workable energy can be obtained at least to 20th harmonic of the klystron frequency of 2,970 mc./s.; hence measurements can be extended up to 5 mm. or so. Using this method spectral lines can be measured to an accuracy of about 50 kc./s. at 25,000 mc./s.

The experimental difficulties in obtaining atomic spectra in microwave regions are great, hence only two investigations have been reported so far. One is the observation of hyperfine structure of cesium by Roberts, Beers and Hill¹⁰ and the other the famous hydrogen experiment of Lamb and Rutherford¹¹. Roberts, Beers and Hill observed directly the hyperfine structure in the 3 cm. wave-region of cesium vapour. They were

able to measure 14 to 15 components of cesium transition by introducing a microgram of cesium vapour at 4×10^{-2} mm. pressure into the frequency controlling resonant cavity of a stabilized oscillator near 9,192.6 mc./s. and observing the 30 c.p.s. component of the audio signal detected by an f.m. receiver when a weak external magnetic field in the cavity is modulated at 30 c.p.s.

Lamb and Rutherford's experiment is of great importance as it gave the first experimental disagreement with Dirac's quantum electrodynamic theory according to which the levels of the same "n" and "j" should be degenerate. They used the atomic or molecular beam technique developed by Rabi, Zacharia Milliman and Kusch at Columbia University.

When the atoms, in meta-stable state $^2S_{1/2}$, are passed through weak microwave radiation of the correct gyrofrequency which is about 1000 mc./s., some are raised to $^2P_{3/2}$ state, from which they decay to $^1S_{1/2}$ state and at this frequency a dip is observed. Bethe¹² has shown that this anomaly may be due to the interaction of the electron with the electromagnetic radiation field and he predicted a shift of 1,050 mc./s. This prediction has actually been experimentally verified by Fowles¹³ in the optical spectra of the ionized helium.

Absorption Spectra of Gases & Vapours

One of the most thoroughly investigated spectra is the inversion spectrum of ammonia. The absorption of 1.25 cm. waves by ammonia gas has been known for sometimes and is so intense that a plane 1.25 cm. wave loses half its power on traversing a 3' layer of ammonia at N.T.P. Besides ammonia a number of gases were found by Hershberger and others⁶ to be strong absorbers of microwaves at room temperature. They also found that microwave energy was converted into heat and sound directly. Also Van Vleck¹⁴ pointed out that the absorption of 6 mm. waves by oxygen and 1.3 cm. waves by water vapour is so large as to limit severely the use of these wavelengths in communication and radar.

Ammonia absorbs microwaves due to its capacity for turning inside out. The two equilibrium positions of the nitrogen on

either side of the plane defined by 3 hydrogens are separated by a potential barrier which nitrogen "tunnels through". The inversion spectrum consists of a number of different lines arising from molecules in different rotational states.

The potential barrier involved in the so-called "tunnel" effect is so low that the energy levels of the molecules are measurably split. Pressure broadening, i.e. the broadening of spectral line that arises from collisions between molecules necessitates working at very low pressure if we wish to observe a spectral line at low radio frequency. All of the molecules which are found to absorb possess either an electric or a magnetic dipole moment which serves to couple them to the applied electromagnetic field.

Sheng, Barker and Dennison¹⁵ gave the theoretical explanation and formula for the fine structure of inversion spectrum of ammonia, which is given below :

$$\nu = \nu_0 - 0.0011 J(J+1) + 0.0016 K^2 \dots (9)$$

Howe was first to detect this fine structure and Good¹⁶ and Bleaney and Penrose¹⁷ observed as many as 30 lines in the inversion spectrum of ammonia. Good found that at pressure of 10^{-2} mm. Hg and below, a definite hyperfine structure appears due to interaction of the nuclear quadruple moment of N¹⁴ with molecular field. Also a definite frequency shift of the various lines was observed when a d.c. field was applied to the absorbing gas. For greater accuracy, a fourth power formula has been employed by Simmons and Gordy¹⁸ to calculate the fine structure of NH₃ inversion spectrum. The Van Vleck, Weisskopf formula¹⁹, when modified for ammonia inversion spectrum, becomes as :

$$\alpha = 18\nu^2 \left[\frac{K^2(2J+1)}{J(J+1)} \right] g \exp. [-0.0475J(J+1) + 0.0174K^2] \dots (10)$$

Simmons and Gordy attribute the hyperfine structure also due to interaction of the nuclear magnetic moment of nitrogen with the magnetic field arising from molecular rotation in addition to the nuclear quadruple interaction with molecular field.

Rotational Spectra

Iodine monochloride is the only diatomic molecule whose pure rotational spectrum

has been investigated in the microwave region by Weidner²⁰. Rotational frequencies of diatomic molecules are given by the following approximate formula :

$$\nu = 2B (J + 1) - 4D (J + 1)^3 \dots\dots (11)$$

where $J = 0, 1, 2, 3, \dots\dots$

$$B = \frac{h}{8\pi^2 c I}$$

$$D = \frac{4B^3}{\omega^2}$$

"I" is the moment of inertia and ω is the fundamental vibration frequency. Rotational spectra of diatomic molecules have been used to determine the mass ratios of different isotopes. Frequency difference in the corresponding isotopic frequencies for the ground vibrational state²² is :

$$\begin{aligned} \Delta\nu &= \nu_r - \nu_r^i \\ &= 2B_e (J + 1) (1 - \rho^2) - \alpha_e \\ &\quad (1 - \rho^3) (J + 1) - 4D \\ &\quad (1 - \rho^4) (J + 1)^3 \dots\dots\dots (12) \end{aligned}$$

Where B_e , e , D refer to the isotope corresponding to ν_r and $\rho = \left(\frac{\mu}{\mu_i}\right)^{\frac{1}{2}}$ mass ratio which can be determined. Townes, Merritt, Wright²¹ have evaluated the mass ratio of Cl^{35}/Cl^{37} which agrees well with Aston's values.

Similar method is used for determining the mass ratio in optical band spectroscopy but as the resolving power in microwave spectroscopy is so much higher, much more precise values are obtained. As a result of the interaction between vibration and rotation a doubling of rotational lines, termed ρ type doubling, occurs for degenerate bending. The separation of the doubled line is given as

$$\Delta\nu = 2q (J + 1) \dots\dots\dots (13)$$

where q is a small constant.

The microwave rotation spectra of a number of symmetric top molecules have been investigated theoretically and experimentally. In Herzberg notation, rotational frequencies are given by Slawasky and Dennison equation as

$$\nu = 2B (J + 1) - 4D_J (J + 1)^3 - 2D (J + 1) K^2 \dots\dots\dots (14)$$

where $B = \frac{h}{8\pi^2 I_{BC}}$

By employing molecules with different isotopes, complete determination of molecular structures have been made in certain

cases like methyl-halides which was not possible either by electron diffraction methods of infra-red spectroscopy. This was made possible by using Johnson and Dennison²² equation given below :

$$\Sigma \Delta\nu_i = \left\{ \left[\frac{6}{I_A} - \frac{7}{I_B} \right] \frac{h}{8\pi^2 c} \right\} \dots\dots (15)$$

It is easy to determine "I_A" when accurate values of "I_B" had already been determined from microwave rotation spectra. It was found that C-H distances are not the same in these molecules but vary in a uniform manner from F to I. Similar works are being carried on with other molecules.

So far only two complete structural determinations of asymmetric top molecules have been determined by microwave spectroscopy. These are SO₂ and HNCS. In the case of asymmetric top molecules all the moments of inertia can be determined by pure rotation spectra alone, the structure of triatomic molecules can be determined without using different isotopic combinations. Rotational energies by asymmetric top molecules have been calculated by King, Hainer and Cross²³ by the following simplified equation :

$$\begin{aligned} \frac{E_r}{h\nu} &= \frac{1}{2} (A + C) J (J + 1) \\ &\quad + \frac{1}{2} (A - C) E_r \dots\dots\dots (16) \end{aligned}$$

where $A = \frac{h}{8\pi^2 c I_A}$

$$B = \frac{h}{8\pi^2 c I}$$

$$C = \frac{h}{8\pi^2 c I_C}$$

I_A, I_B, I_C are the three moments of inertia.

These authors have also constructed tables which allow easy calculations of the line intensities of asymmetric rotators. No free internal rotational spectra have so far been reported in the microwave region.

Stark Effect

The use of Stark modulation for detecting weak lines has been given earlier. Since the last 20 years the theory of Stark effect in rotational spectra has been developed by Debye but its application was made possible with the coming of microwave spectroscopy. The Stark effect in several linear molecules has now been investigated and dipole moments determined by using the equation

for the second order rotational energy perturbation for the ground vibrational state which is given as :

$$W_{Jm} = \left[\frac{4\pi^2\mu E^2 I}{h^2} \right] \left[\frac{J(J+1) - 3M^2}{J(J+1)(2J-1)(2J+3)} \right] \dots (17)$$

where E = strength of the applied electronic field ;
 μ = molecular dipole moment ;
 and
 $M = J, J-1, 0 \dots$

The treatment of the symmetric top molecule is similar but has first order effects as well. Stark effect in symmetric top molecules has not been studied so far.

Zeeman Effect

Zeeman effect in microwave spectroscopy is quite analogous to the nuclear Zeeman effect in atomic spectra and the equation giving magnetic interaction term as found by Jen²⁴ is similar to the formula of Back and Goudsmit for Zeeman effects in the hyperfine structure of the atoms. The interaction term is :

$$\Delta W = M \cdot \mu_o H (\alpha_J g_{mol} + \alpha_I g_N) \dots (18)$$

where $\alpha_J = \frac{F(F+1) + J(J+1) - I(I+1)}{2F(F+1)}$

$$\alpha_I = \frac{F(F+1) + I(I+1) - J(J+1)}{2F(F+1)}$$

$M = F, F-1, F-2, \dots, F$;

μ_o = nuclear magnetron ;

g_{mol} = g factor for the molecule along J ;

g_N = g factor of the nucleus considered ;
 and

H = external mag. field.

With a field of 1,670 gauss Jen detected a broadening of the rotational lines in SO₂ but perhaps the magnetic field was not enough to split it. When $g_{mol} = 0$, interesting splitting terms are obtained in the case of CH₃Cl²⁵ as observed by Jen²⁴. This method can be used for determining unknown magnetic moments, NH₃, CH₃ Cl, SO₂, are the only molecules investigated so far, but Zeeman effect in microwave spectroscopy promises to be a subject of important research.

Applications

In nuclear physics microwave spectroscopy has been employed for precise determination

of nuclear couplings, dipole moments, quadruple moments and nuclear spins. The values of nuclear spin, as determined by the microwave spectroscopy and atomic beam method, are more reliable than those determined by the hyperfine structure of Zeeman effect. Also this method may prove extremely valuable for determination of the spins of radioactive or rare nuclei.

Microwaves have also been used for the study of the dipole moments of several liquids including nitrobenzene, acetone, methyl cyanide, etc., by measuring the frequency of maximum absorption or relaxation time. In addition to the possibilities of microwave resonant frequencies in solids, there exists the chance of resonant absorption in certain liquids or solutions arising from vibrations in molecular chains held together by weak intermolecular bonds such as hydrogen bonds. Also the magnetic resonance absorption of solids in the microwave region provides a new and powerful means of measuring closely spaced energy levels in paramagnetic substances and studying the structures of certain crystals. Microwave spectroscopy is being used for the study of molecular structure and isotopic mass ratio determinations with much greater accuracy than is possible by the method of infra-red spectroscopy.

One of the most important applications of microwave spectroscopy is in the field of "frequency stabilization" at microwave frequencies. Molecular spectral lines possess two properties which make them particularly suitable for use in frequency control work and also as frequency standards. The first is the constancy of the mid-frequency of the absorption line like the constancy of spectral lines in optical spectroscopy and the second is the very high Q value of the ammonia line which is of the order of 1,00,000 at a pressure of 10⁻⁵ mm. and ordinary temperatures, i.e. 300°T. One of the common methods of stabilization consists of employing interaction at the microwave frequency between the high Q absorbing gas and klystron oscillator similar to the interaction between a quartz crystal and its associated amplifier. The arrangement is a feed-back or servo-mechanism arrangement in which an "error voltage" is fed back to the oscillator under control to maintain equality between the ammonia absorption frequency and the klystron frequency. This stable

ammonia absorption line is being used to control a crystal clock at the National Bureau of Standards. The ammonia gas molecules, which vibrate at 24,000 mc./s., regulate a standard quartz crystal clock. The difference or error vibrations of ammonia and quartz is electronically developed and keeps the quartz clock locked to the ammonia frequency.

Also another "atomic clock" is under construction in which the vibrations of cesium atoms are being used in an atomic beam technique. This cesium clock has the highest potential accuracy of time, i.e. 21 sec. in one hundred years! Such a clock will have important applications in microwave communication, molecular structure and astronomy.

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Bibliography of Scientific Publications, South Asia

THE BIBLIOGRAPHY PUBLISHED BY THE UNESCO Science Co-operation Office for South Asia, Delhi, is the first compilation of the kind ever attempted. The inaugural number recently published covers the period January to June 1949, and relates to scientific literature published in India, Burma and Ceylon. A separate series will be issued for other South Asian countries.

All branches of sciences, pure and applied, are included. Special attention is given to papers reporting original research, though review articles and new ideas are also included. The first few pages give a list of

periodicals complete with subscription rates and addresses.

The UNESCO Science Co-operation Office maintains notes of addresses of authors and editors and of libraries where particular articles can be found. Scientists are invited to use the facilities to obtain reprints, microfilm or photostat copies. Short résumés of articles listed in the bibliography can be had from the Office on request.

This compilation listing the titles of scientific contributions of particular region fulfils a pressing need and facilitates exchange of scientific information and ideas between working scientists.

The Colouring of Foodstuffs

THE use of synthetic colouring matters in the preparation of manufactured and preserved foodstuffs has now become a widespread and established practice. It is no longer regarded with suspicion but, on the contrary, is recognized as being an integral part of the processes of manufacture. Natural colours frequently fade or leach out in processing and artificial colours must be added in order either to restore the original shade or to impart the popular tint. The public has come to associate definite shades with certain foods and any departure from a generally accepted shade is construed as an alteration in the product itself, with the ensuing possibility of complaints to the manufacturer and an immediate falling-off in his sales. The public demands that its butter shall be yellow; that its strawberry jam shall be red; that its jellies shall be suggestive of the fruits from which they have been made, and so on. Foodstuffs must appeal to the eye before being accepted by the palate.

We must not lose sight of this psychological aspect. The satisfactory physical appearance of many cooked and preserved foods can only be restored by the addition of artificial colouring matters and it becomes necessary for the food manufacturer to acquaint himself with the elementary principles controlling the use of such agents and with their properties and characteristics. The correct colouring of foodstuffs is a science as well as an art. At one time it was treated with unnecessary reticence by both the colour manufacturer and the colour user and in consequence the interests of each and of the public suffered. It must be emphasized here that the complications arising from the use of colouring matters are so many, the reactions of the foodstuffs so varied and the applications so diverse that it is only by collaboration of these two that a satisfactory product can be presented for the approval of the public.

As the foodstuff manufacturer in most cases has to rely on the dye-maker for purity and quality of colours, ultimately, therefore, it is the responsibility of the dye-maker to ensure that the products used for colouring

foods are harmless. There are many problems in connection with the performance of a colouring matter in food products; high fastness to heat is demanded by bakers and boiled sweet-makers; in many foods the colour must be unaffected by the preservative present; in fruit-canning the hue must be unaffected by fruit acids; colours for mineral waters must possess a fairly high light fastness. The solubility characteristics also are important as undissolved specks of colour would mar the appearance of clear boiled sweets or delicately coloured custard powders or other corn-flour preparations.

A malpractice which has been prevalent for many years and which still exists to some extent is the dilution of colours with inert materials in order to reduce their apparent toxicity. The dilution of colours to specific strengths is recognized as being necessary to ensure standardization and is not normally regarded as being to the disadvantage of the consumer. This practice becomes an abuse, however, when the colouring matter is excessively diluted with the sole object of bringing its content of toxic materials down to an apparently low level, thus enabling the supplier to give a guarantee of suitability for foodstuff purposes. Such a practice is strongly to be condemned as the consumer must then use a correspondingly larger amount of colour to tint the foodstuff to the required shade and the toxic content of the finished goods is the same as would be obtained if the undiluted, impure dyestuff were used. Not only should colouring matters contain minimum amounts of toxic materials, but this minimum must be attained by careful manufacture and not by a process of dilution.

The food laws put the responsibility for the colours used largely on the foodstuff manufacturer. According to the Food and Drugs Act passed in the U.K. in 1938 "no person shall add . . . any substance to any food so as to render the food injurious to health".

No comprehensive legislation exists in India on the use of colouring matter in foods. As early as 1930, the Government of the United Provinces had made certain provisions

for the use of artificial colours in foods in the rules framed under their Food Adulteration Act. Two lists of colouring matters were given, one of colours which could be used and the second of those which were prohibited (APPENDIX A). These rules were recommended for acceptance by the Ministry of Health to the various Provincial Governments in 1947. Subsequently in August 1948, the Ministry of Health, Government of India, brought to the notice of the Provincial Governments the rules adopted under the New Zealand Food & Drugs Regulations, 1946. The list (APPENDIX B) included names of 28 synthetic colours which could be incorporated in foods.

A joint meeting of the Food Standards Committee of the Technical Panel in the Ministry of Food and the Central Committee for Food Standards in the Ministry of Health was convened in March 1950 under the Chairmanship of Dr. K. Mitra, Adviser in Nutrition, Directorate General of Health Services, Delhi, to find out as to what could be done to check the increasing use of colours in foodstuffs — colours which were reported to be injurious to health.

The recommendations made by the Committee prohibit the use of inorganic colouring matters and pigments in all foods and permit a restricted use of certain synthetic and organic dyes, a tentative list (APPENDIX C) of which has been drawn up. The Committee, however, agreed that the incorporation of such vegetable colouring matters or pigments occurring in edible fruits or vegetables should be permitted. Plans for the setting up of an active organization to implement and enforce these recommendations are under consideration.

Reputable dyestuff manufacturers have on the market brands of colours for foodstuffs (e.g. the Edicol range of *Imperial Chemical Industries Ltd.*) which are sold with a guarantee according to the particular food laws to which they conform.

APPENDIX A

EXTRACTS FROM UNITED PROVINCES PUBLIC HEALTH (PREVENTION, ETC., IN FOOD) RULES, 1930

Colouring matters which may not be added to articles of food:

1. *Metallic Colouring Matters* — Compounds of any of the following metals:

antimony, cadmium, copper, lead, arsenic, chromium, mercury, zinc.

2. *Vegetable Colouring Matter* — Gamboge.

3. *Coal-tar Colours.*

No. in colour index of Society of Dyers & Colourists, 1924	Name	Synonyms
7	Picric acid	Carbazotic acid
8	Victoria yellow	Saffron substitutes; dinitrocregol
9	Manchester yellow	Napthol yellow ; Martius yellow
12	Aurantia	Imperial yellow
724	Aurine	Resolic acid ; yellow coralline

Colouring matters which may be added to articles of food :

Red shades	80 Ponceau 3 R
	187 Amaranth
	773 Erythrosine
Orange shade	150 Orange I
Yellow shades	10 Napthol Yellow S
	640 Tartazine
	22 Yellow AB
	61 Yellow OB
Green shades	666 Guinea Green B
	670 Light Green SF. Yellowish Fast Green F.CF
	P. hydroxy derivative of the sodium salt of alpha-zurine F.G. CI 671
Blue shade	1180 Indigo Disulpho Acid

The numbers preceding the names refer to the numbers of the colours as listed in the Colour Index published in 1924 by the Society of Dyers and Colourists of Great Britain.

APPENDIX B

EXTRACTS FROM NEW ZEALAND FOOD & DRUGS REGULATIONS, 1946.

The following shall be deemed to be harmless colouring substances satisfying the regulations :

- Caramel (prepared solely from sugar)
- Cochineal
- Chlorophyll
- Saffron
- Every harmless vegetable colour

THE COLOURING OF FOODSTUFFS

(f) The following synthetic colouring substances, namely :

Red Shades —

- 79 Ponceau 2 R
- 80 Ponceau 3 R
- Ponceau SX
- 179 Carmoisine, Cardinal 3 B
- 184 Amaranth, Brilliant Bordeaux B
- 185 Brilliant Scarlet 4 R, Brilliant Ponceau 5 R
- 225 Chlorazol Pink Y, Direct Thio Red
- 749 Rhodamine B
- 773 Erythrosine
- 448 Benzopurpurine 4 B for colouring sausage skins only

Orange Shades —

- 26 Croceine Orange
- 150 Orange I
- Orange SS

Yellow Shades —

- 10 Naphthol Yellow S
- 16 Acid Yellow G. Kond
- 22 Yellow AB
- 61 Yellow OB
- 640 Tartar Yellow, Tartrazine, Tartratol Yellow L
- Sunset Yellow FCF

Green Shades —

- 666 Acid Green G, Guinea Green B
- 670 Light Green SF Yellowish
- Fast Green FCF

Blue Shades —

- 671 Brilliant Blue FCF (Sodium salt)
- 1180 Indigo Carmine

Violet Shade —

- 698 Acid Violet 5 BN

Brown Shades —

- Certicol Brown RS, Edicol Chocolate NS
- Golden Brown 34832, Edicol Brown OHS

Black Shade —

- 865 Nigrosine

The numbers in the margin opposite the names of the synthetic colouring substances under (f) of this regulation refer to the numbers allotted in Rowe's Colour Index (edited by F. M. Rowe and published in 1924 by the Society of Dyers and Colourists, England).

