# Journal of Scientific & Industrial Research



V. 9, No. 9, SEPTEMBER 1950

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### COVER PICTURE

The picture on the cover shows the Radar arrangement with five Yagi arrays (*see* page 312) set up at the Meteorological Department's Observatories at New Delhi and Poona for obtaining meteorological data.

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# The Indian Salt Industry

**THE Report of the Committee of Salt** Experts constituted by the Government of India in April 1948, which has now become available, deals comprehensively with the present position of the Indian salt industry and discusses the directions in which the industry should be developed so that it may attain, within a short time, the status it is entitled to by virtue of its importance as an article of food and as a basic raw material for the heavy chemical industry. With the abolition of the excise duty on salt, the present Salt Department of the Government of India has ceased to be a revenue-collecting organization; its functions have to be oriented to those of a development department. How should the Department be reorganized so that it may fulfil its new responsibilities? What are the problems of the salt industry which await investigation and research so that it may develop into its full stature? These are the questions which the Report discusses in a refreshingly realistic manner.

The Committee confirms the view generally held in knowledgeable circles that salt production in India can be increased within a short period, and that self-sufficiency with regard to this vital requirement of the population can be attained. Urgent as the improvement in quantitative production is, the need for raising the quality of salt and for recovering the valuable by-products of the salt industry is no less urgent and im-It must be emphasized that portant. common salt has the unique distinction of being the foundation material for the great modern chemical industry. The Leblanc process for making soda ash from common salt is regarded as the beginning of the inorganic chemical industry. Competent authorities aver that the electrolytic chlorine cell is the fundamental basis of the prodigious array of organic chemicals. The production, standardization and utilization of a raw material so basic to the chemical industry, and yet so abundant, should necessarily receive serious attention. The example of Dow Chemicals Ltd. illustrates the possibilities for the production of chemicals from the limitless deep, and thanks to the research programme consistently fostered by the enlightened founder of the organization, Dr. Herbert Henry Dow, a gigantic chemical industry based on common salt has That similar possibilities been built up. exist in India has been amply demonstrated by the pioneering efforts of the late Mr. Kapilram H. Vakil who planned and directed the salt works and the by-product plants at Mithapur. Such developments are, however, exceptional; the Indian salt industry remains, by and large, in a recumbent and primitive condition, and traditional rule-of-thumb methods, uninfluenced by modern technological developments, operate in salt production. The salt manufactured in many areas is sub-standard and that in some areas, such as Didwana, is unfit for human consumption. The industry is in dire need of technical aid, and the time has arrived for directing serious attention to improving the quality of Indian salt.

The industrial plants at Mithapur for the systematic recovery of magnesium chloride, magnesium sulphate, potassium chloride, bromine and, more recently, gypsum, provide an object lesson in the utilization of marine salts for the development of chemical industry. The unique resources of the landlocked lakes of Rajputana have so far received but scanty attention. These inland lakes represent vast chemical reservoirs

constantly replenished by streams flowing over salt-bearing beds. The spontaneous salt formed in the Rann of Kutch and distributed over the catchment area of the lakes by stormy winds is dissolved by rain water and carried to the lakes whose salt contents are thus continuously enriched. The resources of some of these lakes are inexhaustible. Like the Salt Lakes of California and Nebraska, the Great Salt Lake of Utah and the Dead Sea, the Rajputana desert lakes provide resources which await exploration and planned exploitation. Climatic conditions prevailing in the lake areas are conducive to the development of the industry. Low rainfall, long periods of dry weather, high winds and fairly high temperature provide ideal conditions for solar evaporation. The climate is a most valuable asset next only to the richness of the lakes. By supplying proper conditions for the crystallization of marine salts, it is possible to separate the valuable salts from those of lesser value and individual salts from their mixed solutions. The Desert Chemical Co. of California recovers from the saline water of the Dale Lake ( which is similar in many respects to the lake water of Didwana) not only common salt of high quality, but also the valuable by-product sodium sulphate. The Palestine Potash Ltd. has been exploiting the resources of the Dead Sea for the extraction of potassium salts and bromine. Science and technology provide the means for utilizing the natural resources of the inland salt lakes for building up chemical industries.