APPENDIX C

- Brilliant Blue FCF
- Indigotine
- Guinea Green B
- Light Green SF Yellowish
- Fast Green FCF
- Orange I
- Orange SS
- Ponceau 3R
- Amaranth
- Erythrosine
- Ponceau SX
- Oil Red XO
- Naphthol Yellow S
- Naphthol Yellow S — Potassium Salt
- Yellow AB
- Yellow OB
- Tartrazine
- Sunset Yellow FCF

The dyes from the above list when used in foodstuffs should be absolutely pure and free from any harmful impurities. The maximum limit permissible for each of the dyes should be fixed at one grain for each pound of food.

REVIEWS

Physical Principles of Oil Production, by Morris Muskat (McGraw-Hill Book Co. Inc.), International Series in Pure & Applied Physics, 1949, pp. xv + 922. Price not indicated.

OIL IS PRODUCED FROM WELLS DRILLED INTO underground oil reservoirs, which are formed in porous sedimentary rock formations like sandstones, limestones or dolomites, which have at one time taken in and trapped the oil in their pores. The oil is initially at a higher temperature and pressure, depending on the depth and the other environmental factors of the reservoir. Oil reservoirs are sealed in and limited by the surrounding impermeable rocks, or even by partially permeable rocks containing water, the water-oil interface in the capillary pore spaces acting as the sealing blanket. Oil reservoirs are usually closed by local deformations of strata such as a simple folding into closed anticlines or domes.

As an essential preliminary, suitable tests on core samples of the oil-bearing rocks should be conducted to accurately estimate such factors as their porosity, permeability and fluid content. Test data so obtained should be statistically studied and correctly interpreted. The hydrodynamics of the fluid movements within the rock strata and into the oil wells should be thoroughly understood. The bearing of possible anisotropy in reservoir permeability on the problem of fluid flow should also be investigated. Moreover, the fluid itself can be a mixture of many ingredients both in the liquid and gaseous phases. The effects of that heterogeneity in the composition of the flowing liquid should also be assessed. The initial chapters of the book are devoted to a general study of such topics. The treatment favours the usual text-book method; and each problem is treated proposition by proposition, with assumptions of idealized initial conditions of progressively increasing complexities. But, as the author himself says in the preface, the book is not meant to be "a text-book of a particular

grade or level, and directed to a particular class of reader".

The general performance of an oil-producing reservoir is determined by the nature of the energy available for oil expulsion, and the manner in which that energy is made to operate. The oil can, of course, be moved into the well-bore and out of it by virtue of the initial oil pressure itself; but that can be effective only during the starting phases of production, and will become of minor importance at later stages. The major potential source of oil expulsion energy is the pressure exerted by the gas dissolved in the oil itself or present in the free state in the gas-cap above. Oil withdrawal is then said to be through "solution-gas drive" or "gas-cap-expansion drive" mechanisms. Oil is also expelled from a reservoir through displacement, by water entering the oil pay. The withdrawal is then said to be through a "water-drive" mechanism which may be of two kinds — a "side-water drive" brought about by a lateral edge-water encroachment, or "bottom-water drive" effected by an uprise of water from the bottom layers. Between the strict gas-drive and the complete water-drive mechanisms lie a series of intermediate gas-water combination drives. The general mechanics of these different drives are developed and studied in the book with a view to obtaining an insight into the methods of production control, so as to conserve the energy of expulsion, and to realize a significant retardation in the pressure decline and thereby to assure the maximum possible percentage of oil recovery from the reservoir.

On a conservative estimate, the quantity of oil still left in the reservoir, after it has been abandoned as uneconomical, works out to be about equal to the quantity of oil withdrawn previously. The methods of oil production thus appear at first to be inherently wasteful and inefficient. Intelligent primary control minimizes the residual oil left behind; but an appreciable quantity of oil is always available for further recovery. Also there had been a large number of wells

worked and abandoned before such control methods were sufficiently well understood. Secondary recovery methods are adopted in such cases for reclaiming the residual oil. They consist in the injection of gas or water through an interlaced network distribution of injection and production well-bores, or in a general "water-flooding". The mechanics of such secondary recovery systems are well discussed in the book. Experimental technique for studying visually, through scale models, the behaviour of different systems of well distribution like line drives, five spot and hexagonal networks are described in the book with appropriate illustrations, though one would like greater stress placed on the essential three-dimensional nature of the problem.

One chapter is devoted to "condensate reservoirs" wherein, when the pressure is released in the well-bore, a condensate or distillate is initially recovered. But, side by side with this recovery is a retrograde condensation of the saturated vapour back into the reservoir itself. In such cases an injection of the produced dry gas, freed of liquefiable contents, a pressure maintenance operation known as "cycling" is resorted to, partly as a method of prevention of this retrograde condensation and partly as a method of ultimate recovery of the condensed oil itself. Geometrical patterns of borewells to facilitate such "cycling" processes and the experimental studies, through models, of their characteristics and behaviour are described at length in the book.

The book thus covers practically all problems of oil-reservoir engineering. The treatment is essentially theoretical and based on idealized and simplified assumptions. Though, here and there, practical data from a few important oil wells are given and discussed, no sustained attempt is made to effectively connect up the theoretical study with the practical aspects of oil production. The author himself says that his aim is only to give the underlying physics and mechanics of oil-reservoir problems. All the significant and relevant matter have been brought together and presented so as to afford a complete picture of the scientific basis of oil-well technology. The book is a valuable exposition of an important subject which is still in a stage of active development. In addition to those who are actively interested in this particular

subject, this book will be found useful to all specializing in the study of underground water movement and general physics of fluid flow through granular beds. The printing and get-up of the book are up to the well-known standards of McGraw-Hill publications.

T. N. SESHADRI

Bibliography of Research on Heavy Hydrogen Compounds (National Nuclear Energy Series), compiled by Alic H. Kimball & edited by Harold C. Urey & Isidor Krishenbaum (McGraw-Hill Book Co. Inc., New York, Toronto, London), 1949, 1st Edition, pp. 350. Price \$ 3.26.

DEUTERIUM, THE STABLE HEAVY ISOTOPE of hydrogen, was discovered in the year 1931 and since its discovery it has been the object of intensive research study because of its extensive use as a tracer in chemical and biological reactions, in testing theories where mass effects were involved and its application as a moderator in atomic piles. As a result of these research studies conducted in every country, a very large number of articles dealing with the physical and chemical properties of deuterium and its compounds have been published in various scientific periodicals.

The volume under review is a compilation of references to the existing literature on deuterium and its compounds. The references are classified and arranged subject-wise for ready reference. This work should, therefore, be welcomed by any research worker who wishes to survey the bibliography of the past achievements in this field.

SWAMI JNANANANDA

The Indian Cotton Textile Industry (1949 Annual), by M. P. Gandhi (Gandhi & Co., Bombay), 1949, pp. liv + 174 + xxxi. Price Rs. 6.

THE WHOLE WORK IS CHARACTERIZED BY clear-thinking and scientific analysis. The book contains a great deal of statistical data and is a storehouse of information on cotton textile industry in India in its various aspects, viz. production, price, finance, distribution, import, export, labour and textile machinery manufacturing. It is a valuable addition to cotton literature and

will be found useful alike by students, business men, financiers and government officials.

It is, however, suggested that for easy reference a book of this description may include a few maps to indicate at a glance the location of mills, areas of raw cotton production, etc., and other important facts.

K.N.

"Analar" Standards for Laboratory Chemicals (formulated and issued jointly by the British Drug Houses Ltd. and Hopkin & Williams Ltd., London), 4th Edition, 1949, pp. 302 + xviii.

THE ANALYTICAL CHEMIST HAS BEEN LOOKING forward keenly from 1944 to a new edition of "Analar" standards extending the specifications for more reagents required for his work. The 4th edition of the "Analar" standards under review has amply justified the expectations in that specifications for 58 new "Analar" chemicals have been included. Of the new additions, the most welcome inclusions are some of the organic reagents used in micro-organic analysis and some important reagents for the identification of organic compounds. The only omission in the present edition from the previous one is ferric chloride and that is perfectly justifiable.

Besides extending its usefulness on the quantitative side, the present edition has got some special features which are very valuable for the analyst. The following are the two notable points: (i) the technique of electrolytic deposition for the assay of certain metallic salts and for the removal of principal metals from such salts in order to enable easier determination of the other impurities and (ii) the modern Karl Fischer technique for the estimation of water in organic liquids. Another feature of the publication is the substitution of newer methods of assay leading to greater sensitivity and greater accuracy wherever appropriate, thereby bringing down the value of maximum permissible impurities, thus contributing to the preparation of better quality chemicals.

The printing and get-up of the book leaves nothing to be desired, except for a slight mistake on p. 227 under item 8 of "NaHSO" for "NaHSO₄". Under assay of cupric oxide on p. 292 the method suggested requires a little modification as it is not usual to perform iodimetric titration of copper in the presence

of hydrochloric acid or any mineral acid. Usually the titration is conducted in acetic acid medium and in fact this has been suggested by the same publication for the other copper salts. An alternative procedure in this case might be to dissolve the oxide in dilute phosphoric acid and then proceed as usual for iodimetric titration.

It appears to the reviewer that the publication will have wider significance if a small paragraph is devoted at the beginning of each chemical as to the reasons that have prompted the publishers to choose the list of reagents for a selected chemical before giving the methods of estimation.

S.A.C.

Modern Plastics, by Harry Barron (Chapman & Hall Ltd., London), 2nd Edition, pp. xi + 779 with 272 illustrations. Price 50s. net.

ONE, WHO HAS TO LABOUR THROUGH THIS big volume, cannot help closing the book with a feeling of disappointment, wondering as to what extent it has helped in clarifying ideas or giving him a real insight into the intricacies of the subject. The type of details which are recorded in this book impress upon the reader its encyclopaedic scope. One notices, however, that the book suffers from a lack of purposiveness which has inevitably resulted in a rather incohesive and jumbled presentation of the numerous findings of modern research. The uniform emphasis, if any, laid on all the facts and details presented in this book makes it a task for the reader to pick out the more important facts, fundamental principles and processes from the relatively less important ones. It may not be an exaggeration to state that precision and clarity of thought are conspicuous by their rarity.

The many tables and figures which are inserted in the body of the subject-matter do not fully serve their purpose. Although these are numbered they are not, as a rule, referred to in the subject-matter, nor are the salient points intended to be brought out by them duly stressed. The historical background of the industry which has been attempted is poorly delineated. The impact of the extraordinary advances made in other fields of invention has given a forceful incentive and drive to the development of chemical processes and technological methods which

are responsible for the remarkable achievements in this field. The pictures of the motor car and the aeroplane showing the various gadgets which are made from plastics are but a poor, apologetic and old-fashioned device for illustrating the immense and far-reaching applications and potentialities of plastics made possible by novel processes and ingenious techniques.

The author's attempt at covering *all* about modern plastics in this volume evidently makes precise or clear-cut presentation of important topics difficult. The book conveys to the reader an impression of a conglomerate of loose and approximate statements and disjointed paragraphs. It contains sentences which are structurally unsound, involved and clumsy. Here are a few examples:

(a) *Page 76* — "American production of alcohol for 1944 was more than 631,000,000 gallons. Produced from molasses, grain, petroleum, natural gas, sulphite liquors and wood waste."

(b) *Page 78* — "Considering both this process from the engineering point of view on a large scale, it is clear that the vaporising of alcohol is not a difficult operation, nor is the passage over a catalyst."

(c) *Page 83* — "The low cost of ingredients have made this plastic of interest for industrial applications involving large quantities of material such as building units, furniture and wall panelling, etc."

(d) *Page 80* — "The cheapest wood waste is saw-dust, comprising about one-third of the total mill waste. It is already in the physical form suitable for hydrolysis without further processing. Therefore it has been found that the minimum mill that could operate on saw-dust alone must be three times the size of one turning all its waste into chemicals. In order to support a hydrolysis plant using the American process, this mill must cut about 150,000,000 feet a year.

"Seventy tons of wood per day, or a daily cut of 70,000 feet of lumber would be required to support a Schöller plant capable of producing 3,600 gallons of alcohol per day, at a yield of 50 gallons of alcohol per ton of wood. Such a mill would cut 20,000,000 feet annually. An alcohol plant of the same capacity using the American process at a yield of 20 gallons per ton of wood would require 180 tons of wood per day, the waste from a daily

cut of 180,000, or roughly, an annual cut of 54,000,000 feet."

The last two paragraphs could, doubtless, be expressed more precisely in a shorter span and with better effect. Practically all the sections dealt with in this book disclose that the general approach is devoid of simplicity, clear thinking and directness of presentation. The versatile uses of plastics as building materials, which the author has chosen to indicate in a pictorial manner in Fig. 13 is, to say the least, queer and unnecessarily complicating.

It is not easy to grasp what the author attempts to impress when one reads sentences such as the following:

Page 34 — "If U.S. plastics consumption in 1939 is taken as 100, in 1947 cellulosic moulding materials are at 750.

Phenolic Resins	300
Urea Resins	300
Melamine	300
Polystyrene	12,500
Vinyl Resins	15,000"

It will serve no useful purpose to mention in this review the numerous chapter headings dealt with in the book, since the author's aim seems to be to describe and discuss *all* and sundry about plastics, without any attempt at judicious discrimination. It will, therefore, suffice here to give the titles of the main parts of the book, viz. Part I: Introductory. Part II: Thermosetting Resins and Their Plastics. Part III: Cellulose Plastics. Part IV: Vinyl Plastics. Part V: Other Leading Plastics. Part VI: Some Important Aspects of Plastics. In the last part are included chapters on High-frequency Heating for Plastics, Analytical Aspects and Physical Testing of Plastics.

This is a book which may be kept on the shelves for occasional reference to get some abstruse detail, which a researcher, a factory foreman or a prospective industrialist will like to get. It is not likely to be of much use to the post-graduate research worker for reason of its inadequate treatment of the basic and fundamental, theoretical and thought-provoking aspects; nor can it be safely recommended as a text-book study for the under-graduate. It will serve to capture the interest of the layman who yearns to have a comprehensive view of what these "mysterious" substances, in reality, are, with their oft-mentioned ubiquitous applications.

A.J.R.

Vacuum Equipment and Techniques, National Nuclear Energy Series — Manhattan Project Tech. Section, edited by A. Guthrie & R. K. Wakerling (McGraw-Hill Book Co. Inc., New York, Toronto, London), 1949, 1st Edition, pp. 264. Price 2-5s.

HIGH VACUA ARE ESSENTIAL REQUISITES FOR pure research in many branches of modern physics and chemical engineering. Without high vacua certain types of production engineering, such as manufacture of incandescent lamps, radio valves, X-ray tubes, etc., would not have been possible. The development of atomic power engineering has been rendered possible only by the application of high vacua. Thus modern physical and chemical laboratories, production engineering plants and atomic power engineering works are constantly confronted with problems of the technique of production of high vacua, measurement of pressures of high vacua and the construction of vacuum equipment for various purposes. A study of these problems, therefore, has become an essential necessity, both for the research scientist and the production engineer.

There are already excellent treatises, monographs and articles dealing with principles, production, measurement and techniques of high vacua. The work under review, *Vacuum Equipment and Techniques*, does not propose to compete or supersede the existing literature on the subject lately published. It, however, proposes to concern itself with the study and development of high vacuum equipment and large-scale vacuum installations developed by the personnel of the University of California Radiation Laboratory in contract with Manhattan Project. As such the work should be of immense use in helping to devise some of the best methods of vacuum techniques and to design efficient large-scale vacuum installations and equipment.

The work is divided into five chapters. The first chapter deals with the basic theory of vacuum practice, the second with the elements of vacuum systems, the third with vacuum gauges, the fourth with vacuum materials in equipment, and the fifth with leak-detection instruments and techniques. The work also includes a number of appendices at the end of the volume giving very important data concerning some of the properties of gases and vapours, mechanical

pump oils, diffusion pump oils, cold traps, effectiveness of drying agents, vacuum greases and cements, solder brazing alloys and properties of solids and liquids frequently used in vacuum practice. Liberal reference has been made to the available literature throughout the book.

The book will be found very useful by research workers and may be recommended as a valuable addition to libraries.

SWAMI JNANANANDA

The Presentation of Technical Information, by Reginald O. Kapp (Constable & Co. Ltd., London ; Orient Longmans Ltd., Bombay, Calcutta, Madras), 1948, pp. 147. Price 6s.

THE NEED FOR AN IMPROVEMENT IN THE standard of presentation of technical information is all the more evident in India where no systematic training in this field is given to post-graduate students in any of our universities. The reviewer, who is familiar with material received for publication in a technical journal, has often felt that his job would have been far easier and less exacting had the contributors a basic grounding as to how a technical paper or report has to be written. Unfortunately, none of our universities provide for such a course for post-graduate students in science or applied science subjects and the result is that often the contributions received are not what they ought to be.

This book is the outcome of a series of lectures delivered by the author at the University College, London, where a course on presentation of technical informations was started in the Faculty of Engineering. The course, it is pointed out, was to be at the post-graduate stage, to cover a whole session and to include both lectures and exercises. This book should be of special interest to our university authorities and induce them to consider the question of introducing similar courses in the universities. Such a step would immensely benefit our young scientists and technicians.

The subject-matter is classified in 15 short chapters covering practically all aspects. Written in simple style and illustrated with a number of appropriate citations from the works of well-known authors and scientists, the book makes a very good reading.

It is difficult to pick out any one chapter as more important than the rest. All are, but there are some which provide new ideas. The first chapter, which the author terms as "Functional English", deserves a little more attention as it provides the basis for the whole subject, viz. the presentation of technical information not only in English language but, in a broader sense, in any other language. What applies to English applies equally well to other languages too. Now that the question of presenting technical information in vernaculars is becoming imperative, all those interested in this field would benefit a great deal by a study of the basic principles discussed in this chapter.

The rules to be followed in writing technical books, preparation of matter for technical reports and papers and how to deliver talks on technical subjects are the other topics dealt with in the book. The hints provided are very useful. A series of "don'ts" are given in chapters II and VIII, the latter also containing some important "do's".

In the opinion of the reviewer, the book is an useful and important contribution to scientific literature and should find a place in all libraries, even sectional libraries in all research laboratories and technical institutions. As a matter of fact, this volume should find a place in the shelf of all research workers.

On the last point the reviewer would like to make a suggestion. Such a useful publication is rather too highly priced to be within the reach of every research worker. The only way to make it accessible to one and all is to bring out a cheaper edition on the lines of Pelican books. That would be doing a real service in the cause of science.

A.K.

Bengal in Maps, by Dr. S. P. Chatterjee (Orient Longmans Ltd.), 1949, pp. 105. Price Rs. 18.

THIS IS NOT THE CONVENTIONAL COLLECTION of locational maps; it is a praiseworthy attempt to give in the form of maps a fairly complete picture of the socio-economic pattern of present-day Bengal. The author in his preface has traced the circumstances which led to the compilation of these maps.

The atlas falls into three broad sections. The first part deals with the geographical

location of Bengal in relation to the rest of India and Pakistan, the physical features and the climatic conditions throughout the year. In this section there are two maps — provocative of interest. One is entitled Greater Bengal, and shows the whole region where Bengali is spoken. As the author points out, "Bengal may be politically divided today; but the community of language will continue to be the most powerful unifying factor of Greater Bengal". This map and the one on transport reveal clearly the gross injustice of the division of Bengal. It may not be possible to agree with the author's view that if these maps had been in print before the final award of Sir Cyril Radcliffe, there might have been a more reasonable division of Bengal, for after all that award was largely the result of political considerations. The other map, of interest in an entirely different field, is the one dealing with the rivers of the eighteenth century. It shows clearly how the lapse of two centuries has changed the course of several of these rivers and consequently the layout of the land.

The second section deals with the people and their occupations. The population changes from 1901 to 1941 are shown in a series of four maps. The other maps deal with the distribution of the two main communities, the Hindus and the Muslims, and the minorities both in East and West Bengal. We may, however, point out one minor defect in these excellent maps. The "dot" system of representation makes these maps look almost alike; perhaps if there had been much bigger maps, representation by "dots" might have been useful.

The last section dealing with the distribution of the economic resources of Bengal is perhaps the most interesting part. In a series of maps the author shows the production and yield of various agricultural crops. The brief statements about the various crops accompanying the maps are admirably concise and to the point. It might have been better if the author had indicated the sources of his information for the details of yield and production. This section would have been more complete if the author had added a series of map dealing fully with mineral and animal resources of Bengal. The Raniganj coal-mining area is, however, dealt with in one map. The pattern of industry in Bengal is also represented in a few maps.

On the whole, the author and his associates deserve every praise for having brought out this excellent atlas. They have shown that geography is not merely a listing of physical features and climatic conditions of a land, but that it deals with the close and variegated relation between man and the country he lives in. We hope that the brilliant example set by Dr. Chatterjee may be followed by others and, ere long, we will have similar atlases for other parts of the country.

Elements of Internal Combustion Turbine Theory, by H. T. Adams (Macmillan & Co. Ltd., London), 1949, pp. 178 + xii. Price 16s.

THE AUTHOR OF THIS BOOK WAS, FOR SOME TIME, Director of Studies to Power Jets Ltd. under the Ministry of Aircraft Production. He is now Senior Lecturer in Mechanical Engineering at Canterbury University College, and is known as the author of *The Aircraft Gas-Turbine*, issued by His Majesty's Stationery Office.

The present volume is most useful and timely. Inside the jacket we are told that "this book presents the theory and some of the methods used in the design of the internal combustion turbine during recent years. It is intended for advanced students and for engineers. A reasonable mathematical background and some knowledge of aerodynamics is assumed, but some fundamentals are given in the first chapter, and worked examples are included where these will help towards a clearer understanding of the principles in the text."

This is a true and concise description. Terseness and precision indeed are distinguishing features of the author's style. He understands compression in language almost as well as in mechanical compressors, with the result that, although the book is not lengthy, it is a veritable *multum in parvo*. The book is divided into 9 chapters as follows: (1) Introduction, (2) Thermodynamic Considerations, (3) The Centrifugal Compressor, (4) The Axial Flow Compressor, (5) The Turbine, (6) Stress Calculations, (7) Vibration, Critical Speeds and Endloads, (8) Overall Performance Correction and Estimation, (9) Heat Exchange and Intercooling.

Broadly speaking, the internal combustion turbine is a comparatively late arrival in the engineering world. We do not possess the store of practical experience available, for instance, to the designer of steam turbines. Consequently, the numerous technical problems which arise in the design of internal combustion turbines have largely to be solved from first principles, and particularly must the science of fluid dynamics be our guide, philosopher and friend. The author is well aware of this, and in the elucidation of the scientific principles involved, he chooses actual and pertinent data for working out examples to elucidate the theory. Particularly commendable are his treatment of centrifugal and axial flow compressors, and of the turbine itself and the stresses set up. The only disappointment is that the author has seen fit to exclude anything beyond a casual note on combustion on the grounds that "the subject of combustion is a study in itself". True, but this can be said of all subjects.

However, it is impossible to expect everything from a first edition. There is no doubt that young engineers and others concerned in the design and performance of internal combustion turbines will be helped considerably by this book which is excellently, yet modestly, printed and produced.

J.W.W.

PUBLICATIONS RECEIVED

Progressive Mathematics, by P. CLYNE, Chapman & Hall Ltd., London, 15s.

Modern Workshop Technology, by H. WRIGHT, Cleaver-Hume Press Ltd., London, 32s.

Data and Circuits of Receiver and Amplifier Valves (Series of Books on Electronic Valves, Book II), N. V. Philips Gloeilampenfabrieken (Philips Industries), Eindhoven, Netherlands.

Data and Circuits of Receiver and Amplifier Valves (Series of Books on Electronic Valves, Book III), N. V. Philips Gloeilampenfabrieken (Philips Industries), Eindhoven, Netherlands.

Organic Chemistry (4th English Edition), by PAUL KARRER, Cleaver-Hume Press Ltd., London, 57s. 6d.

NOTES & NEWS

Viridin

A NEW ANTIBIOTIC, VIRIDIN, IS produced by the common soil fungus, *Trichoderma viride* (*Nature*, 1950, 165, 528). Viridin is extracted from the culture filtrate with chloroform. After evaporation of the extract under reduced pressure and treatment of the oily residue with ice-cold methanol, a slightly coloured crystalline material is isolated. Ketonic and non-ketonic forms are separated by reacting with Girard's reagent. The ketonic part is further purified by chromatography using a strongly acidic alumina column to separate α and β viridins. The α isomer is isolated from the benzene-ether eluate. After recrystallization from methanol, acetone and finally from dilute acetic acid, α -viridin is obtained as fine colourless needles, decomp. 208°-17°C. Analyses and molecular weight determinations reveal that the hitherto accepted molecular formula of the compound should be replaced by $C_{19}H_{18}O_6$.

β -viridin isolated from the chloroform eluates and recrystallized from methanol forms fine needles.

Viridin is not anti-bacterial but is highly anti-fungal. Germination of the spores of *Botrytis allii* is prevented by a concentration as low as 0.019 p.p.m. of α -viridin or 0.156 p.p.m. β -viridin. Among synthetic fungicides, only the organo-mercurials are of the same order of activity.

Quantitative Study of Viruses

ESTIMATION OF THE POTENCY OF virus preparations is an important factor in the progress of biological investigations. A detailed study of the validity of various means of assessing the relative potencies of the virus of foot-and-mouth disease has been made in a Report No. 8 of the Agricultural Research Council, London (*H.M.S.O.*, price 2s. 6d.).

The usual method of virus potency by inoculating guinea pigs is inadequate because bovine strains require serial adaptation before they are fully effective upon the smaller host. Inoculation of each dilution to a separate

bovine host is prohibitively costly. To combine effectiveness with reasonable cost, 4 dilutions are introduced each with 5 inoculation sites, upon the tongue of one Devon Stear 1½ to 2 years old. The results are more significant if two animals are used, that is, 10 inoculations of each dilution. The observations are of fundamental importance in the study of plant viruses also.

Estimation of Alcohols

THE METHODS USUALLY EMPLOYED for estimation of alcohols are based upon the reaction of the alcohol with acetic anhydride or acetyl chloride followed by hydrolysis of the excess reagent requiring 2 to 24 hr. for each determination. A procedure requiring only 20 min. for the determination of methanol, using acetyl chloride as the reagent, is described. The reagent which does not enter into ester-formation is hydrolysed and titrated with standard alkali (*Proc. Indian Acad. Sci.*, 1948, 58, 142).

A 20 c.c. bottle is used as the reaction vessel. A measured amount of the reagent is introduced into it by means of a syringe, through a rubber stopple in the neck of the bottle. The blank was determined as follows: About 0.3 c.c. of acetyl chloride was placed in the bottle and allowed to stand for 5 min. The bottle was half-filled with water and the contents emptied after shaking. Both the stopple and the bottle were then rinsed with acetone and dried in a current of air. 0.3 c.c. of acetyl chloride was then introduced into the bottle through the stopple by means of a syringe, and allowed to stand for 5 min. 10 c.c. of water was then injected and after shaking, 11 c.c. of air was withdrawn from the bottle. The contents were transferred to an Erlenmeyer flask, bottle was filled with water, one-half emptied into the Erlenmeyer, the bottle shaken and remaining water poured into the flask. The contents were titrated with standard alkali using phenolphthalein. An actual determination is similarly made, the only difference in the procedure

being that a weighed sample of alcohol sealed in a small thin-walled glass ampoule was placed in the bottle before the stopple was inserted and the glass bottle was broken after the addition of the acetyl chloride.

If a series of determinations is interrupted for more than a few minutes, it is necessary to pre-treat the rubber stopple again with acetyl chloride before continuing. Each day a new blank should be run.

Blast-furnace Slag Cements

THE SHORTAGE OF CEMENT DURING the war led to investigations on the possibility of increasing its production by making greater use of granulated blast-furnace slag. A survey of suitable slags for this purpose was undertaken by the Building Research Station, England (*Technical Paper No. 3*, National Building Studies, H.M.S.O., price 9d.).

The normal method of slag granulation is to prepare batches of about half a ton by slowly pouring the molten slag into a large excess of cold water. Whenever possible the granulation is carried out as the slag leaves the furnace. The slags tested have considerable hydraulic value and most of the experimental cements satisfied B.S. 146. Although haematite slags gave the best results, there is considerable overlapping with the basic and foundry-iron slags. The early strength is considerably affected by the ratio of gypsum to clinker and by impurities such as chlorides introduced in the slag. There is little change in the hydraulicity of a slag when granulated by different methods. The method of granulation did, however, make considerable differences in the weight per cu. ft., the water retained after draining and grindability.

Samples of granulated slag have been incorporated into experimental blast-furnace cements. The standard proportion employed was 65 : 35 of dry slag to clinker, gypsum being added so that the ratio of SO_3 to clinker was the same as that in Portland cement. The hydraulic value of the slags is best judged by a comparison of the 90 days strengths of the concretes made from the corresponding cements using the same clinker for activation.

These slags with a high ratio basic oxides to acidic oxides are

preferred for cement manufacture and many operators consider that there is an optimum value for the Al_2O_3 content.

Granulated slags are only feebly hydraulic when mixed with water alone; in order to develop their full strength they need to be mixed with another material usually called an activator. The activator in blast-furnace cement is the lime set free during the hydration of the Portland cement clinker. It is possible, therefore, that the hydraulicity of a slag may depend on the type of clinker used. Rapid hardening Portland cements develop the hydraulicity of the slag more than normal types.

Salt Manufacture in Philippines

PRODUCTION OF SALT IN THE Philippines employing modified solar process technique has been described (*Philippine J. of Sci.*, 1949, 78, 85).

In the local method raw sea water is evaporated in a series of fish ponds (which are used to produce salt during the dry months) to about 15°Bé . which is then dipped with buckets into crystallizing ponds where it is evaporated almost to dryness and the salt recovered. The crystallizing ponds are about 5 m. \times 7 m. equipped with board sides and tile bottoms and usually represent from $\frac{1}{10}$ to $\frac{1}{3}$ of the entire area of the salt works. Every day in the season, the ponds are filled to a depth of about 1 cm. and late in the afternoon after each day of evaporation the salt is raked into a pile on the side of the crystallization bed and the pond re-filled. This process is continued throughout the season, without any change, until the brine level builds up with a heavy brine containing a large percentage of the magnesium salts. When this happens, the salt producer will probably let the pond go for a day or more without adding fresh brine, and will then, after harvesting the salt, splash out of the pond the remainder of the brine. Owing to the high magnesium content, this method of handling produces a very inferior product. It is the common practice of the salt producers in the Manila area to fill the crystallization beds with brine of 12° to 17°Bé . which is the point at which calcium sulphate begins to precipitate, and thus practically all the calcium sul-

phate is found in the final products as an impurity. Further, it may be noted that the practice of using an unsaturated brine in the crystallizers results in a lower yield since a portion of each day is spent in evaporating the brine to a point where salt will begin to form at 25°Bé .

In the Californian process which is a great improvement over the above practice, the operations are conducted in various ponds: (1) the storage or intake pond, (2) concentrating ponds, and (3) crystallizing ponds. The salt water is pumped from the intake pond into the concentrating ponds to a depth of 25 to 50 cm. before the next supply of sea water is taken in. When the strength of the brine reaches 25°Bé , it is run into crystallizing ponds to a depth of 15 cm. or more. This depth of brine causes the deposition of large crystals which grow into massive solid crystals of pure salt. The brine evaporates until it has reached a density of 29°Bé , at which time the bittern is either discarded or evaporated in other ponds to a density of 32°Bé . This salt formed from the heavier bittern is less pure. The process is repeated until the end of the season or until a thick enough bed of salt is obtained to make harvesting economical. All the ponds have mud bottoms, and often a split is made in the harvesting, i.e. the lower 5 cm. layer is removed separately and used as a cheaper salt. The harvested salt is washed with brine and with fresh water whereby salt of over 99.5 per cent sodium chloride content is obtained.

Experiments designed to increase operational efficiency showed that it was possible to reduce the area of crystallization beds by about one-third the present space without affecting the yield. In these experiments one crystallizing pond was filled with the brine at 14°Bé . After it had evaporated to 25°Bé , it was pumped into a second pond. 56 kg. of salt was realized from the experimental plot in 2 weeks as against only 30 kg. from the control plot. Studies on conditions affecting the rate of evaporation showed that the rate is not influenced by depth of brine, when the bottom does not reflect the sun's rays. When, however, the bottom reflects the sun's rays, the evaporation proceeds more rapidly from the deeper water. No

significant differences are observed in the rates of evaporation with tile and mud bottoms.

Measurement of temperature and density variations at various points on a salt farm over a period of most active evaporation indicated that the temperature of the brine in the ditch was higher at the bottom than at the surface. Also the brine at the bottom of the ditch was of greater density than that at the surface.

New Industrial Carbons

DEVELOPMENT OF A NEW TYPE of industrial carbon and graphite for chemical engineering plant components, where inertness and high mechanical properties are essential, is reported at the *Powell Duffryn Research Laboratories*. The new materials (Delanium) are characterized by their homogeneity as opposed to other types of carbon products. In the production of these materials an extraneous binder is not employed thereby eliminating porosity.

Finely coking coal, ground with small quantities of materials inhibiting swelling, is pressed or extruded to the desired form and fired in a controlled atmosphere in a temperature cycle giving maximum shrinkage (as much as 45 per cent) without distortion. The final product has low porosity and high mechanical strength. By a suitable choice of coal, swelling inhibitors, firing atmospheres and temperatures, a variety of Delanium carbons having a wide range of mechanical, thermal, electrical and chemical properties can be produced. With appropriate heat treatment, this carbon graphitizes and products with a wide range of physical, chemical and electrical properties are obtained.

Some of the basic characteristics of the new carbon and graphite are:

PROPERTY	DELANIUM CARBON	DELANIUM GRAPHITE
Apparent density	1.55	1.70
Electrical resistance ohm/in.	.007	.0007
Thermal conductivity, B.Th.U./sq. ft./hr./°F./ft.	3	40
Transverse strength lb./sq. in.	12,000	8,000
Compressive strength lb./sq. in.	38,000	20,000
Scleroscope hardness	85	65
Coefficient of thermal expansion	$3.8 \times 10^{-4}/^\circ\text{C}$.	$3.8 \times 10^{-4}/^\circ\text{C}$.

A honeycomb block made of this graphite has been employed as a heat exchanger. This exchanger is strong and compact and is capable of conducting 500 B.Th.U./sq. ft./hr./°F. A conducting area of 50 sq. ft. is afforded by a perforated 15 in. cube. The new carbon in a special form has been successfully employed to form a new type of packing for scrubbing and similar towers, chemically resistant and with good wetting properties. Formation of tiles from the carbon to line chemical vats is another suggested use for the product (*Chem. Age*, 1950, **62**, 405).

Improved Alkali Processes

AT A RECENT MEETING OF THE American Institute of Chemical Engineers, some of the modern trends in alkali production were discussed (*Chem. Age*, 1950, **62**, 395). The early cells were designed for 1,000 to 1,500 amp. but the modern practice is to employ cells operating at higher amperage. It is more economical to use Hooker type cells at 8,000 to 10,000 amp. in plants with a capacity of 5 tons than to use smaller cells. In plants of 50 tons capacity and over, a 20,000 amp. cell is more economical than a 10,000 amp. cell.

Larger electrolytic cells are being designed to increase their efficiency in the manufacture of chlorine and caustic soda; the largest unit, a 50,000 amp. mercury cell, is being installed in France. Bigger *type S* cells have been found even in small plants to be more economical than the smaller ones.

Surplus hydrochloric acid gas is used as the raw material to yield high purity chlorine gas in a newly developed process. Cupric chloride is electrolysed to produce chlorine gas and cuprous chloride. The latter is then oxidized by hydrochloric acid and oxygen to produce more cupric chloride. This method is claimed to be superior to current methods in practice.

A recent improved process for refining caustic soda is to evaporate a 50 per cent solution nearly dry at 700°F. using Dowtherm vapour to supply heat. Contamination of the product and corrosion of equipment are avoided by purifying the lye before evaporation and using nickel equipment. The practical pos-

sibilities of this process have been demonstrated in a small plant producing continuously 60 tons per day.

Chlorination of benzene can be effected in a single step by passing chlorine, air and benzene through tubes containing newly developed catalysts which permit operations at below 500°F.

Colourless Fluorescent Dyes

COLOURLESS FLUORESCENT DYES are finding increasing application in the textile, soap, paper and laundry industries since they enhance the whiteness of materials to a considerable degree (*C.T.J.*, 1950, **126**, 827). These substances, applied after the normal bleaching process, give a remarkable luminosity which could not previously be achieved and which does not have any detrimental effect on the textile fibre. The general requirements of a satisfactory white-dyeing agent are: it should be absorbed substantively by the fibre from aqueous solution so as to give a satisfactory, i.e. greenish to blue tint; it should have good wet-fastness properties; the agent should be stable towards weak acids and alkalies and should be compatible with other finishing agents; it should be suitable for direct use in the bleaching vats and should be fast to light.

Nearly all the known technical products belong to the aromatic or heterocyclic series; many of them contain condensed ring systems. Most of the whitening agents used on cellulosic fibres are derivatives of diaminostilbenedisulphonic acid. These are also used as additions to detergents and rinsing products. Benzimidazole and glyoxaline derivatives are also advocated. Diphenylglyoxalinesulphonic acid is known especially for application to wool.

In the textile industry, whitening agents have been employed with success in the bleaching of white goods, especially of cotton, but one cannot completely dispense with chemical bleaching. If the fabric is not already bleached, i.e. if it has not a sufficient degree of whiteness, then a colourless whitening agent with blue fluorescence cannot compensate for the yellow or brown colour of the fabric. White-dyeing agents have proved economical in the treatment of multi-coloured piece-goods because their applica-

tion permits grey or scoured instead of bleached yarns to be used. In the treatment of cotton knitted goods made of grey yarns, bleaching time can be substantially shortened by performing only one bleach with chlorine, followed by white dyeing, instead of a combined chlorine-hydrogen peroxide bleach. Whitening agents have afforded new possibilities in the manufacture of viscose rayon and viscose staple fibre (spun viscose). The product can be added directly to the xanthate before the spinning process when the viscose rayon is brightened in the mass. In the dyeing of pastel colours the addition of the fluorescent dye to the exhausted dye-bath gives a better effect than when the normal dye is applied to a previously white-dyed material. White-dyeing agents brighten up white discharge patterns and ameliorate the contrast of coloured prints in calico printing by a preliminary treatment or in a separate bath following printing and rinsing.

In the paper industry, apart from treating cellulose wadding, cigarette papers and crepe papers, whitening agents can be applied to wool-mottling fibres meant for bank-note paper and paper for securities. White-dyeing agents can be used in the soap industry as additives to soap powders, being adsorbed by and bleaching the textile during washing; whitening of soap or soap powder itself to improve its appearance; as a combination of both and as additives to rinsing agents.

There have been few products suitable for application to wool. For this purpose, products should be much as could be used in neutral or slightly alkaline baths to obtain good whitening effect on raw wool by treating it either in a scouring operation or in a peroxide bleaching bath. No satisfactory white-dyeing agent exists for acetate rayon but it is possible to white-dye nylon, which is comparatively difficult to bleach to a pure white by using the typical white-dyeing agents for wool.

Carbonate-free

Sodium Hydroxide

CARBONATE-HYDROXIDE ION exchange studies on the strong base resin *Amerlite IRA-400* (manufactured by Rohm & Haas Co.) have indicated the possibility of

separating carbonate from hydroxide by chromatographic means, thus offering a simple method of preparing carbonate-free sodium hydroxide.

A little in excess of the required amount of alkali is made up from washed sodium hydroxide sticks and a column of the resin, which may be initially in the form of its chloride, is prepared. The capacity of the air-dry resin is 1.4 milliequivalents per gram, and a 50 c.c. tube two-thirds full of resin would be suitable for preparing 1 litre of 0.1 N sodium hydroxide. The alkali is passed through the resin until the effluent is chloride-free and the tube is then transferred to the neck of the stock bottle. When the rest of the alkali has passed through, the resin column is replaced by a soda-lime guard tube and the carbonate-free sodium hydroxide is then ready for standardization.

The column can be easily regenerated by the passage of hydrochloric acid (*Nature*, 1950, 165, 237).

Adulteration of Mustard Oil

PROBLEMS RELATING TO THE DETOXICATION of mustard oil contaminated with argemone oil (*Argemone mexicana*) to render it safe for human consumption and the separation of argemone seeds from a mixture of argemone and mustard seeds were discussed at a joint meeting of the Food Standards Committee of the Technical Panel (Ministry of Food) and the Central Committee for Food Standards (Ministry of Health), held in March 1950 in New Delhi.