The waters of Sambhar and Didwana lakes are rich sources of sodium sulphate,

sodium carbonate and sodium bicarbonate. For every ton of common salt recovered from Didwana Lake, it is possible to recover  $\frac{1}{3}$  to  $\frac{1}{4}$  ton of sodium sulphate. This valuable material has many industrial applications among which may be mentioned the production of caustic soda and sulphuric acid by electrolysis. The recovery of all these by-products would not only help in building up a flourishing chemical industry but, at the same time, also improve the quality of salt produced and lower the costs of production.

The Indian salt industry poses many problems for solution. The Salt Research Committee of the Council of Scientific & Industrial Research, which is fully seized of their importance, has already initiated a few research projects of practical impor-tance. The Committee of Salt Experts has recommended the establishment of model factories with facilities for undertaking largescale development programmes in the more important salt-producing regions. This recommendation is opportune and must be welcomed. If this recommendation is accepted and brought into effect, new facilities would become available for the commercial utilization of the results of research and inquiry sponsored by the Council of Scientific & Industrial Research. Knowledge and means for acquiring more knowledge required for solving the problems of the salt industry exist. What is needed now for developing the industry is a determined effort by all concerned — scientists, technologists and industrialists-to put knowledge into action, to transform plans and visions into plants and processes.

# Characteristics of the Ionosphere over Calcutta (June 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 June 1950.

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



5 HOURS 54 MINUTES AHEAD OF G.M.T. FIG. 1 — JUNE 1950.



AT POINT OF REFLECTION. FIG. 2 — PREDICTED M.U.F. VIA F<sub>2</sub> LAYER DURING THE MONTH OF SEPTEMBER 1950.

ing values of penetration frequencies and heights.

Sporadic E ionization was found to occur frequently during evening and night and sometimes during the afternoon. Sporadic E occurred more frequently during night than during day in the present month. Region  $F_2$  ionization was found to remain steady up to midnight and during the evening there was a slight rise. The fall in ionization during night is very slow and the lowest value of  $f^{\circ}F_2$  reached (6-8 Mc./s.) is several times larger than the corresponding value (3-0 Mc./s.) during the last winter months.

1	TA	BLE I				TABLE	I - contd.		
MONTH &	DATE	Hours	<sup>f°</sup> E <sub>s</sub>	<sup>h</sup> E <sub>s</sub>	Month &	DATE	Hours	f° <sub>E</sub> s	<sup>h</sup> E <sub>s</sub>
IEAR			Mc.	Km.	1 EAK			Mc.	Km.
June 1950	1	00.00	6.00	120	June 1950	15	20.00	3.35	105
		01.00 02.00	$3 \cdot 70 \\ 3 \cdot 50$	$105 \\ 105$			22.00	4.00	120
						16	09.00	9.50	150
× .	2	18.00	3.30	90			17.00	4.15	135
		22.00	4.10	105			18.00	4.15	135
		23.00	5.00	120			21.00	3.00	120
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		20.00	3.60	135		20	03.00	4.50	120
		21.00	4.00	120			04.00	4.50	120
		22.00	3.20	105			18.00	4.50	120
	-						17.00	4.00	105
	7	17.00	3.70	105		×	18.00	4.05	120
		18.00	4.25	120			10.00	1 00	120
		19.00	8.00	135			01 00	0.05	105
		20.00	8.15	150		21	21.00	3.85	105
		21.00	6.20	150			22.00	3.85	90
		22.00	4.50	150			23.00	4.10	120
		23.00	4.00	135		10.00	100 M		
•	0	00 00	5.00	195		22	. 23.00	3.00	105
~	0	07.00	5.00	100					
		07.00	4.20	120		23	00.00	4.25	120
	0	01.00	5.00	195			04.00	3.00	105
	9	02.00	4.90	190			23.00	3.60	105
		02.00	* 20	120					
	14	20.00	4.15	120		24	00.00	4.05	105
	**	21.00	8.70	105		-1	01.00	3.00	120
		22.00	3.50	105			03.00	3.40	105
		22.00	0 50	100			00.00	0 20	100
					-				