In the investigations conducted by Dr. A. C. Roy and Dr. Surajdeo Prasad of the Industrial Research Laboratory, Patna, the alkaloid sanguinarine is precipitated from a mixture of mustard oil and argemone seed oil as phosphate by addition of phosphoric acid. Subsequently, acid-activated Fuller's earth and excess of chalk are added to neutralize the free phosphoric acid. The filter-pressed oil retains most of the original mustard oil colour and flavour and does not give a test for the presence of the argemone oil alkaloid. This treatment costs Rs. 2 per md. of oil processed.

A method by Dr. Surajdeo Prasad for a complete separation of argemone seeds from mustard seeds consists in blowing the seed

mixture through a glass tube with the help of an air current. Argemone seeds being lighter are pushed out and the rape and mustard seeds collect at the bottom of the tube.

The Committee agreed that the ferric chloride test evolved by Sarkar and modified by Sen, Sundaram and Roy be accepted as the standard method of detection of argemone oil contamination in mustard oil. It was pointed out that if the ferric chloride concentration was doubled, the test was more pronounced and even 0.1 per cent adulteration could be detected.

Control of Seed-borne Diseases

HIGH FREQUENCY DIELECTRIC heating has been employed for the control of internal seed-borne fungal diseases (*Nature*, 1950, 165, 532). Preliminary work has been carried out with the seed-borne phase of leaf stripe of oats (*Helminthosporium avenae*), as the amount of this disease can be estimated fairly rapidly by growing the oats to the seedling stage or by plating out the seeds in petri-dishes. Oats, having a 13 per cent infection with leaf stripe and contained in a cardboard box having no metal parts, were placed between the electrodes of a standard RH 23 Redifon radio frequency generator. They were heated to the required temperature and maintained at that value for 20 min. The oats were then spread out to cool and 50 seeds were sown in each of the 8 pans of sterilized sand. Subsequent growth in a greenhouse allowed the estimation of the number of plants showing the seedling phase of leaf stripe.

Heating at 60°C. for 20 min. reduced the amount of leaf stripe to about 50 per cent without greatly impairing their germination. Higher temperatures reduced the amount of disease but depressed germination also; lower temperatures had little effect. Heating at 60°C. for longer periods affected the germination more than the leaf stripe. Moisture content of grain also affects the rate of heating.

Preservation of Fish

THE OLDER METHODS OF PRESERVING fish such as heavy salting, drying and smoking, and the cur-

rent method of chilling the fish with crushed ice have various disadvantages. In contrast with these, freezing and cold storage properly applied can preserve fish of all kinds, either fresh or lightly smoked, in good condition for periods up to 9 months. The basic principles underlying modern preservation techniques are outlined in leaflet No. 11 of the D.S.I.R., London (*H.M.S.O.*, price 6d.).

Factors affecting the quality of frozen fish are: temperature and conditions of storage, rate of freezing, freshness of the raw material and conditions of thawing. *Sharp* rather than *rapid* freezing of smoke-cured fish with an overall freezing time of up to 24 hr. usually results in a good product.

For efficient freezing and cold storage it is recommended that fish should be frozen within 24 hr. of catching. The period elapsing between removal of fish from storage in ice to the commencement of freezing should be as short as possible. Glazing of fish at 35°-40°F. has been found more suitable for protecting whole fish or blocks of small fish than wrapping.

New Stainless Steel

THE NEW STAINLESS STEEL CONTAINING 17 per cent chromium, 4 per cent nickel and 4 per cent copper has excellent corrosion resistance, high strength and hardness, ease of fabrication and low temperature hardenability. It is recommended for use in the chemical, petroleum, textile and food processing industries, for springs, pressure tanks, conveyer parts, etc., and especially in the aircraft and automobile industries.

This steel is available in soft temper strip, sheet and plate and can be easily fabricated by standard procedures and then brought to full strength and hardness by heating at 1,400°F. for 30 min., air cooling and reheating to about 900°F. The hardening process eliminates cracking, distortion and decarburization encountered in conventional heat treatment (*Sci. Newsletter*, 1950, 57, 105).

New Dyeing Process

A CONTINUOUS NOVEL DYEING process employing molten metal as the medium to fix the dye has been developed (*C.T.J.*, 1950, 126, 874).

The unit comprises a continuous vat dyeing machine with an iron vessel filled with a fusible alloy (m.p. 70°C.) and a conventional soaping range running as a single unit. Cloth is passed through the dye liquor directly into the molten metal maintained at 95°-105°C. where all excess of dye liquor is squeezed out.

The molten metal serves as a source of rapidly transferable heat, excludes all air and exerts a uniform pressure on the cloth surface. The process is reported to give increased penetration and full fixation and fastness in a few seconds. Any vat colour or a combination of vat colours may be employed at any depth of shade at speeds from 30 to 120 yd. per min.

Selective Herbicide

MALEIC HYDRAZIDE HAS BEEN found to be highly selective in its action as a weed killer. It has rapid lethal effect on weed grasses—water grass, Johnson grass, barley, etc., but has no noticeable effect on cotton plants. The age of the sprayed plants, however, makes a difference; even cotton is affected if sprayed too young (*Science Newsletter*, 1950, 57, 107).

Aeroplane Load Adjuster

THE WEIGHT AND BALANCE CONTROL of transport aircraft is at present achieved by the use of mechanical computers or load and trim sheets. These have serious limitations in that they lack flexibility, are not sufficiently accurate and are time-consuming. A portable electrical instrument for rapid determination of the centre of gravity of Viking Mk. IB aircraft for any load distribution has been developed in the *Research & Development Laboratory* of the Civil Aviation Department, Government of India. The basic principle of the instrument is also applicable to other types of aircraft.

The general working arrangement is indicated in Fig. 1. Switches located in a similar configuration as the passenger seats correspond to the number of passengers in the aircraft. The on and off positions of each switch will correspond to whether the seat is occupied or not. The weight of fuel in the tanks and freight and baggage in the holds are adjustable by variable

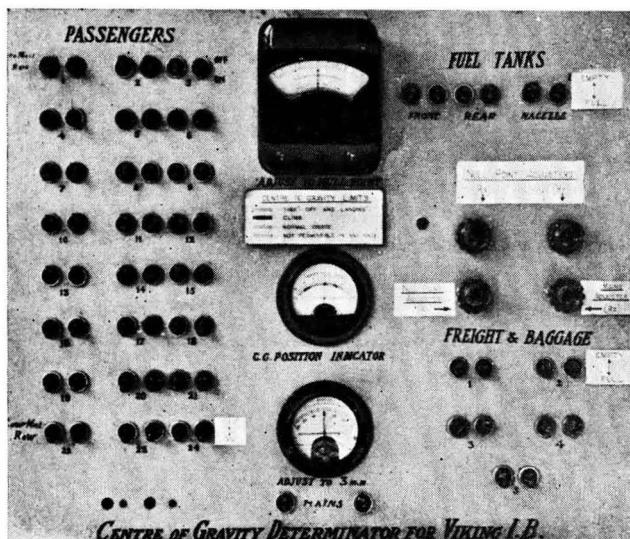


FIG. 1

control tanks suitably graduated in convenient steps. The actual position of C.G. is given on a C.G. dial indicator whose scale is graduated directly in inches aft or forward of the specified datum point.

To operate the instrument, the switches and control knobs are set to correspond to the actual load distribution and the galvanometer is adjusted to read zero. The main current is adjusted to a specified value which is an instrument constant. The actual position of C.G. is then read directly on the dial indicator. The instrument is highly accurate, adaptable and easy to operate.

Aluminium & Its Alloys

THE REVISED EDITION (DECEMBER 1949) of *The Properties of Aluminium & Its Alloys* (Information Bulletin No. 2, Aluminium Development Association, London) has been considerably enlarged and brought up to date to include specified alloy composition, types of treatment and minimum mechanical properties of aluminium and its alloys, together with other data on physical and chemical properties. The data are in accordance with the new series of BSI Specifications (B.S. 1470-7 and 1490). Characteristics affecting design such as elasticity, rigidity, shear, bearing and fatigue strengths and

strengths in compression and at elevated temperatures are enumerated. Durability and working methods in relation to properties of materials are discussed briefly with reference to other publications on the subject. There are two appendices. Appendix I gives average weights of aluminium alloys in standard forms. Appendix II relates to the new nomenclature and contains specifications which are the nearest equivalents to those in the new series of standards. This data have also been given in detail in a separate chart issued during February 1950. The bulletin is supplemented by an useful bibliography on fatigue and high temperature properties.

Standard Methods of Analysis for Textiles

THREE INDIAN STANDARDS FOR the analysis of textile materials have been issued prescribing standard methods for estimation of (1) moisture, total size, starch, ash and wax content in grey and bleached cotton materials, sampling procedures, (2) determination of copper number of cotton textile materials, and (3) methods for analysis of and tolerances for water for textile purpose.

Copies of these standards are available from the Secretary (Administration), Indian Standards

Institution, 19 University Road, Delhi 2.

The Institution of Land Surveyors

AT ITS ELEVENTH MEETING HELD in New Delhi on May 8, 1950, under the presidentship of the Hon'ble Pandit Jawaharlal Nehru, the Advisory Committee for Co-ordinating Scientific Work Sub-Committee finalized a plan for the establishment of the Institution of Land Surveyors in India. The headquarters will be located at Delhi with branches at other centres. The membership will consist of four classes: students, associate members, members and honorary members.

The aims and objects of the Institution are: to function as a learned scientific body and promote the science and practice of surveying, namely land surveying, building and quantity surveying and other branches such as valuation, land agency, mining and agriculture. Further, it would endeavour to foster all objects having a bearing on the professional activities of surveyors, including liaison with other allied recognized professional bodies; provide a channel with other allied recognized professional bodies and diffuse among corporate members information on all matters affecting surveying.

The Institution also will *inter alia* test by examination the competence of persons for admission to corporate membership of the Institution. At present the only place of training in India is the Geodetic Training Centre of the Survey of India at Dehra Dun and although departmental examinations are held for trainees, they are not given any diplomas or certificates as a result of the tests, but have to go abroad if they wish to obtain any degree or diploma. This lacuna would be remedied by the holding of examinations by the Institution of Surveyors.

Further information on the subject may be obtained from the Surveyor General of India, New Delhi.

Marine Biological Station at Andamans

AT ITS MEETING HELD ON THE 14th December 1948, the Advisory Committee for Co-ordinating Scientific Work considered a scheme for the establishment

of a Marine Biological Station prepared by Dr. Baini Prashad, Fisheries Development Adviser to the Government of India, and recommended that it would be of advantage from the point of view of developing the fishery industry, if a "Fishery Development Station combined with a Research Station" could be set up in the Andamans. This station could later on be developed further, and as the UNESCO has under consideration the establishment of one or more Institutes of Oceanography and Fisheries in Asia, the existence of this station would be an invitation to the United Nations to set up such an Institution in the Andamans.

The Committee, at its recent meeting, held on the 20th May 1950, considered that the scheme for the establishment of a Marine Biological Station in the Andamans should be proceeded with by the Department of Scientific Research and as a first step the Department should appoint a scientist to plan the setting up of a station on an adequate scale. The plan should be such that the scheme could be implemented by stages, so that funds are not required all at once.

River Research Institute, West Bengal

THE FOUNDATION-STONE OF THE central laboratory of the River Research Institute at Haringhata was laid on the 21st May 1950 by Dr. B. C. Roy, Chief Minister of West Bengal.

The Institute will investigate into the causes of deterioration in the drainage system in West Bengal and find ways and means to revive Bengal's rivers which are fast silting up and causing formation of swamps and difficulties in navigation.

Abstracting Services

THE FIRST EDITION OF THE "LIST of Current Specialized Abstracting and Indexing Services" has been prepared by the International Federation for Documentation. The list is a first attempt to prepare a guide of abstracting and indexing services, mainly in the field of pure and applied science. The list contains the titles and places of publication of abstracting and indexing periodicals; also the numbers of abstracts they produce, their frequency of pub-

lication, subscription rates and the languages in which they appear. Copies (Price, 26.13 Dutch florins) of the original card-files of abstracting and indexing services at the FID Secretariat may be obtained from the FIX, 6 Willem Witsen-plein, The Hague Netherlands.

Japanese Technicians for India

SCAP HAS NOW AGREED, SUBJECT to certain conditions, to permit the employment of Japanese technicians in India. Indian industrialists desirous of obtaining their services should forward their applications through the Ministry of Industry & Supply which, in consultation with the Ministry of Finance, scrutinizes these applications before forwarding them to the Indian Liaison Mission to Tokyo which is the actual recruiting authority.

Announcements

International Congress on Archives—The first International Congress on Archives is due to meet in Paris from August 23 to 26, 1950. Dr. Purnendu Basu, Director of Archives, Government of India, has been elected as a member of the Congress vice Dr. S. N. Sen, former Director of Archives.

U.S. Travel Grants for Indian Students—The United States Educational Foundation established in India under the Fulbright Agreement to finance educational exchange programmes, will also utilize part of its funds to provide free transportation to selected Indian scholars who wish to attend U.S. educational institutions in the United States, Hawaii, Alaska (including the Aleutian Islands), Puerto Rico and the Virgin Islands. Applications on prescribed forms may be made direct to the Executive Secretary, U.S. Educational Foundation, American Embassy, Queensway, New Delhi. The forms are obtainable from the same office.

Government of India Overseas Scholarships—The Government has decided to award 6 scholarships under the modified scheme this year by an open selection to be conducted by the Union Public Service Commission. Applications will be received by the Commission in accordance with the instructions to be issued shortly.

Organization & Progress of Scientific Research in India

THE annual report of the Department of Scientific Research, Government of India, for the year 1949-50 records considerable progress in all its activities and particularly in the organization of scientific work. The report is in two parts: the first deals with the organizational activities, the work of the Atomic Energy Commission and the Advisory Committee for Co-ordinating Scientific Work, grants-in-aid to scientific societies and research institutions and scientific liaison. The second part gives a brief résumé of the activities of the Council of Scientific & Industrial Research under the Directorship of Dr. Shanti Swarup Bhatnagar.

The Atomic Energy Commission is taking stock of the country's resources for the development of atomic energy process with a view to planning its future development. Developments in the field of atomic energy will depend a great deal on fundamental research, and to encourage this the commission has sponsored a large number of research schemes on nuclear physics. During 1948-49 and 1949-50 the Commission sanctioned grants totalling Rs. 2,33,332 and Rs. 2,25,150 respectively for this purpose.

Considerable progress has been recorded in the field of cosmic rays and in the training of physicists, chemists and technicians for specialized work on atomic energy. Instruments employed in atomic energy research are being made at the Tata Institute of Fundamental Research, Bombay. The Commission's laboratory is located in Bombay in close proximity to the Tata Institute.

Under the auspices of the Commission, a conference of representatives of universities and scientists was held in January 1949 to draw up syllabuses for the teaching of mathematics, physics and chemistry in Indian universities. The courses have been finalized. The expert assistance of Prof. S. N. Bose of Calcutta University has been taken for the promotion and teaching of these subjects in universities. A chair of Theoretical Physics is being maintained at the Physical Research Laboratory, Ahmedabad. The Commission proposes to subsidize the teaching of other universities, where necessary, to enable the training of students to the standard required for work under the Atomic Energy Commission.

A Rare Minerals Section of the Commission has been set up to survey the location of minerals useful for atomic energy work. This unit has surveyed the beryl deposits in the country. The geologists of the unit are advising mine-owners on the methods of mining the ore and exploratory surveys have been undertaken for the location of new deposits. An assessment of India's monazite resources is also being made and detailed survey is in progress. A factory for processing 1,500 tons of monazite sands is being set up. When the factory will be working in full swing, India would be self-sufficient with respect to thorium, cerium and other rare earth metals, which are now being

imported for use in the manufacture of gas mantles, metallurgical industries, etc.

The Department of Scientific Research effects co-ordination of scientific work through an Advisory Committee of scientists representing the various Ministries of the Government of India. The Committee, presided over by the Hon'ble Prime Minister, has met 5 times during the year. The more important schemes drawn up under the aegis of this Committee are: (1) A scheme for the establishment of a Marine Biological Station in the Andamans; the scheme is being implemented. (2) A micro-film service unit, which was previously working for the Indian Research Fund Association, has now agreed to carry out work for all the Ministries of the Government of India, as well as for private scientific societies, research institutes and universities. (3) A special committee has been set up to compile instructions for the preparation of scientific communications. The same committee will draw up instructions for the training of scientists for information services. (4) Ways and means for setting up of national parks and sanctuaries in India for the conservation of wild life are being examined by a sub-committee constituted for the purpose. (5) The Indian National Committee on Type Cultures is preparing a directory of type cultures maintained at different centres in India. Supplies of type cultures are being made to laboratories and industries. (6) Various committees functioning both under Government and semi-Government auspices have been examined and suggestions made for setting up of committees for subjects which were previously not being studied. The most important of these is the committee under the Ministry of Communications to advise that Ministry on technical, administrative and economic aspects of air services in India. (7) A plan for the formation of an Institution of Land Surveyors has been finalized. (8) A science congress of people living around the Indian Ocean is to be held in India in 1951 under the aegis of the Department of Scientific Research and the Indian Science Congress Association. (9) A monthly publication describing in popular language the progress of scientific research in India is to be published and (10) the Department issues bi-monthly reports giving the latest scientific and technical information.

The bi-monthly reports from the Indian Scientific Liaison Office in U.K. describing technical and scientific developments in the laboratories of the U.K. have proved most useful to research institutions and universities in the country. The Indian Scientific Liaison Officer in U.K. works as the Assessor to the Pest Infestation Board of the Department of Scientific & Industrial Research, U.K.

The Department has, since its inception, served as a clearing house for scientific information. An index of scientific articles translated from various languages into English has been prepared.

India is a member of the International Council of Scientific Unions and of a number of Inter-

national unions. The Department collects literature from these Unions and circulates them to Ministries, universities, research institutions and scientific societies.

A project report on "Science and Its Social Relations" is being prepared by Mr. J. M. Sen with a view to introduce that subject for study in the universities of India.

The year under review has been a landmark in the progress of activities of the *Council of Scientific & Industrial Research*. The following 5 National laboratories started work in their own buildings: The National Physical Laboratory, New Delhi; The National Chemical Laboratory, Poona; The National Metallurgical Laboratory, Jamshedpur; The National Fuel Research Institute, Dhanbad; and The Central Glass & Ceramic Research Institute, Calcutta.

The first two, the National Physical Laboratory and the National Chemical Laboratory, were formally declared open in January 1950. The Central Drug Research Institute at Lucknow and the Central Food Technological Institute at Mysore also started work in the palatial buildings acquired for the purpose.

There are at present 23 Research Committees, each devoted to special branches of science. The Committees keep a watch on the progress of scientific research and developmental work in India in their special fields and advise the Council from time to time on the steps to be taken for the furtherance of these activities. 89 schemes of research were sponsored by the Council during the year and

are being investigated in the various universities and research institutions.

The more important projects carried out during the year are: production of pure manganese from Indian raw materials; dyestuff intermediates for direct cotton colours by electrolytic reduction of aromatic nitro compounds in acid and alkaline media and the analysis of well-known dyestuffs and their duplication in India; printing ink for newspapers; synthetic tanning materials from bhilawan nuts; washability of Indian coals and blending of coals for coking; studies in the construction of low-cost houses; anti-malarial drugs; production of radio valves in India; substitution of nickel by manganese in the production of austenitic steels; developments of new strains of yeast for producing high-concentration distillery washes; insulation boards for prefab. houses; survey and collection of medicinal plants; fundamental researches on X-ray investigation of coal; ionospheric investigations; spectroscopy of fuels; industrial catalysts; infra-red radiation from the atmosphere; properties of quartz; and diamond tools — all sponsored by the Council.

Three new committees, viz. the Salt Research Committee, the Mining Research Station Committee and the Malaria Chemotherapy Committee, were constituted during the year.

With a view to foster the study of microwaves and radar and their applications in meteorology, defence and civil aviation and scientific research in general, the Council organized a symposium on microwaves during the year.

FORTHCOMING INTERNATIONAL CONGRESSES — 1950

<i>Date</i>	<i>Conference</i>	<i>Sponsored by</i>	<i>Venue</i>
Aug. 10	Executive Board International Council of Scientific Unions	ICSU	Berne
Aug. 14	Union Internationale d'Histoire De Sciences General Assembly and 6th International Congress	IUHS	Amsterdam
Aug. 15	16th International Congress of Physiology	...	Copenhagen
Aug. 17	5th International Congress for Microbiology	...	Rio de Janeiro
Aug. 22	International Academy of History of Sciences	...	Amsterdam
Aug. 28	Executive Committee, International Geographical Union	IGU	London
Aug. 30	International Conference on Mathematics	American Mathematical Society	Cambridge, Mass., U.S.A.
Sept. 4	International Congress of Cell-Biology	...	Yale University
Sept. 4	Joint Commission on Ionosphere	ICSU	Brussels
Sept. 11	International Meeting of the Association for the Advancement of Science	UNESCO	Paris
Sept. 11	Union Radio Scientifique Internationale	URSI	Zürich
Sept. 12	Symposium on Motions of the Earth's Crust	I.U.P.M. I.G.G.U.	Washington
Oct. 2	Pan American Institute of Geography and History	...	Chile Santiago
Oct. 4	International Union for Exploration of the Ocean	...	Edinburgh

INDIAN PATENTS

The following is a list of a few of the Patent Applications notified as accepted in the *Gazette of India*, Part II, Section 1, for May 1950.

Organic Chemicals

41176. Improvements in the production of aromatic hydrocarbons: *Vaporized hydrocarbon is passed through a reactor fitted with non-absorptive ceramic bodies in the temperature region of 1,000°C.* — PETROCARBON LTD.

41178. Conversion of hydrocarbons by thermal treatment: *Passing the liquid starting material through a number of reactors fitted inside a furnace heated by burners or gases wherein a cooling of blanket of gases is introduced into the furnace* — PETROCARBON LTD.

41927. Improvements in the preparation of pteridines: *Mixing a 2-amino-4-hydroxy-6 or 7-halomethyl pyrimido (4, 5-b) pyrazine with para-amino benzoic acid of a salt, ester or amide thereof in water at a pH of 9 to 11.5* — AMERICAN CYANAMID Co.

42888. Improvements in the preparation of phenothiazine derivatives: *Condensation effected in presence of alkali metals or their derivatives which form metal derivatives of phenothiazine under conditions of reaction* — SOCIETE DES USINES CHIMIQUES RHONE-POULENC

42037. Manufacture of new phenanthrene carboxylic acid nitriles and derivatives thereof: *By treating a 1-keto-2:13-dimethyl perhydrophenanthrene with a halogenating agent and reacting the resulting halide with a metal cyanide* — CIBA LTD.

42145. Improvements in the chlorination of organic compounds: *Introducing water or aqueous solution into reaction vessel at the top so that it passes through the compound to be chlorinated* — KONINKLIJKE INDUSTRIEEL MAATSCHAPPIJ VOORHEEN NOURY & VAN DER LANDE N.V.

42164. New pyrimidine derivatives: *Reacting a halogenated lower alkylamino pyrimidine derivative with a quarternary salt-forming agent* — I.C.I. LTD.

42165-66. New quinoline derivatives: *2:4 and/or 6-amino, alkyl and/or alkylamino pyrimidine nuclear is condensed with alkylamino quinaldine nuclear, 4-alkylamino-6-(2', 6' or 4' alkyl and/or alkylamino pyrimidyl)-quinaldine is reacted with a quarternary salt-forming agent* — I.C.I. LTD.

42430-37. Process for the manufacture of organic amino diols: *Phenyl alkyl ketone is condensed with β -nitroethanol and thereafter reducing the nitro diol into amino diol compound.*

Process for the manufacture of diacylated organic amino diols: *1-phenyl-2-aminopropane-1:3-diol is heated with excess of acyl halide or acyl anhydride to obtain 1-phenyl-2-acylamido-3-acyloxypropane-1-ol compound.*

Process for the manufacture of completely acylated organic amino diols: *1-phenyl-2-amino (or acetamido) propane 1:3-diol is reacted with acyl anhydride or acyl halide under anhydrous*

condition to obtain the corresponding acylated compound.

Process for the manufacture of aromatic nitro compounds: *1-phenyl-1:3-diacloxy-2-acylamino propane is nitrated with sulphuric acid-nitric acid mixture to obtain 1-para-nitrophenyl-1:3-diacloxy-2-acylamino propane.*

Process for the manufacture of organic amino diols: *1-p-nitrophenyl-1:3-diacloxy-2-acylamino propane is hydrolysed with an acidic or alkaline hydrolytic agent to obtain a free amino diol.*

Process for the manufacture of diastereoisomers of organic amino diols: *Crystallizing the compound from an organic solvent in which one of the structural forms is more soluble than the other and separating the least soluble therefrom.*

Process for the manufacture of optically active organic amino diols: *Reacting the amino diol with an optically active acid and then separating the acid addition salts of the dextro and levo isomers* — PARKE, DAVIS & Co.

Fuels & Lubricants

41993. Improvements relating to medium for sink-and-float coal washing plants: *Subjecting coarse blast-furnace flue dust to a magnetic concentration, the recovered finely divided magnetic iron oxide provides the separating medium* — SIMON-CARVES LTD.

Metal & Metal Products

41831. Method of producing nodular cast iron: *Melting a carbide metal stabilizer agent to a molten iron, then graphitizing the melt and pouring a casting from the melt* — SMALLEY

41832. Method for desulphurizing iron and steel with metal hydrides: *Introducing a reagent comprising at least one metal to reduce sulphur content* — SMALLEY

Paper & Allied Products

42068. Lowering the hemicellulose and ash content of chemical wood or bamboo pulp in the manufacture of high alpha cellulose content pulps for use in the manufacture of paper, rayon, acetate silk, plastics, etc.: *Treating with alkali and finally bleaching with hydrochloride* — TRIVEDI

Miscellaneous

42370. Improvements in the treatment of cellulosic material to impart flame resistance thereto: *Impregnating cellulosic material with an ingredient comprising metallic oxides, a thermoplastic substance, a water soluble salt of phosphorous and a water soluble nitrogenous substance, such as urea* — AMERICAN CYNAMID Co.

42826. Improvements in method of separating stalk and foreign matter from dried or "fired" tea and for cutting or breaking the tea at the same time, if desired — RUDDIMAN

Patented Inventions of the Council of Scientific & Industrial Research

CONVERSION OF CASHEW-NUT SHELL LIQUID TO A DRYING PRODUCT

Patent No. 28424

S. SIDDIQUI & AZHAR ALI KHAN

CASHEW-NUT SHELL LIQUID IS HEATED WITH BHILAWAN shell liquid or bhilawan nuts at temperatures between 200° and 350°C. The heating may be with aeration and should preferably be in contact with sulphur or a metal whose melting point is below 1,000°C. The metal may be substituted by its alloy or amalgam or any of its oxygen, sulphur or halogen-containing compounds. The resulting drying product can be used for the manufacture of lacquer varnishes, stoving enamels, waterproofing, electric insulating and plastic compositions or like materials.

Examples — (I) 100 parts, by weight, of cashew-nut shell liquid and 25 parts of bhilawan shell liquid are mixed and heated for about 4 hr. in contact with tin at 300° to 350°C.

(II) 100 parts, by weight, of cashew-nut shell liquid and 100 parts of bhilawan nuts are heated at a temperature of 300° to 350°C. for about 3 hr.; the liquid portion is separated by straining and reheated for about 4 hr. with aeration and in contact with tin.

CONVERSION OF BHILAWAN SHELL LIQUID

Patent No. 29051

S. SIDDIQUI & AZHAR ALI KHAN

RESINS, RESINOLS OR OILS ARE ADDED TO BHILAWAN shell liquid or to the converted drying product of bhilawan shell liquid or to the vegetable oil extract of bhilawan nuts and the mixture is heated, usually for 6 to 8 hr. at temperatures varying from 150° to 320°C. Addition of one or more of the following driers to the mixture improves the speed of conversion and the drying properties of the resulting products: oleates, stearates, acetates and other organic or inorganic salts, oxides and sulphides of metals such as manganese, cobalt and lead. The ingredients are heated alone or in contact with metals, or their alloys or amalgams or their oxygen, sulphur and halogen-containing compounds.

Example — 500 gm. of oil like linseed, castor, neem, groundnut or a mixture of the oils are added to 500 gm. of bhilawan shell liquid and this mixture is gradually heated to 300°C. and maintained at this temperature for 6 to 8 hr. in contact with tin. Instead of oil, the same quantity of pitch, asphalt, bitumen or the like may be added. The resulting product is a solid resin soluble in hydrocarbon solvents.

Patent No. 29938

S. SIDDIQUI & AZHAR ALI KHAN

Bhilawan shell liquid is heated with sulphuric acid or sulphonated products such as methyl

sulphate or sulphonated oils, e.g. sulphonated castor oil. Other resinous materials or synthetic resins may be incorporated. The amount of sulphuric acid or sulphonated products may vary from 3 to 20 per cent by weight of the bhilawan shell liquid. The heating is conducted at a temperature of 100° to 120°C. for 3 to 6 hr. The resulting product varies in its consistency and characteristics according to the duration of heating from a soluble semi-solid or solid to an insoluble, brittle and thermoplastic state. It can be used in the manufacture of lacquer varnishes, stoving enamels, waterproofing, electric insulating and plastic compositions or like materials.

Patent No. 38062

S. SIDDIQUI

Bhilawan shell liquid is copolymerized with turpentine by treatment with nitric acid. Commercial nitric acid may be used or nitric acid may be produced *in situ* by reacting saltpetre with hydrochloric acid. The copolymerized product is thoroughly washed with water, and the acid-free semi-solid resinous mass is dissolved in a mixture of benzene and turpentine or the like solvents for the manufacture of air-drying varnish compositions, enamels or the like.

Example—40 parts of bhilawan shell liquid are dissolved in 60 parts of turpentine in an acid-resisting enamelled vessel kept below 50° to 60°C. 80 parts of commercial saltpetre and 120 parts of commercial hydrochloric acid are added alternately in small quantities, stirring the contents. The entire operation takes about 4 hr. to complete. The resulting semi-solid, polymerized product is washed and further treated as above.

Patent No. 38064

S. SIDDIQUI & D. C. DHAR

Vegetable oils such as groundnut oil or linseed oil are copolymerized with bhilawan shell liquid, and turpentine or rosin (or oleo-resinous materials) by treatment with nitric acid. To make the reaction milder, nitric acid may be produced *in situ* in the reaction mixture by the action of concentrated hydrochloric acid on saltpetre. The acid is first mixed with bhilawan shell liquid and the saltpetre is then added to the mixture in small proportions. The resulting plastic mass is washed with water, pressed dry and dissolved in a mixture of benzene or white spirit and turpentine and filtered to remove any undissolved particles. Varnishes prepared can be employed for waterproofing textiles or imparting artificial leather finishes to cloth, paper or cardboard.

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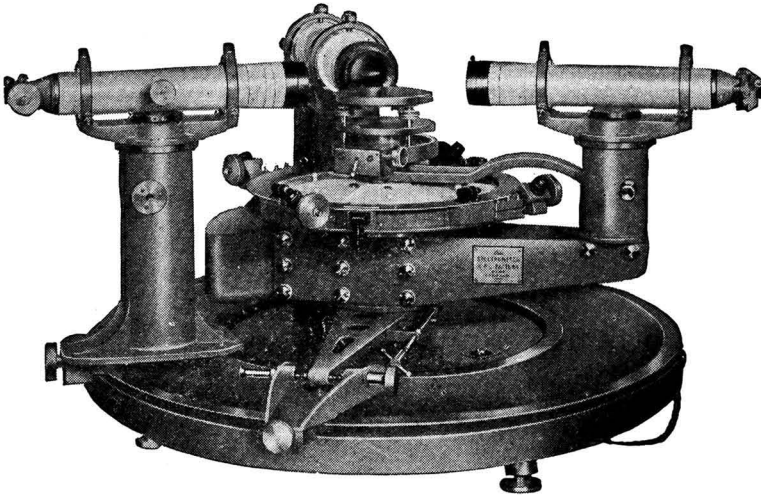
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Alkaloidal Constituents of the Bark of *B. aristata* (Rassaut)

K. K. CHAKRAVARTI, D. C. DHAR & S. SIDDIQUI
Chemical Laboratories, C.S.I.R., Delhi

Berberine, berberine chloride and palmatine have been isolated from the alcoholic extract of the bark of *Berberis aristata*.

ABOUT 13 species¹ of berberis occur in India, mostly in the Himalayan region and in Assam. Four of them also occur in Central India and the Nilgiris and one variety of *B. asiatica* Roxb. in the Parasnath Hills of Chota Nagpur. Some of these species are not easily distinguishable from one another and considerable difficulty is experienced in their identification, particularly because most of the berberis species are known by the same Indian name and are reputed for similar therapeutic properties. Berberis roots and bark are employed in the indigenous systems of medicine as bitter tonics and stomachics. The aqueous extracts of the roots serve as a household remedy in acute conjunctivities, in chronic ophthalmia and also in the treatment of oriental sores. More recently the principal alkaloid of the drug, namely berberine, has been shown to possess antibiotic activity².

In view of the facts stated above, it was considered of interest to undertake a systematic study of the alkaloidal constituents of the various Indian species of berberis. The present paper deals with a study of the alkaloids from the bark of *B. aristata* obtained from Kangra (Punjab). Earlier work³ on this species of berberis was limited to the isolation of berberine (yield, 2.23 per cent) and a minute quantity of the oxalate of an unidentified base. As a result of the present investigation, three naturally occurring crystalline, yellow-coloured salts of bases

melting at 235°C., 220°C. and 165°C. respectively (decomp.) have been obtained by a process of fractionation of the initial crystallize from the alcoholic extract of the bark, on the basis of varying solubilities of the native salts in various organic solvents and water. The mother liquors of these natural salts, all of which proved to be chlorides, further gave berberine through the chloride, and a crystalline iodide, orange-yellow needles, m.p. 241°C. (decomp.). The total yield of the alkaloids in the form of salts amounted to c. 4 per cent on the weight of the dry powdered bark.

The nitrate prepared from the natural chloride, m.p. 220°C. (decomp.) (Cl 9.24 per cent), analysed for $C_{20}H_{18}N_2O_7$ and gave the characteristic colour reactions of berberine. It formed an acetone compound, m.p. 175°C., identical with the anhydroberberine-acetone prepared from berberine chloride. The natural chloride on reduction gave a colourless base, m.p. 170°C., which analysed for $C_{20}H_{21}O_4N$ and gave no depression in melting point with tetrahydroanhydroberberine prepared from berberine chloride. The native salt melting at 220°C. (decomp.) was thus established as berberine chloride.

The iodide, m.p. 241°C. (decomp.), analysed for $C_{21}H_{22}O_4NI$, $2H_2O$ and gave methoxyl value for 4 methoxyl groups. It gave the chloride in the form of greenish yellow needles, $C_{21}H_{22}O_4NCl$, $3H_2O$, m.p. 205°C. (decomp.), and the nitrate, yellow needles, $C_{21}H_{22}O_7N_{21}1.5H_2O$, m.p. 239°C. (decomp.). The analysis and melting points of these salts agreed with those of the corresponding

salts of palmatine. From the chloride a colourless crystalline base, $C_{21}H_{24}O_4N$, was obtained on reduction and the reduced base was found to be identical in melting point with that of tetrahydro-anhydro-palmatine. Although a mixed melting point of the reduced base (m.p. 147°C .) with tetrahydro-palmatine could not be taken owing to the lack of a sample of palmatine, the data presented above leave little doubt as to the identity of the iodide of the base isolated from *Berberis aristata* with palmatine iodide.

Results of studies of the natural chlorides melting at 235°C . (decomp.) and 165°C . (decomp.) will be communicated in a subsequent paper.

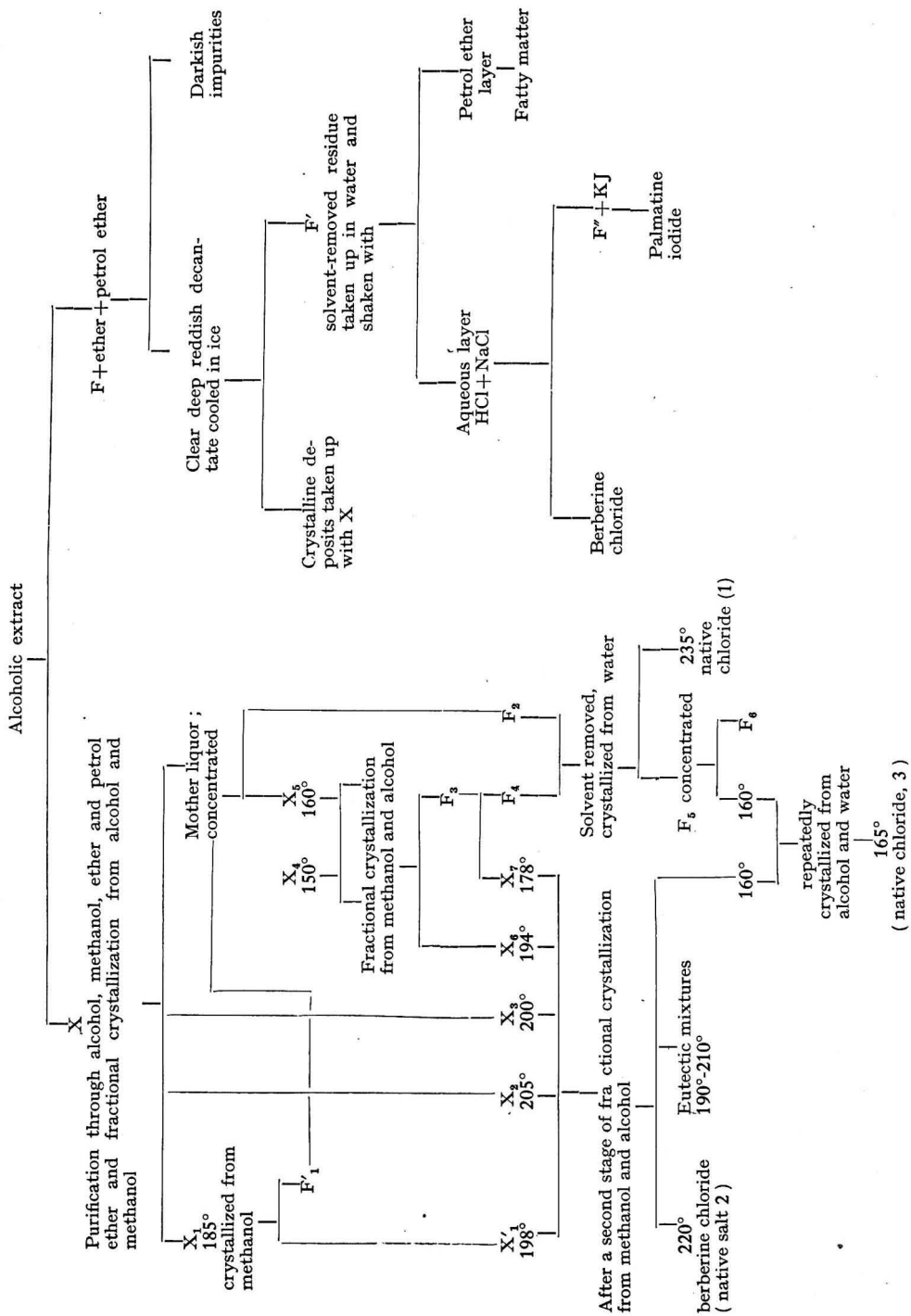
Experimental

7 kg. of well-powdered bark were percolated 4 times with alcohol and the combined percolates concentrated below 50°C . *in vacuo* to a soft extract, which was of a dark yellowish colour. After keeping the concentrated extract for 2 weeks, the initial crystallizate (X) was filtered, washed with cold water and dried on the porous plate (filtrate and washings, marked F in the CHART).

Purification of Initial Crystallizate (X) — 100 gm. of the crude yellowish-brown crystallizate (X) were well powdered, digested with water in the hot and filtered. The undissolved reddish-yellow residue was extracted with hot alcohol and the alcohol-insoluble portion was dissolved in a mixture of alcohol and methanol (1:1). The alcoholic solution was treated with a little ether and petrol ether which removed dark reddish impurities, leaving a clarified liquid from which a light reddish coloured crystallizate (X_1), m.p. 185°C . (decomp.), was obtained on the addition of excess of ether. The filtrate from the ethyl alcohol-insoluble fraction of the initial crystallizate was allowed to stand for sometime in the cold and the yellow crystalline mass (X_2), m.p. 205°C . (decomp.), which separated out was filtered and washed with alcohol. The filtrate was then concentrated to a small volume, when it gave a further quantity of a yellow crystalline product (X_3), m.p. 200°C . (decomp.). The mother liquor from (X_3) gave on treatment with ether a crystalline precipitate (X_4) which melted at 150°C . By this method of initial purification and fractionation 4 crystallizates were obtained, the melting

points of which ranged from 205°C . down to 150°C . in the case of the last fraction. After a second stage of fractional crystallization (*vide* CHART), the higher melting fractions (m.p. 178°C .- 205°C .) were again fractionally crystallized from methanol and alcohol, when they finally gave a product from the top fractions which melted at 220°C . (decomp.). The filtrates obtained in the course of initial fractionation of the crystallizates (F_4 and F_2) were freed of the solvent by evaporation in smaller lots, the residues were dissolved in water, the solution treated with activated charcoal, filtered and allowed to crystallize, when a product melting at 230°C . was obtained which, on repeated recrystallization from alcohol, finally melted at 235°C . The intermediate fractions melting between 210°C . and 190°C . obtained in the course of fractional crystallization of crops melting from 178°C . to 205°C . were set aside for the present, as they appeared to constitute intractable eutectic mixtures. From the combined tail fractions from this side and from the mother liquors of the fraction melting at 235°C . (m.p. 150°C .- 60°C .), repeated crystallizations from alcohol and water ultimately led to a third definitely uniform product, which melted at 165°C . and the melting point of which could not be raised higher on further crystallization from these solvents.