# Electronic Meteorological Instruments in the India Meteorological Department

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THE techniques of electronics have found application in many branches of experimental science. In meteorology, the tendency to use these techniques has been growing rapidly and, in India, various instruments employing radio tubes are being designed or are being used for meteorological observations on the ground and in the upper air. Some of the instruments designed and in use by workers in agricultural meteorology at Poona have been described in a paper by Ramdas<sup>1</sup>. The present paper describes briefly a few of the other instruments designed or in use in the India Meteorological Department.

### The Radiosonde

The first instrument to be designed and constructed was the radiosonde to measure the pressure, temperature and humidity in the upper air. This consists of a meteorograph in which the elements which vary with pressure, temperature and humidity modulate a light single-valve wireless transmitter on a wavelength of about 4 metres. The wireless signals are received and recorded on the ground with special equipment and

the data evaluated. Two types of instruments are in use by the Department, one of which was developed at Delhi and employs a small clock-work rotating a special contacting arrangement to transmit the signals. This is in regular use at four observatories in India. The second type (FIG. 1), which was designed at Poona and is now in use at eight stations in India, employs a paper fan rotating during the ascent of the balloon and operating a different type of switching arrangement. These instruments have been described in some of the publications of the India Meteorological Department<sup>2,3</sup>. Many accessories required for their manufacture. calibration, etc., have been designed and constructed<sup>4,5</sup>. The ground equipment to receive and record the wireless signals from the F-type meteorograph sent up with the balloon is shown in Fig. 2.

### The Radar

A later and more recent application of electronic equipment for the measurement of meteorological elements has been made by using the radar. Knowledge of the speed and direction of the wind in the upper



FIG. 1 — F-TYPE METEOROGRAPH.



FIG. 2 — GROUND EQUIPMENT TO RECEIVE AND RECORD WIRELESS SIGNALS FROM THE F-TYPE METEOROGRAPH.

air is helpful for the forecasting of weather and this information is normally obtained by observing the movement of hydrogenfilled balloons with the help of theodolites. This method fails completely during bad weather, as the balloon cannot be followed when it enters the cloud. No information about winds will, therefore, be available above the cloud base. This difficulty has been overcome by the meteorologist by adapting the radar for this purpose. An artificial target is suspended from the hydrogen-filled balloon to reflect the energy from the radar transmitter and this enables the position of the balloon to be located in the sky. It is thus possible to obtain information about upper winds to great heights even above the cloud- base and at night when the usual theodolite method fails.

The India Meteorological Department has acquired a few radars which are being used regularly at the observatories at New Delhi and Poona. This radar (A.A. No. 3. Mk. III) is of the early type and works on a frequency of 204 mc. One Yagi array is used for transmission and four Yagi arrays are used for reception through a lobe switching arrangement (see Cover). The range is determined by an automatic range unit. The balloon is tracked both in elevation and azimuth by two observers and one observer keeps note of the range. Corner reflector type of targets are efficient at microwaves, but they are not so effective at 200 mc. and, therefore, crossed dipole are used, which absorb the targets radiation of the radar transmitter and re-radiate.

The method of determining upper winds with the radar by using a passive target, i.e. a target which does not originate a signal but merely reflects the incident energy, has some disadvantages. Usually, only shorter ranges are obtainable with radars. The greatest handicap is the ground clutter and other unwanted echoes which tend to mask the desired signal. This difficulty can be overcome if, instead of a passive target, a small wireless transmitter on 204 mc. is attached to the balloon, which can be followed with the receiving equipment of the radar. Equipment employing active targets is more expensive, if employed only for obtaining upper wind information. However, active targets are employed in all types of radio-meteorographs and, as such, it should be possible to track them with the receiving equipment of the radar for upper winds. Maximum advantages will be obtained from the radar if it can be used to follow both active or passive targets. When an active target like the radio-meteorograph

signaller is followed, the transmitter in the radar set will not be operated.