Isolation of Berberine & Palmatine from the Mother Liquors (F) of the Initial Crystallizate — The alcoholic filtrate (F) from the initial crystallizate of the native salts was purified by the addition of ether and petrol ether, which precipitated out a dark brownish sticky mass. The clear deep reddish decantate was freed of the solvent and the residue was taken up in about 500 c.c. water and shaken out repeatedly with petrol ether to remove fatty matter. The yellow-brown aqueous layer was acidified with hydrochloric acid and treated with sodium chloride to saturation. The heavy yellow precipitate was filtered, well washed with cold water, dried on a porous plate (125 gm.) and repeatedly crystallized from alcohol and water, when berberine chloride, m.p. 220°C . (decomp.), was ultimately obtained. The filtrate from crude berberine chloride was treated with potassium iodide and the orange-yellow ppt. was filtered, washed with cold water, dried (50 gm.) and repeatedly crystallized from a mixture of methanol and 10 per cent



acetic acid when it finally melted at 241°C. (decomp.) (CHART). Found C, 48.65; H, 5.25; N, 2.90 per cent; OMe, 23.82; I, 24.12 per cent; required for $C_{21}H_{22}O_4NI$, $2H_2O$ (palmatine iodide): C, 48.93; H, 5.04; N, 2.72; OMe, 24.66; I, 24.07 per cent.

Characterization of the Natural Alkaloidal Salts — The aqueous solution of the natural salt melting at 220°C. was treated in the hot with a saturated solution of KNO_3 , and the crystalline nitrate of the base which came out on cooling was filtered. Addition of silver nitrate solution to the filtrate gave the curdy-white precipitate of silver chloride. The salts melting at 235°C. and 165°C. also showed a similar behaviour and all the natural salts were thus proved to the chlorides.

Nitrate from the Natural Salt (m.p. 220°C.): Berberine Nitrate — The nitrate prepared from the natural salt in the manner described above or by warming an aqueous solution of it with dilute nitric acid was repeatedly crystallized from water, when it was finally obtained as fine yellow needles, m.p. 275°C. (decomp.). Found C, 59.92; H, 4.67; N, 7.12 per cent; required for $C_{20}H_{18}N_2O_7$: C, 60.30; H, 4.52; N, 7.04 per cent.

Acetone compound, prepared according to the prescribed method, melted at 175°C., and its mixed melting point with an authentic sample of the acetone compound of berberine showed no depression in a parallel observation of the melting points.

Reduction of Natural Salt (m.p. 220°C.): Tetrahydro-berberine — 2 gm. of the natural salt were catalytically reduced with platinum oxide in methanol solution. The residue left over after filtration from platinum oxide and evaporation of the solvent was treated with fairly concentrated ammonia to liberate the reduced base, which was filtered, washed with cold water, dried and crystallized from a mixture of benzene and petroleum ether, colourless needles, soluble in benzene, insoluble in petrol ether and ether were

obtained, m.p. 170°C.; yield, 1.8 gm. Found C, 70.28; H, 5.92; N, 4.01 per cent; required for $C_{20}H_{21}O_4N$: C, 70.8; H, 6.19; N, 4.12 per cent.

Palmatine Chloride — 5 gm. of iodide melting at 241°C. were treated in methanol medium with an excess of freshly precipitated silver chloride and the pasty mass extracted with methyl alcohol and filtered. The filtrate was freed of the solvent, taken up in a little warm alcohol from which palmatine chloride crystallized out in orange-yellow needles, m.p. 205°C. (decomp.). Found Cl, 7.99 per cent; required for $C_{21}H_{22}O_4NCl$, $3H_2O$: Cl, 8.04 per cent.

Palmatine Nitrate — 0.2 gm. of the chloride was treated with 5 c.c. of dil. nitric acid, when the nitrate of the base came out as a yellow crystalline mass, which crystallized from water in greenish yellow needles, m.p. 245°C. (decomp.). Found C, 56.10; H, 6.01; N, 6.46; OMe, 23.4 per cent; required for $C_{21}H_{22}O_7N_2$, $1\frac{1}{2}H_2O$: C, 57.14; H, 5.66; N, 6.35; OMe, 24.07 per cent.

Tetrahydro-palmatine — 2 gm. of the chloride reduced with platinum oxide in methanol solution and worked up in the manner described in the case of tetrahydroberberine gave the reduced base in the form of colourless needles, soluble in alcohol, methyl alcohol, chloroform, sparingly soluble in ether, and insoluble in petrol ether, m.p. 147°C.; yield, 1.7 gm. Found, C, 71.18; H, 6.77; N, 3.95 per cent; required for $C_{21}H_{24}O_4N$: C, 71.02; H, 6.98; N, 4.13 per cent.

Our thanks are due to Mr. Baldev Singh for his help in providing the plant material and to Mr. S. C. Misra for his assistance in the initial stages of the isolation work.

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2. Dick: *Arch. Surg. Chicago*, 1940, 41, 287.
3. RAY & ROY: *Sci. & Cult.*, 1942, 7, 619.

Studies in the Syntheses of Quinolino- α -Pyrones—Part I

D. P. AHUJA, K. K. CHAKRAVARTI & S. SIDDIQUI
Chemical Laboratories, C.S.I.R., Delhi

The syntheses of 4-hydroxy-quinaldino-6-5- α -pyrone ($C_{13}H_9O_3N$), (2:3-cyclopenteno)-4-hydroxy-quinolino-6:5- α -pyrone ($C_{16}H_{11}O_3N$) and (2:3-cyclohexeno)-4-hydroxy-quinolino-6:5- α -pyrone ($C_{18}H_{13}O_3N$) have been effected by the application of Conrad and Limpach reaction.

IN view of the physiological activity characterizing both coumarins and alkaloids, it was considered of interest to synthesize and study the physiological activity of compounds containing both the lactone group of the coumarins and the basic nitrogen system of the alkaloids. Some compounds of this type, viz. quinolino-6:5- α -pyrone, have already been synthesized by Dey *et al.* by the application of the Skraup's reaction to 6-aminocoumarin¹ and also by applying the Doebner-Miller reaction to 6-aminocoumarin, 6-amino-4-naphthopyrone², 5-amino-8-methoxycoumarin and 6-amino-8-methoxycoumarin³, but no mention of their physiological activity is available in literature.

In the present investigation, 4-hydroxy-quinaldino-6:5- α -pyrone, $C_{13}H_9O_3N$ (I), (2:3-cyclopenteno)-4-hydroxy-quinolino-6:5- α -pyrone, $C_{16}H_{11}O_3N$ (II) and (2:3-cyclohexeno)-4-hydroxy-quinolino-6:5- α -pyrone, $C_{18}H_{13}O_3N$ (III) have been synthesized. These bases have been prepared by the cyclization of the intermediate products, ethyl-3'-(6-aminocoumarin)-crotonate, $C_{15}H_{15}O_4N$ (IA), ethyl-5'-(6-aminocoumarin)-1':5'-cyclopentene carboxylate, $C_{17}H_{17}O_4N$ (IIA) and ethyl-6'-(6-aminocoumarin)-1':6'-cyclohexene carboxylate, $C_{18}H_{19}O_4N$ (IIIA) obtained by the condensation of 6-aminocoumarin with ethyl acetoacetate, ethyl-cyclopentanone-2-carboxylate and ethyl-cyclohexanone-2-carboxylate respectively, under conditions which exclude the formation of the corresponding anilides, according to the method of Conrad and Limpach⁴. Angular structures have been assigned to

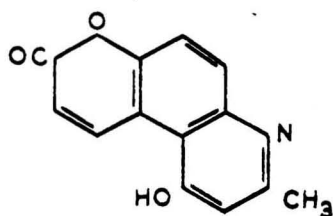
these bases in consideration of the observations of Dey and Goswami¹.

The bases have been obtained in good yields. They give copious precipitates with the usual alkaloidal reagents and respond to the characteristic colour reactions of alkaloids. The basic nature of these substances is not prominent owing to the presence of a phenolic hydroxyl group, which gives them an amphoteric character and is responsible for their solubility in dilute alkalis. The weak basic character is also responsible for the ease with which the hydrochlorides get hydrolysed in the presence of water. Further work in this line is in progress.

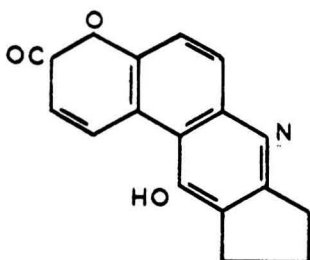
Experimental

Ethyl-3'-(6-aminocoumarin)-crotonate, $C_{15}H_{15}O_4N$ (IA) — A mixture of 6-aminocoumarin (2.1 gm.) and ethyl acetoacetate (2.7 gm.) was warmed on the water bath and the resulting solution kept overnight after adding a drop of 50 per cent hydrochloric acid, in a flask fitted with calcium chloride guard tube. The condensation product which separated as a white crystalline mass was collected, washed with 5 per cent sodium hydroxide solution (50 c.c.) and washed free of alkali. It crystallized from dilute alcohol in white plates, m.p. 126°C., yield, 70 per cent. Found: N, 5.06 per cent; $C_{15}H_{15}O_4N$ requires: N, 5.13 per cent.

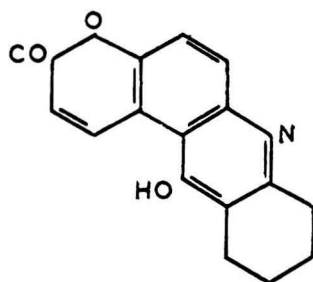
4-hydroxy-quinaldino-6:5- α -pyrone, $C_{13}H_9O_3N$ (I) — 40 c.c. of liquid paraffin was heated to 260°C. and 1 gm. of the intermediate (IA) was quickly added on to it, the heating being continued for 1 min. at a temperature range of 260°-70°C. It was then allowed to cool down to the room temperature, diluted with a little petrol ether. The resultant product was filtered, washed with petroleum ether, dissolved in hydrochloric acid and precipitated with ammonia after filtering off the suspended



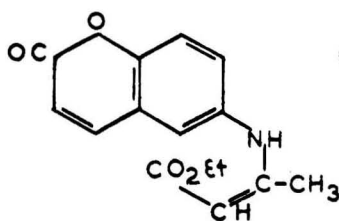
I



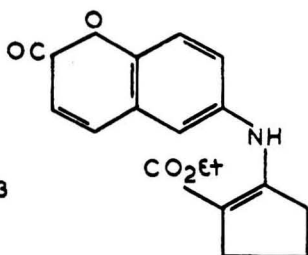
II



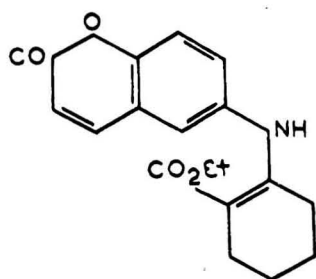
III



IA



IIA



IIIA

impurities. The base thus obtained crystallized from absolute alcohol in light-yellow needles, m.p. 312°C. (decomp.). It is sparingly soluble in benzene and ethyl acetate, fairly soluble in absolute alcohol, moist acetone, xylene and cyclohexanone, insoluble in ether and petroleum ether; yield 90 per cent. Found: N, 6.21 per cent; $C_{13}H_9O_3N$ requires: N, 6.17 per cent.

The *hydrochloride* obtained by dissolving the base in concentrated hydrochloric acid and evaporating off the acid, formed white crystalline needles which, after washing with alcohol, melted at 250°C. (decomp.). The hydrochloride is sparingly soluble in alcohol, insoluble in other organic solvents and is unstable in water.

The *chloroplatinate* obtained by adding a 2 per cent solution of platinum chloride to a solution of the base in fairly concentrated hydrochloric acid, formed buff-coloured needles melting at 280°C. (decomp.).

The *picrate* was prepared by adding a saturated solution of picric acid in glacial acetic acid to the base dissolved in a little

of the same solvent. It formed deep-yellow rhombic crystals, m.p. 283°C. (decomp.).

Ethyl-5'-(6-aminocoumarin)-1':5'-cyclopentene carboxylate, $C_{17}H_{17}O_4N$ (IIA) — To a clear solution of 6-aminocoumarin (1.08 gm.) in cyclopentanone carboxylic ester (1.30 gm.), obtained by warming the mixture on a water bath, a drop of 50 per cent solution of hydrochloric acid was added and the solution kept in the flask fitted with a calcium chloride guard tube for 12 hr. The separated crystals were filtered, washed first with 5 per cent sodium hydroxide solution and then repeatedly with water. On re-crystallization from alcohol the product formed pale-yellow rods, m.p. 153°C.; yield, 90 per cent. Found: N, 4.67 per cent; $C_{17}H_{17}O_4N$ requires: N, 4.68 per cent.

(2:3-Cyclopenteno)-4-hydroxy-quinolino-6:5- α -pyrone, $C_{18}H_{11}O_3N$ (II) — The intermediate (IIA) was cyclized by following the procedure noted for base I. The resulting base crystallized from absolute alcohol in cream-coloured needles, m.p.

330°C. (decomp.). It is sparingly soluble in benzene and ethyl acetate and fairly soluble in absolute alcohol and cyclohexanone; yield, 60 per cent. Found: N, 5.60 per cent; $C_{16}H_{11}O_3N$ requires: N, 5.53 per cent.

Hydrochloride — Colourless hexagonal plates, m.p. 360°C. (decomp.).

Chloroplatinate — Brownish-yellow hexagonal plates, m.p. 250°C. (decomp.).

Picrate — Deep-yellow rhombic crystals, m.p. 238°C. (decomp.).

Ethyl-6'-(6-aminocoumarin)-1': *6'-cyclohexene carboxylate*, $C_{18}H_{19}O_4N$ (IIIA) — 6-Aminocoumarin (0.3 gm.) was heated on a water bath with an excess of cyclohexanone carboxylic ester (0.5 gm.) for 2 hr. and the resultant solution left overnight after adding a drop of 50 per cent hydrochloric acid. The solid mass which separated was treated with sodium hydroxide solution (5 per cent), filtered, washed free of alkali and crystallized from alcohol; pale-yellow needles, m.p. 132°C.; yield, 95 per cent. Found: N, 4.50 per cent; $C_{18}H_{19}O_4N$ requires: N, 4.47 per cent.

(2:3-Cyclohexeno)-4-hydroxy-quinolino-6:5- α -pyrone, $C_{16}H_{13}O_3N$ (III) — 0.3 gm. of the intermediate (IIIA) was added to liquid paraffin (20 c.c.) heated to 270°C. and kept at that temperature for 5 min. The dark-brown product which separated out after the initial solution of the inter-

mediate was filtered after cooling down to room temperature and dilution with a little petrol ether and washed with petrol ether. The product thus obtained was warmed with a little conc. hydrochloric acid and filtered from the non-basic, insoluble, darkish resinous matter. The filtrate was cooled after dilution with twice its volume of water, and then treated with excess of ammonia. The precipitated base was filtered, washed, dried on a porous plate and crystallized from absolute alcohol. It forms pale-yellow needles, m.p. 330°C. (decomp.), is fairly soluble in hot cyclohexanone and sparingly in benzene and ethyl acetate; yield, 70 per cent. Found: N, 5.32 per cent; $C_{16}H_{13}O_3N$ requires: N, 5.24 per cent.

Hydrochloride — White rhombic crystals, m.p. 285°C. (decomp.).

Chloroplatinate — Buff-coloured needles, m.p. 241°C. (decomp.).

Picrate — Yellow rhombic crystals, m.p. 210°C.

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Optical Rotation of Abietic Acid in Alcohols

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The optical rotation of abietic acid from Indian rosin in monohydric alcohols has been determined. The specific rotation of abietic acid in methyl, ethyl and *iso*-amyl alcohols increases with the concentration of the acid; in the case of other alcohols the rotation increases up to a point beyond which it falls.

MITRA and Ghatak¹ and Rau and Simonsen² have shown that abietic acid is the chief constituent of Indian rosin obtained from *Pinus longifolia*. The physical properties (but not the chemical properties) of abietic acid depend upon the method employed for its preparation and purification². Optical rotation is the most important physical constant of abietic acid and is often quoted to the exclusion of all others.

The optical rotation of abietic acid from Indian rosin in monohydric alcohol solvents has been determined. The results obtained are recorded in this paper.

Experimental

Abietic acid was prepared from "water-white rosin" (*Jallo Rosin & Turpentine Factory*, Lahore) by distillation with superheated steam³. Freshly powdered sample of rosin, m.p. 160°-63°C., was used, as powdered rosin on storage gets oxidized. The acid gave: C, 79.02; H, 10.22 per cent; C₂₀H₃₀O₂ requires: C, 79.47; H, 9.93 per cent.

The acid was stored as sodium tetrabutetate and was released as needed from the salt.

The optical rotation of abietic acid in methyl, ethyl *n*-propyl, *iso*-propyl, *n*-butyl, *sec*-butyl, *ter*-butyl, amyl and *iso*-amyl alcohols was determined at 25°C. and the results obtained are recorded in Tables I and II. Pure, re-distilled solvents were employed.

Discussion

The specific rotation of abietic acid was found to obey Drude's simple dispersion

formula, $[\alpha] = \frac{k}{\lambda^2 - \lambda_0^2}$. Straight line graphs are obtained when $\frac{1}{[\alpha]}$ is plotted against

λ^2 . The differences between the observed rotatory power (*c*) and the rotatory power (*C*) calculated from the dispersion equation is within the limits of experimental error. In general the deviation is $\pm 0.03^\circ$, but for Hg₄₃₅₈, which is a difficult line to read, the deviation is $\pm 0.09^\circ$.

The rotatory power of abietic acid in monohydric alcohols is found to decrease in the following order: amyl alcohol > *iso*-butyl alcohol > *sec*-butyl alcohol > *n*-propyl alcohol > *iso*-amyl alcohol > *ter*-butyl alcohol > *n*-butyl alcohol > ethyl alcohol > *iso*-propyl alcohol > methyl alcohol. This sequence does not run strictly parallel to the dielectric constants of the solvents, namely methyl alcohol > *iso*-propyl alcohol > ethyl alcohol > *n*-propyl alcohol > ethyl alcohol > *n*-butyl alcohol > amyl alcohol > *sec*-butyl alcohol > *ter*-butyl alcohol > *iso*-amyl alcohol. It is more logical to correlate the rotatory powers of a substance with the dielectric constants of the solutions and not with those of the solvents used. The values for dielectric constants for solvents have been used in these experiments, as the values for abietic acid solutions were not available. The rotatory power of abietic acid in various solvents was deduced from the rotatory power data for Hg₅₄₆₁. This line was chosen at random. The same sequence was found to hold good with other wavelengths also.

The absorption maxima values obtained from rotatory dispersion measurements are found to be in good agreement with the values obtained by measurements of absorption and by calculation using Woodward's method⁴.

Du pont, Rouin and Dubourg⁵ and George⁶ have shown that the specific rotation of rosin acids depends not only on the solvent

employed, but also on their concentration in the solvent. Systematic work undertaken in this laboratory shows that in methyl, ethyl and *iso*-amyl alcohols, there is an increase in specific rotation of abietic acid as the concentration in the solvent increases. With *iso*-propyl, *n*-propyl, *iso*-butyl, *n*-butyl, amyl, *sec*-butyl, *ter*-butyl alcohols, the specific rotation increases with concentration of abietic acid up to a certain point beyond which it begins to decrease.

The values obtained for the optical rotation of abietic acid in monohydric alcohols can be easily explained on the basis of

association and compound formation of organic acids in solvents.

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Vaccenic Acid & Its Relation to the Alleged Grass-juice Factor

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Feeding experiments on rats show that vaccenic acid has no growth-promoting properties. A butter diet increased the vaccenic acid content of the body fat of the rat, but the increase is unaccompanied by any change in the growth rates. Neither butter nor paddy grass contain any unidentified factors necessary for rat nutrition.

THE superior growth-promoting property of butter has been the subject of many investigations¹⁻¹⁵. Schantz *et al.*¹⁴ claimed that prematurely weaned rats did not grow well owing to the lack of an essential factor present in butter, and that this growth retardation disappeared after six weeks if the animals were fed on a stock diet. When weanling rats were treated to a lactose-rich diet, Boutwell *et al.*¹⁶ found that those receiving butter fat grew better than those receiving vegetable or hydrogenated fats. This superiority was no longer recorded when a mixed carbohydrate diet was given. The reported superiority of butter fat, if lactose was present in the concentration found in skimmed milk¹⁴, has not been confirmed by others^{2,8}. It has been suggested that the better growth

observed with butter fat might be explained on the basis of food preference for the butter flavour, whereby the animals consumed more and grew more rapidly².

Deuel and his associates^{3,4} were unable to confirm the presence of any unidentified nutrient in butter. Their results are supported by those of Zialcita and Mitchell¹⁵, and those of Euler *et al.*^{5,6}. The experiments of Jack *et al.*⁹ as well as those of Henry, Kon, Hilditch and Meara⁸ indicated that there was no one fraction of butter the growth-promoting action of which was superior to that of whole butter. But Geyer *et al.*⁷ and Nath *et al.*¹² have recently reported the superior growth-promoting action of a liquid fraction of butter remaining after separation of the solid portion from an acetone solution at -4°C .

Boer and Jansen¹⁷⁻¹⁹ found that "summer" butter, obtained from the milk of cows fed on green grass, contained a growth-promoting factor, other than vitamin A, and the other known fat-soluble vitamins. They reported¹⁹ that it was the vaccenic acid fraction of summer butter that was responsible for the increased growth. Addition of vaccenic acid to a rape seed oil diet

was found to promote growth as much as summer butter did.

A significant factor in connection with the disputed growth-promoting action of "summer" butter, as compared to "winter" butter, is the reported occurrence of an unidentified nutrient in grasses²⁰⁻²³. The work of Kohler *et al.*²² indicates that grasses contain a grass-juice factor, and that young rats maintained on the milk of cows fed on green grass grew at the rate of 4 gm. a day, whereas rats maintained on mineralized winter milk (i.e. milk yielded by cows on a non-grass winter regimen plus the necessary mineral salts) grew at the rate of only 2.5 gm. per day. The work of Cary and Hartman²⁰ also indicates that besides certain other substances like milk and liver, alfalfa hays, timothy hays and Kentucky blue grass contain an unidentified nutrient or nutrients.

The announcement by Boer *et al.* (loc. cit.) that vaccenic acid was the growth-promoting factor present in "summer" butter, coupled with the report of the presence of still another unidentified factor in fresh grasses, leads one to the plausible supposition that a grass diet enables the cow to elaborate an unidentified "summer" butter factor, which may be identical with vaccenic acid. Feeding green grass might, therefore, result in an increase of the vaccenic acid content of either the body fat alone or both milk fat and body fat. This hypothesis supports the claim of Brouwer and Sjoncker-Schaffner²⁴ that the milk of cows fed on grass contains higher amounts of vaccenic acid.

In the present investigation experiments were carried out to enquire into the presence of still unidentified nutrients in paddy grass and in summer butter, and the possible conversion of the one to the other in the rat. The vaccenic acid content of the body fat of the rats was also determined in order to correlate any higher growth rates with higher contents of vaccenic acid.

Experimental

Young weanling rats (28-30 days old) were divided into 3 groups, each group consisting of 3 males and 3 females. Care was taken to effect equal distribution of litter mates. The diet of these rats was made up as shown below :

Constituent	% by weight of diet
Rice powder*	35
Wheat powder*	35
Casein* (B.D.H. light white)	10
Dried yeast*	6
Salt mixture†	4
Fat	10

Vitamins A and D were added as the unsaponifiable part of a shark liver oil concentrate, dissolved in coconut oil along with vitamin E. For groups I and II the fat given was coconut oil (purified to solidify at 20°-26°C.) and the rats in group III were fed on butter. In addition, the rats in group II received fresh paddy grass (finely ground up) to the extent of 10 per cent of the whole diet. The rats were fed *ad libitum* and care was taken to supply more food to each rat than it actually consumed. The cages were placed in such a manner that coprophagy was eliminated to a considerable extent.

Besides these 3 groups, 4 female rats from the same litters were maintained on our stock diet, which was composed as shown below :

Constituent	Parts by weight
Paddy, rice and wheat finely ground	60
Groundnut cake	10
Pulses	5
Skim-milk powder	10
Salt mixture	1
Arachis oil	1
Dried fish	3
Fresh radish leaves	5
Germinated pulses	5

with additional supply of fresh milk (10 c.c. per rat per day). The growth of these rats was measured by weighing them every week.

After feeding for 40 days on the experimental diets, the rats were killed in the morning and were dissected to see that no food was present in the intestinal tract. The carcasses were minced and dried with plaster of Paris. The dried product was ground and each group was divided into 3 batches for purposes of analysis. Each batch was extracted thoroughly by

* Freed from fat by extraction with light petroleum.

† Phillips and Hart salt mixture²⁵.

TABLE I—VACCENIC ACID CONTENT OF SOME COMMON SOUTH INDIAN FATS

FAT	VACCENIC ACID %
Coconut oil	0.88
Arachis oil	1.14
Butter (pooled sample)	2.36
Pig back fat	0.96
Beef back fat	1.53

TABLE II—INCREASE IN THE WEIGHTS OF RATS (GM.)

NO. OF WEEKS	GROUP I: CONTROL—COCONUT OIL ALONE		GROUP II: COCONUT OIL & PADDY GRASS		GROUP III: BUTTER	
	Males	Females	Males	Females	Males	Females
	1	18.9	15.9	18.9	19.7	15.4
2	37.4	38.7	43.0	36.3	41.9	30.0
3	79.7	59.6	70.3	61.6	70.7	49.8
4	101.4	75.5	95.2	73.8	93.5	63.2
5	114.6	83.7	112.9	85.2	118.2	75.2

refluxing repeatedly with petroleum ether (b.p. 40°-60°C.). The fat was prepared from the extracts by distilling off the petroleum ether on a water bath. The fat was saponified and the fat acids were liberated from the soaps. The fat acids were washed with lukewarm water and weighed. The iodine value of the fat acids was determined according to A.O.A.C. methods (Hanus method). The content of vaccenic acid in the fat acids of the body fat of rats was determined according to the method of Cocks, Christian and Harding²⁶.

of growth. Paddy grass, which is similar to barley, wheat and oat grasses, used by Kohler *et al.* (loc. cit.), did not contain any unidentified growth-promoting factor necessary for the white rat. Also feeding fresh paddy grass has not been found to increase the vaccenic acid content of the body fat of the white rat. Perhaps the elaboration of vaccenic acid from a grass diet is carried out by the cow and not the rat, and the rumen is probably the site of its formation. The ability of the cow to do so seems to be indicated by the fact that beef fat contains higher amounts of vaccenic acid than pig fat.

A butter diet increased the vaccenic acid content of the body fat of the rat. This is due to the higher content of vaccenic acid in butter than in coconut oil. However, the higher vaccenic acid content of the body fat of rats fed on butter was not accompanied by increased growth rates. This demonstrates that vaccenic acid has no growth-promoting properties, a conclusion which is in conformity with other observations^{11,13,27}.

Summary

1. From our feeding experiments, it is concluded that neither butter nor paddy grass contain any unidentified nutrients necessary for the rat.

TABLE III—ANALYSIS OF THE BODY FAT OF RATS

Sex	GROUP I			GROUP II			GROUP III			Stock	
	1	2	3	1	2	2	1	2	3	1	2
	M & M	M & F	F & F	M & M	M & F	F & F	M & M	M & F	F & F	F & F	F & F
Carcass weight, gm.	317.00	302.50	290.00	321.00	373.00	271.20	338	277.00	254.00	273.00	308.00
Fat in carcass, %	16.10	17.00	14.80	16.00	15.30	14.70	Lost	15.30	14.20	10.50	11.20
Iodine value of fat acids	32.27	35.34	38.64	37.15	35.32	33.50	...	42.84	41.62	23.80	26.50
Vaccenic acid in fat acids, %	1.46	1.36	1.53	1.50	1.40	1.38	...	2.10	1.99	0.95	1.21

Vaccenic acid content of some common South Indian fats are given in Table I, while the weekly increases in the growth of the rats placed on the different diets are given in Table II and the vaccenic acid contents of the body fat of rats are given in Table III. The data show no significant differences in the growth of male or female rats over a 5-week period, when the diet contained butter fat or coconut oil. Addition of fresh paddy grass to the diet of rats did not result in any increased rate

2. The white rat cannot elaborate vaccenic acid from a diet containing 10 per cent of paddy grass.

3. Vaccenic acid has no growth-promoting properties.

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Supplementary Value of Groundnut Cake to Tapioca & Sweet Potato

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Addition of groundnut cake flour to diets largely composed of tapioca or sweet potato flour promotes good growth in young rats, whereas the rats fed on tapioca or sweet potato flour containing pulses and vegetables lost body weight. Animals fed on a poor rice diet in which 25 per cent of rice has been replaced by a mixture of 4 parts of tapioca or sweet potato flour and 1 part of groundnut cake flour also grew well, showing thereby that the nutritive value of poor rice diet was improved by replacing some rice by the above mixtures.

IN Travancore and certain other parts of India, tapioca is used as a staple food. More recently the use of sweet potato has also been advocated and attempts are being made to popularize its use. With a view to meeting the present shortage in the supply of cereals, the Subsidiary Food Production Committee of the Government of India recommended: (1) the extensive cultivation and use of tapioca and sweet potato as these tubers give comparatively higher yields (3 to 4 times) of calories per acre than the commoner cereals;

and (2) the utilization of specially prepared groundnut cake flour as human food.

Using the rat growth method, Aykroyd and Krishnan¹ showed that poor diets based on tapioca are deficient in proteins as compared with the poor rice diet. They also investigated the possibility of improving the poor tapioca diet with various supplements like skimmed-milk powder, casein and different pulses. Their investigations showed that casein and skimmed-milk powder proved effective supplements by markedly increasing the growth rate, while the different pulses were comparatively ineffective. Presumably, the relatively low biological value of pulse proteins² partially accounts for the failure of these foods to act as effective supplements.

Since foods of animal origin which are rich in proteins of high biological value are costly and not within the reach of the poor people, there is a need to search for cheap and effective protein supplements to tapioca diet.

Groundnut cake flour is a rich source of proteins, and certain vitamins of the B-

TABLE I.—THE CHEMICAL COMPOSITION OF FOODSTUFF USED IN THE EXPERIMENT

(Values per 100 gm. of food)

FOODSTUFF	MOISTURE, gm.	PROTEIN, gm.	FAT, gm.	FIBRE, gm.	ASH, gm.	CARBOHYDRATE BY DIFFERENCE, gm.	CALORIFIC VALUE, gm.	CALCIUM, gm.	PHOSPHORUS, gm.	IRON, mg.	VIT. B, mg.	NICOTINIC ACID, mg.	RIBOFLAVIN, mg.
Tapioca flour	9.0	1.7	0.5	1.9	1.4	85.5	353	0.06	0.08	33.0*	0.12	1.8	0.03
Sweet potato flour	8.3	1.7	0.9	2.5	3.8	82.8	346	0.11	0.13	28.7*	0.15	2.8	0.05
Groundnut cake flour (without outer red cuticle)	11.0	52.7	8.9	1.0	4.6	21.8	378	0.07	0.50	2.9	0.95	19.5	0.20
Raw milled rice	13.0	6.9	0.4	...	0.5	79.2	348	0.01	0.11	2.2	0.10	1.2	0.02
Whole wheat flour	12.8	12.0	1.5	2.0	1.5	70.2	342	0.05	0.32	5.3	0.45	4.5	0.15
Mixture of 4 parts of tapioca flour and 1 part of groundnut cake flour ¹	9.4	11.9	2.2	1.7	2.0	72.8	359	0.06	0.16	27.0	0.30	5.3	0.06
Mixture of 4 parts of sweet potato flour plus 1 part of groundnut cake flour	8.8	11.9	2.5	2.2	3.9	70.7	353	0.10	0.20	23.0	0.31	6.1	0.08

* Due to contamination during powdering the dried chips in mills.

complex (TABLE I). Consumer trials carried out recently in our laboratory showed that it could be consumed by adults without any adverse effect up to at least 1 ounce per day. It appeared to us that it would be worth while to investigate whether groundnut cake flour has any supplementary value to tapioca and sweet potato. The investigations reported in this paper have shown that groundnut cake flour has a remarkable supplementing value to poor diets based on tapioca and sweet potato flour and a mixture of 4 parts of tapioca or sweet potato flour and 1 part of groundnut cake flour has a nutritive value which is much superior to that of rice.

Experimental

The chemical composition of tapioca, sweet potato and groundnut cake flour as also certain mixtures prepared out of them, as compared with rice and wheat, are given in Table I. It will be seen that the main deficiency of the tuber flours is in regard to the protein. This is largely made up by admixture with groundnut cake flour.

The experimental diets used in the present study are given in Table II. The rat growth method employed by other workers^{1,3} was adopted in these studies.

Seven groups of young albino rats (8 in each group), about 4 weeks' old and weighing 45 to 50 gm., were fed the above diets. To make the feeding uniform, the cereals and pulses were powdered and mixed with the diet. The mixed diet was cooked with one and half times its weight of water to a pasty mass and fed *ad libitum* to the rats. The average weekly increase in body weights of the different groups of animals is given in Table III; the rate of growth of rats on the different diets is shown in Fig. 1.

Discussion

The results confirm the earlier findings of Aykroyd and Krishnan (loc. cit.) that a poor diet based mainly on tapioca does not support life. The response to sweet potato is also of the same order. The addition of groundnut cake flour to the tapioca or sweet potato diet produced, however, a remarkable improvement in the nutritive value of the diet, making it much superior to the poor rice diet. This observation is of further interest as Kuppaswamy *et al.*⁴ showed that addition of groundnut

TABLE II — THE COMPOSITION OF THE EXPERIMENTAL DIETS

NAME OF FOODSTUFFS	DIET I %	DIET II %	DIET III %	DIET IV %	DIET V %	DIET VI %	DIET VII %
Tapioca flour	78.5	...	62.8	15.7	...
Sweet potato flour	...	78.5	...	62.8	15.7
Groundnut cake/flour	15.7	15.7	...	3.9	3.9
Raw milled rice	78.5	58.9	58.9
Red gram (Tur dhal)	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Non-leafy vegetables (brinjal, potato and raw plantain)	8.2	8.2	8.2	8.2	8.2	8.2	8.2
Leafy vegetables (amaranth, cabbage, etc.)	2.1	2.1	2.1	2.1	2.1	2.1	2.1
Crude groundnut oil	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Milk powder	0.9	0.9	0.9	0.9	0.9	0.9	0.9
Common salt	0.3	0.3	0.3	0.3	0.3	0.3	0.3

cake flour at a 10 per cent level to poor rice diet had practically no supplementary value.

It is interesting to note that even in the poor rice diet, replacement of 25 per cent of rice by a mixture of (1) tapioca (80 parts) and groundnut cake (20 parts) or (2) sweet potato (80 parts) and groundnut cake (20 parts) produces striking growth response, thus showing that the supplementary effect of the mixture of the tuber and the cake is to be seen at even comparatively low levels of intake. The available evidence is not, however, sufficient to show whether the supplementation is essentially one of protein or the overall effect including that of minerals and vitamins.

Arising from their earlier investigations, Aykroyd and Krishnan had sounded a note of warning against the extended cultivation and use of tapioca as a staple food unless the diet could be supplemented by proteins of high biological value such as those from milk, fish, meat and egg. Such supplements being beyond the means of a large section of the population, the striking effect produced

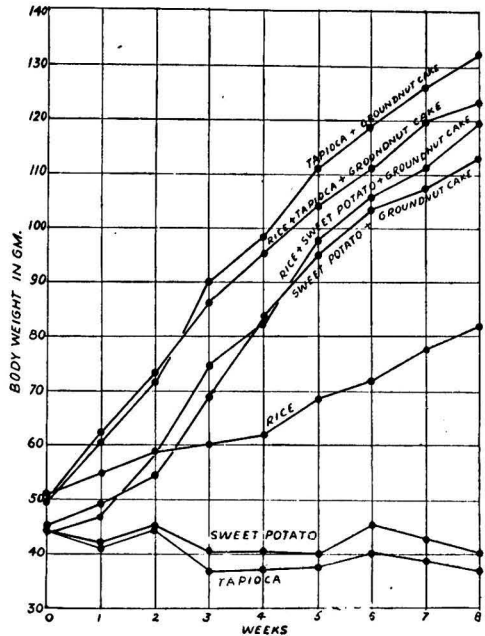


FIG. 1 — INCREASE IN BODY WEIGHT OF RATS ON DIFFERENT DIETS.

TABLE III — AVERAGE WEEKLY INCREASE IN BODY WEIGHT OF YOUNG RATS ON DIFFERENT DIETS (DURING 8 WEEKS)

DIET No.	CHIEF INGREDIENT IN THE DIET	AVERAGE WEEKLY INCREASE IN BODY WEIGHT, GM.
1	Tapioca flour	The animals lost weight at an average of 0.5 gm. per week (3 died in 8 weeks)
2	Sweet potato flour	The animals lost weight at an average of 0.8 gm. per week (3 died in 8 weeks)
3	Tapioca plus groundnut cake flour (4:1)	10.3 ± 0.45
4	Sweet potato plus groundnut cake flour (4:1)	8.6 ± 0.58
5	Raw milled rice	3.9 ± 0.2
6	Raw milled rice plus tapioca plus groundnut cake flour	9.2 ± 0.37
7	Raw milled rice plus sweet potato flour plus groundnut cake flour	9.4 ± 0.54

by a cheap material like groundnut cake would come as a welcome relief. It is possible that, besides groundnut cake, there are also quite a number of other food materials, equally cheap and abundant, which could adequately supplement the poor diets based mainly on tubers. Further work in this direction is in progress.

Summary

1. Young rats fed on a diet largely composed of tapioca or sweet potato flour and containing, in addition, pulses, vegetables, etc., in amounts corresponding to the usual consumption of poor rice-eaters in South

India, lost body weight. Some of the animals died within 8 weeks.

2. Addition of groundnut cake flour to the above diet proved an effective supplement, promoting good growth, the average weekly increase in body weight being 10.3 gm. and 8.6 gm. respectively, as against 3.9 gm. obtained with poor rice diet.

3. Animals fed on a poor rice diet in which 25 per cent of rice have been replaced by a mixture of 4 parts of tapioca or sweet potato flour and 1 part of groundnut

cake flour also grew well, showing thereby that the nutritive value of poor rice diet was improved by replacing some of the rice by the above mixture.

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Investigation on the Chemical Composition of Triplite

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The formula $(Fe, Mn)_2PO_4.F$ has been assigned to the mineral triplite on the basis of quantitative chemical analysis.

TRIPLITE occurs in quartz veins of granites accompanied by apatite at Limoges in France. It is also found in masses in quartz with beryl, apatite, columbite at Sierra de Cordoba, Argentina. The Indian mineral under investigation was collected from a mica mine, 2 miles south-east of Singar, Gaya district, Bihar. The mineral was deep brown in colour with a resinous lustre, its streak was brown, hardness, 4-5 and specific gravity, 3.7.

Experimental

The mineral was crushed to small pieces and a selected portion was powdered to pass through 200 mesh. The analytical procedure is given in the following scheme.

For the estimation of ferrous iron, some of the crushed fragments of the mineral were decomposed by heating with hydrochloric and hydrofluoric acid in an atmosphere of CO_2 , cooled, boric acid added and the solution titrated with standard $K_2Cr_2O_7$ solution using diphenylamine sulphonate as internal indicator.

The mean values from a duplicate analysis are given below.

ANALYSIS OF TRIPLITE FROM GAYA

	PER CENT
P ₂ O ₅	30.60
FeO	29.20
Fe ₂ O ₃	2.30
MnO	28.55
SiO ₂	1.05
CaO	1.75
MgO	1.85
F	8.00
Th + R.E.	0.00
	103.80
O = F	3.40
Total	99.90

Chemical Formula

Rammelsberg calculated the formula of triplite as $R_2P_2O_8.RF_2$ or $(RF)RPO_4$, where R represents Fe, Mn, also Ca and Mg. The ratio Fe : Mn varies widely in different samples of the mineral from different localities, viz. Fe : Mn = 1 : 1, 2 : 1, 1 : 2 and even 1 : 7. On calculation from the analytical data obtained by us we arrived at the formula $(Fe, Mn)_2PO_4.F$, where the ratio of Fe (Ca) : Mn (Mg) = 1 : 1.

Our thanks are due to Prof. P. B. Sarkar for his keen interest in the work and to the Council of Scientific & Industrial Research for financial help. We are also thankful to the Director, Geological Survey of India, for supplying the mineral.