Earlier observations on the influence of the troposphere were found to be most marked for radio-wave trajectories near the surface of the earth, and they were mostly reports of unorthodox vision beyond the geometrical horizon, maximum ranges sometimes extending to even ten times the distance of the geometrical horizon. This started a new branch of study of propagation of these waves and how they are influenced by the meteorological conditions of the atmosphere. Some interesting observations regarding the behaviour of the radar waves on 204 mc., when employed to follow balloons carrying dipole targets, have been made at Poona. It is observed that in certain seasons and under certain temperature and humidity distributions, reflections from the balloon target are lost below certain angles of elevation. Detailed examination of this phenomenon is on hand.

Though the India Meteorological Department has, at present, the old type of radars operating on a wavelength of 1.5 metres, it is most likely that a few radars operating on much lower wavelengths, of the order of about 10 cm. or less, may be obtained in the near future. When radio waves are incident on rain drops, a part of the energy is absorbed and a part is scattered. Due to these processes, the incident beam is attenuated and this effect increases as the wavelength is reduced. The effects are not important on wavelengths of one metre and above, but become appreciable at wavelengths of 10 cm. and below. The scattering by water drops or precipitation in frozen form are observed as radar echoes, and the meteorologist is able to get from them an idea of the distances of the region of precipitation and also the distribution of rain in the weather phenomenon. Radars operating on such short wavelengths can be used both for tracking balloons with suitable targets for determining upper winds and in connection with storm detection and consequent improvements in short-range weather forecasting.

### Distant Reading Wind Instruments

With the increased use of aviation for civilian transport, the demand for meteorological reports from aerodromes have also increased considerably. The data have to be



FIG. 3 — THE DISTANT READING ELECTRONIC WIND-SPEED INDICATOR.

supplied both to the planes in the air and on the ground with the least delay. Different types of instruments have been designed to measure the velocity and direction of the surface winds from the room of the observer without going up to the anemometer or windvane which are usually located in places which are not easily accessible. The Instruments Section at Poona has developed a distant reading wind-speed indicator and a windvane using Selsyn motors<sup>6</sup> (FIG. 3). In the electronic windspeed indicator, a radio frequency voltage of 30 kc. is generated in the observer's room and fed through a pair of cables into a coil fixed in a box at the foot of the spindle of the 4-cup anemometer. Another coil is fixed just below the first and the voltage induced in this is fed to an amplifier in the observer's room through a pair of cables. A brass circular vane with ten sectors fixed on the anemometer spindle rotates in the space between the two coils and varies the mutual inductance between them. The induced voltage in the secondary coil is modulated by the rotation of the vane and the modulation frequency will be proportional to the rotation of the spindle, i.e. the velocity of the wind. The induced

voltage is amplified and the varying portion of the voltage, which is obtained after detection, is converted into square wave pulses of constant amplitude and then fed into a frequency discrimination circuit which indicates the frequency of the modulation by means of a microammeter, calibrated in miles per hour. The direction of the wind is obtained at a distance by coupling a Selsyn motor to the spindle on the vane.

### The Ceilometer

The knowledge of the height of low clouds is very important to the aviator. At night it is determined quickly by measuring the angle of elevation of the illuminated patch under the base of the cloud from a vertical beam of light from a searchlight situated at a known distance from the place of observation. However, this method cannot be used during the day. A photoelectric method of measuring cloud heights in the day time has been developed in the U.S.A. The light spot on the cloud cannot be seen in the day time as the brightness of the underside of the cloud is very much brighter than the spot. When a modulated light beam is used, and a photoelectric telescope which is sensitive only to the modulated beam and not to the steady light of the day is employed, the angle of elevation of the illuminated patch can be determined. It appears that the circuit we have already employed in the Poona electronic anemometer appears to be suitable for this purpose. Steps are being taken to design in our laboratories a ceilometer on the above lines which can be used to determine the height of the base of clouds both by day and by night.