AN OUTLINE OF THE SCHEME OF ANALYSIS

1 gm. of the mineral fused with 10 gm. Na_2CO_3 (avoiding blast). Extracted the melt with hot water. Washed with 2 per cent Na_2CO_3

F_1 : Dissolved in HCl. Twice evaporated to dryness with HCl, taken up with dilute HCl. Filtered and washed

F_1 : Added 10 gm. $(\text{NH}_4)_2\text{CO}_3$, let stand at 40°C . overnight. Filtered, washed with $(\text{NH}_4)_2\text{CO}_3$ solution

$P_2 + P_4$: Ignited and weighed. Treated with HF and H_2SO_4 , ignited and weighed.
Loss = SiO_2 .
Residue fused with sodium bisulphate, took up with HCl and combined with filtrate (8)

F_8 : Passed H_2S . Filtered. Rejected precipitate of platinum sulphide. Treated the filtrate with $(\text{NH}_4)_2\text{S}$. Filtered. Precipitate dissolved in HCl and the precipitation repeated. Filtered and washed with ammoniacal H_2S water

P_3 : Dissolved in HCl + HNO_3 . Treated with NH_4Cl , NH_4OH . The precipitation repeated. Filtered and washed

F_9 : Acidified with HCl, boiled off H_2S , made slightly ammoniacal and Ca precipitated as oxalate

F_{11} : Dissolved in HCl, acidity adjusted to N/2, saturated with oxalic acid; no precipitate obtained. Added tartaric acid made ammoniacal, passed H_2S

F_{11} : Evaporated to fumes with H_2SO_4 and determined Mn volumetrically with sodium bismuthate

P_{10} : Determined Ca volumetrically

F_{10} : Mg. precipitated as MgNH_4PO_4 and determined volumetrically

P_2 : Added to P_3

$P_2 + P_3$: Evaporated to dryness with HCl, taken up with dilute HCl. Filtered and washed with water. The process repeated

F_3 : Added a few drops of phenolphthalein, cautiously neutralized with HNO_3 till the red colour disappeared. Added AgNO_3 solution in excess, heated slightly. Filtered and washed with minimum quantity of cold water

P_5 : Combined with R_2

F_6 : Combined with the solution of P_6 in HNO_3 . Precipitated with HCl. Filtered and washed

F_4 : Added NaCl warmed, filtered and washed with a little water

P_4 : Dissolved in dilute HNO_3 and added to F_6

P_7 : Rejected

F_7 : In an aliquot phosphoric acid was precipitated as ammonium phosphomolybdate and finally determined as $\text{Mg}_3\text{P}_2\text{O}_7$. In another portion Al was found to be absent

P_5 : Rejected

F_5 : Determined fluorine as PbClF

P_{12} : Dissolved in HCl and determined Fe volumetrically with $\text{K}_2\text{Cr}_2\text{O}_7$

F_{12} : Rejected

Study of Mullite Crystals in Porcelain Bodies

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The formation of mullite crystals in a porcelain body fabricated by different methods has been investigated. Slow firing and cooling favour the formation of mullite in larger crystals. Articles prepared by pressing and casting contain more mullite than those obtained by extrusion.

MANY properties of porcelain have been attributed to the presence of mullite crystals formed in the porcelain body during firing. Porcelain bodies containing mullite show good mechanical strength and refractory properties, possess a low and uniform rate of thermal expansion, and withstand high stresses at elevated temperatures and voltages.

Mullite is always present in a porcelain body in varying quantities, either in a crystalline or other form. It is an aluminium silicate ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) containing 71.8 per cent alumina and 28.2 per cent silica by weight.

The formation of mullite crystals in the ceramic body, both in size and quantity, is dependent on (1) the nature of raw materials, (2) body composition, (3) particle size of the ingredients, (4) homogeneity and compactness of the mixture and (5) the thermal treatment during manufacture. Oxides of chromium, boron, copper, manganese, magnesium, calcium and iron, and talc are known to catalyse the formation of mullite¹⁻⁴. Hydro-thermal synthesis also helps in the formation of mullite⁵.

The present investigation was undertaken to investigate the following aspects of mullite formation: (1) quantity of mullite formed in porcelain bodies by casting, dry-pressing and extrusion, (2) effect of different temperatures, (3) growth and distribution of mullite, (4) effect of firing period, (5) influence of cooling period and (6) mullite formation in specimens of varying thicknesses.

Experimental

The test articles were fabricated by casting, extrusion and dry-pressing from the same mix. The body was prepared from a mixture of kaolin, feldspar and quartz in the proportion of 50:25:25, by weight. The raw materials (which were obtained from Czechoslovakia) had the following chemical composition (TABLE I).

TABLE I

	KAOLIN*	FELDSPAR †	QUARTZ ‡
	%	%	%
SiO ₂	46.50	65.15	99.80
Al ₂ O ₃	39.00	18.91	...
Fe ₂ O ₃	0.50	0.145	0.02
CaO	0.60	1.765	...
TiO ₂	0.15
MgO	...	0.235	...
K ₂ O	0.30	11.92	...
Na ₂ O	0.30	2.31	...
Loss on ignition	13.00

*Sedlic. †Krupka. ‡Jilove (Czechoslovakia).

The following is the Seger formula for the body composition:

RO : R₁O₂ : RO₂ is

Na ₂ O — 0.0039	Al ₂ O ₃ — 0.9925	SiO ₂ — 4.4780
K ₂ O — 0.1385	Fe ₂ O ₃ — 0.0075	TiO ₂ — 0.0038
CaO — 0.0551		
MgO — 0.0063		

Feldspar and quartz were first washed clean and calcined in an electric furnace at 800°C. Requisite quantities of feldspar and quartz needed for the body mix of 2 kg. were ground in a pot-mill for 25 hr., the required amount of kaolin added, and ground for another 2 hr. The slurry obtained was passed through a ferro-filter, then through a 900 mesh/sq. cm. sieve and finally filtered on a Buchner funnel. A portion of the material was dried in an air oven at 105°C. and used for dry-pressing; another portion was partially dried (c. 25 per cent moisture) for extrusion; from the remainder a casting slip was prepared.

Articles of different thicknesses (10 mm. and 1.9 mm.) were prepared. The samples were first dried in air for 24 hr. and then in an air oven at 90°C. for another 24 hr. before firing. The different test samples were fired together in the same furnace during each firing.

The firing schedules employed are given in Table II.

TABLE II

BURNING	COOLING
a. Gas-fired muffle up to 1,400°C. in 24 hr.	Cooled down to 1,200°C. in the same furnace in 7.5 hr. and then quenched in cold water
b. do	Cooled to 825°C. as above in 15.5 hr., then in the same furnace to room temperature in 22 hr.
c. do	Cooled to 825°C. in 15.5 hr., then annealed in an electric kiln and cooled down to room temperature in 34 hr.
d. Burnt in an electric muffle up to 1,300°C. in 8 hr.	Cooled in the same kiln in 15 hr. without any control
e. Burnt in the gas-fired muffle up to 1,400°C. in 36 hr.	Cooled in the same furnace in 26 hr. without any control
do	Cooled in the same furnace to 880°C. in 24 hr., then in gas-fired furnace to room temperature in about 81 hr.
g. Burnt in an electric furnace up to 1,580°C. in 8 hr.	Cooled in about 15 hr. without any control
h. Burnt in the gas-fired muffle up to 1,400°C. in 10.5 hr.	Cooled in the same furnace for 26 hr. without any control

Firings (a), (b) and (c) were carried out in one operation and, during cooling, samples were taken out at 1,200°C. and 825°C. for further cooling as in firings (a) and (c) respectively. Firings (e) and (f) were made separately. Firing schedules (d) (g) were carried out in an electric resistance kiln. In the gas-fired furnace, the firing (h) was first carried out in an oxidizing atmosphere up to 900°C., then in a reducing atmosphere up to 1,200°C. and finally in a slightly reducing atmosphere up to 1,400°C. Only oxidizing atmosphere was employed in the electric kiln.

The test samples prepared according to the different processes and firing schedules are given in Table III.

Testing — The mullite formed in the samples (excepting 20, 21) was quantitatively estimated by the hydrofluoric acid method¹. Thin sections of the samples were examined under the microscope and the growth and distribution characteristics of



FIG. 1 — PHOTOMICROGRAPH OF SAMPLE NO. 8.

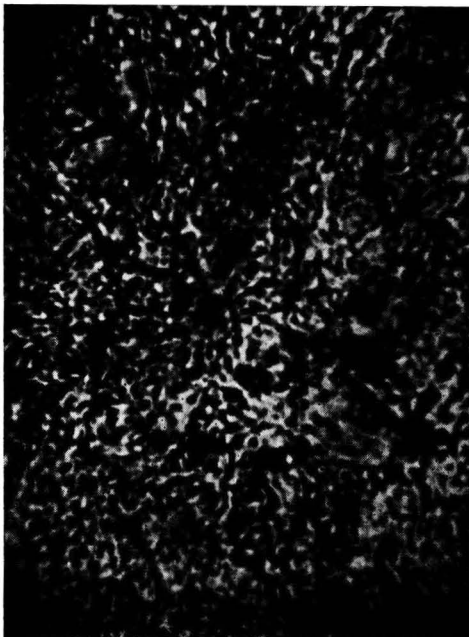


FIG. 2 — PHOTOMICROGRAPH OF SAMPLE NO. 14.

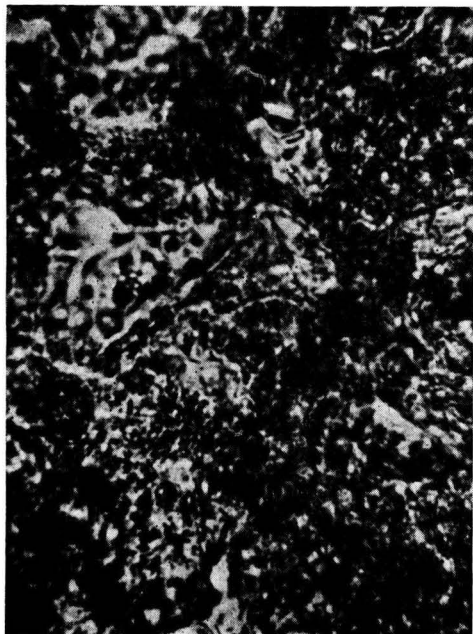


FIG. 3 — PHOTOMICROGRAPH OF SAMPLE No. 15.

the mullite crystals noted. The crystals could be identified only under high magnification through an oil immersion lense. The amounts of mullite found in the samples are given in Table III.

Photomicrographs (X 820) of a few test specimens are reproduced in Figs. 1-11.

Discussion

Slow cooling of fired articles to room temperature gives a maximum yield of mullite, whereas slow cooling up to 825°C. only gives a lower value (TABLE III). The data relating to samples 3 and 6 reveal that slower firing and cooling than that of schedule (c) does not enhance the mullite content; on the contrary, the values are lower, thereby demonstrating that a slower firing schedule than 24 hr. and a cooling schedule of 50 hr. are not necessary for ensuring optimum mullite formation. Sample 5 gave a much lower yield of mullite than sample 6, thereby emphasizing the need for slow cooling. The data for samples 4, 7 and 8 indicate that low firing temperatures produce little mullite; they also show

TABLE III — AMOUNT OF MULLITE IN DIFFERENT SAMPLES

Sr. No.	DESCRIPTION OF SAMPLES	WT. OF SAMPLE TAKEN gm.	WT. OF MULLITE FOUND gm.	CORRECTED WT. OF MULLITE gm.	PERCENTAGE OF MULLITE IN THE MASS
1	This was a portion of thin crucible, made by casting and fired according to the firing (a)	0·4158	0·0818	0·0986	23·71
2	Same as above but fired as per (b)	0·4037	0·0838	0·1010	25·02
3	Same as above but fired as per (c)	0·3907	0·0821	0·0990	25·33
4	Same as above but fired as per (d)	0·4177	0·0360	0·0434	10·39
5	Same as above but fired as per (e)	0·4023	0·0727	0·0876	21·77
6	Same as above but fired as per (f)	0·4033	0·0837	0·1009	25·02
7	This was made by dry-pressing and fired as per (g); the sample was partially melted and became porous	0·4037	0·0625	0·0786	19·47
8	Same as above but fired as per (h) and was not melted	0·4033	0·0754	0·0909	22·53
9	This was made by extrusion and fired as per (c)	0·3957	0·0810	0·0998	24·66
10	Same as above but fired as per (d)	0·4004	0·0195	0·0235	5·86
11	Same as above but fired as per (e)	0·4030	0·0705	0·0849	21·06
12	Same as above but fired as per (f)	0·4033	0·0827	0·0997	24·72
13	This was made by pressing and fired as per (c)	0·4020	0·0831	0·1001	24·90
14	Same as above but fired as per (d)	0·4036	0·0213	0·0257	6·36
15	Same as above but fired as per (e)	0·4010	0·0745	0·0898	22·39
16	Same as above but fired as per (f)	0·4040	0·0842	0·1015	25·12
17	This was made by casting and of about 5 times the thickness of the thin crucible and fired as per (c)	0·4046	0·0843	0·1016	25·11
18	Same as above but fired as per (e)	0·4034	0·0726	0·0875	21·69
19	Same as above but fired as per (f)	0·4046	0·0826	0·0995	24·95
20	Same as No. 14 of this table but fired as per (a)
21	Same as above but fired as per (b)



FIG. 4 — PHOTOMICROGRAPH OF SAMPLE NO. 6.



FIG. 6 — PHOTOMICROGRAPH OF SAMPLE NO. 13.

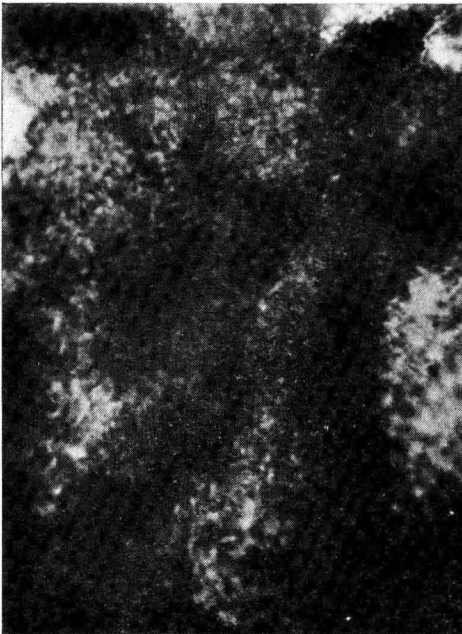


FIG. 5 — PHOTOMICROGRAPH OF SAMPLE NO. 12.



FIG. 7 — PHOTOMICROGRAPH OF SAMPLE NO. 16.

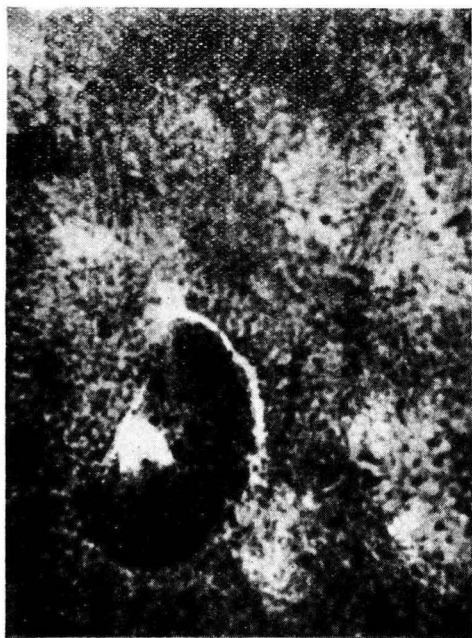


FIG. 8 — PHOTOMICROGRAPH OF SAMPLE No. 19.

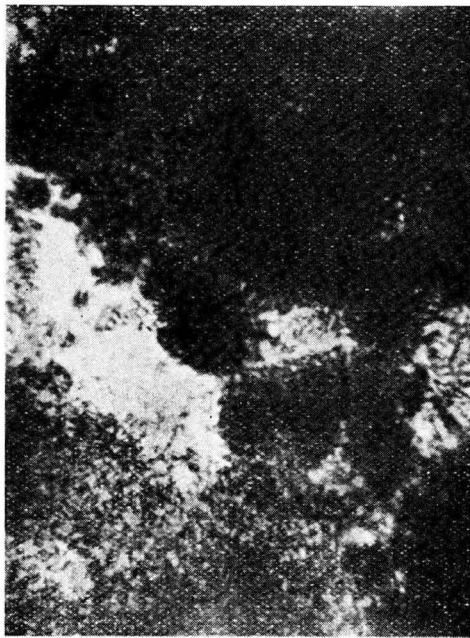


FIG. 10 — PHOTOMICROGRAPH OF SAMPLE No. 21.



FIG. 9 — PHOTOMICROGRAPH OF SAMPLE No. 20.



FIG. 11 — PHOTOMICROGRAPH OF SAMPLE No. 7.

that higher temperatures alone do not produce more mullite.

These conclusions are more clearly brought out in Tables IV and V.

TABLE IV

FIRING NO.	BURNING PROCESS	COOLING PROCESS	PERCENTAGE OF MULLITE		
			Casting	Pressing	Extrusion
3	slow	slow	25.33	24.9	24.66
5	slow	quick	21.77	22.39	21.06
6	slow	slow	25.02	25.12	24.72
8	quick	quick	...	22.53	...

TABLE V

METHOD OF MAKING	PERCENTAGE OF MULLITE		
	1,300°C	1,400°C	1,580°C
Casting	10.39	21.77	...
Pressing	6.36	22.39	19.47
Extrusion	5.86	21.06	...

Table IV shows that if cooling is quickly effected slow burning does not favour mullite formation. This shows that the mullite formed remains in solution, and recrystallizes out on slow cooling. At low temperatures such as in (d), thin castings give optimum yields of mullite. Very slow firing as in (e) and (f) produces a maximum yield of mullite with pressed specimens. Articles produced by extrusion always give low yields. Pressing and casting are presumably better than extrusion for producing porcelain ware. Microscopic examination of thin sections reveals that wherever undissolved quartz occurs in the body, mullite needles are absent. Figs. 1, 2 and 3, which relate to samples obtained by quick cooling, show undissolved quartz; mullite crystals are conspicuously absent. Dissolution of quartz during cooling influences mullite formation. Slow cooling up to 1,200°C. helps to dissolve the quartz

(FIG. 9). Quartz is absent in Fig. 11; the sample was cooled quickly. This is probably due to the high firing temperature employed. Small mullite needles are observed in specimen 15 (FIG. 3) subjected to slow heating, though it was not cooled slowly, showing thereby that slow firing helps mullite formation.

Samples 6, 12, 13, 16, 19, 20 and 21 (Figs. 4-10) show marked similarity in structure. All of them show a ground of molten glass in which medium-sized mullite needles are distributed. These specimens were burnt and cooled slowly. Sample 7 (FIG. 11) shows a good distribution of big mullite needles. This sample was fired at the highest temperature employed in the series and was practically melted. The low viscosity of the molten mass may have been responsible for the growth of long needles, though the sample was fired and cooled quickly.

Conclusions

1. The formation of mullite crystals in a porcelain body is favoured by slow firing (24 hr.) and slow cooling (54 hr.).
2. The size of mullite crystals increases with the temperature of firing.
3. High temperatures are not essential for the maximum formation of mullite.
4. Articles obtained by pressing and casting contain more mullite than those obtained by extrusion.

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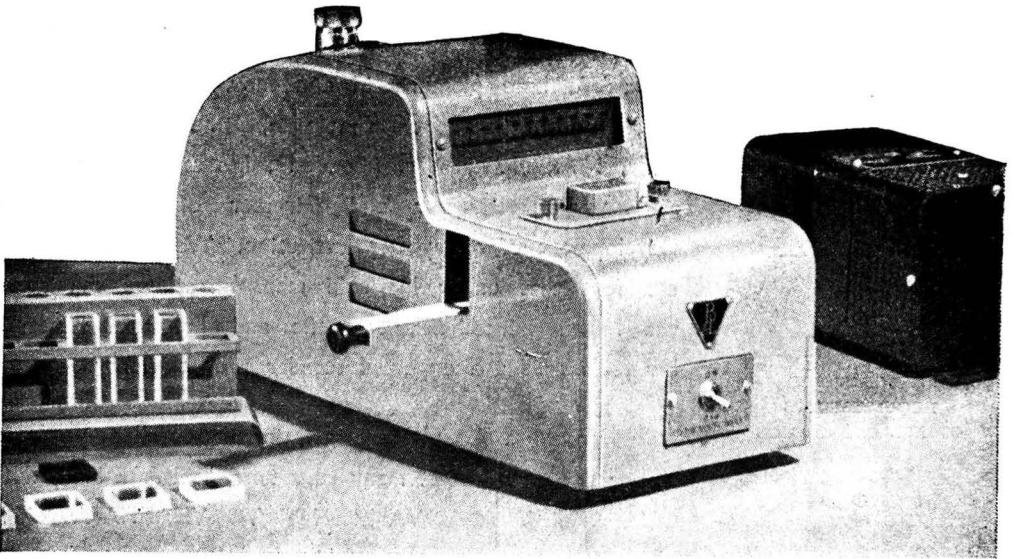
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