### REFERENCES

- 1. RAMDAS, L. A. : J. sci. industr. Res., 1948, 7, 16.
- 2. MATHUR, L. S.: India Met. Deptt. Sci. Notes, 9, No. 112.
- 3. VENKITESHWARAN, S. P., THATTE, R. P. &
- KESAVAMURTHY, A.: ibid., 9, No. 113.
   VENKITESHWARAN, S. P.: ibid., 11, No. 134.
   VENKITESHWARAN, S. P., KESAVAMURTHY, A., HUDDAR, B. B. & GUPTA, B. K.: ibid., 11, No. 422 No. 133.
- 6. KESAVAMURTHY, A. & VENKITESHWARAN, S. P. : Under publication.

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NE of the outstanding achievements of chemical engineering in recent years which has revolutionized a number of industries and which has opened up new possibilities in other industries is the technique of fluidization. It is only in recent years that the importance of this technique is being more and more realized and a number of countries including India are engaged in intensive research on its applications. In this paper, an attempt has been made to present the salient features of fluidization, its applications to a few industries, and indicate the recent trends in its development.

The imparting of certain fluid characteristics or properties to solid particles is known as fluidization. When a stream of fluid is passed upwards through a mass of solid particles, reproducible changes in physical behaviour are observed which pass through successive stages as the velocity of the fluid is increased. When the velocity of fluid flowing up through a mass of solid particles is insufficient to lift or support any of the solid, the mass is called fixed or moving bed, depending on whether the solid is stationary or moving with respect to the With increasing fluid velocities, container. in the absence of chanelling, the pressure drop through the bed rises until it approaches the net effective weight of the solid per unit area, when the packing arrangement of the particles becomes more open so that the bed expands; with further slight increases in velocity, the particles are fully supported and the expanding bed becomes fluidized. Just at the point of fluidization the mass may form a quiescent fluidized bed. With liquid-solid systems, further increases in fluid velocity give a progressive separation of the particles which remain individually and uniformly dispersed in particulate fluidization. With many gas-solid systems, the bed expands to only a limited extent and a portion of the gas tends to accumulate in gas pockets or bubbles passing through the

fluidized bed in aggregative fluidization. In small tubes, the bubbles may grow to a size substantially filling the cross-section of the vessel resulting in slugging. Normally. however, these bubbles remain small with respect to the vessel and the mass becomes a turbulent fluidized bed. Such a turbulent bed can even be maintained above the freefalling velocity for the solid particles in a gas-solid system if the rate at which solids are fed is kept high enough. Finally, if the fluid velocity is still further increased or if the solid feed rate is too low, the surface of the fluidized bed disappears and the whole mass becomes a dispersed suspension.

The variation of pressure drop with increase in the velocity of fluid is shown in Fig. 1. It will be noticed that the pressure drop increases with increase in fluid velocity up to the fluidizing stage when it becomes fairly constant and remains independent of further increase in the velocity of fluid. In Fig. 2 are shown the relationship of bed height with fluid velocity and fractional voidage with fluid velocity.

When the solid particles are in turbulent fluidized state or in dispersed suspension, they flow through pipe lines and valves as easily as if they were liquids. They also respond to pressure heads. The turbulent motion of solid particles in the fluidized state also leads to homogeneous mixtures being formed rapidly ; or if suitable arrangements are made to measure the temperatures at different regions of the bed, it will be seen that equalization of temperature takes place throughout the bed. The freedom of movement which solid particles possess when in this state is analogous to the freedom of movement which distinguishes the molecules of a fluid from those of a solid. Hence the term fluidization has been given to the process.

The first outstanding application of fluidization technique was to the Winkler gas generator developed in Germany as long back as 1929. This was followed by its



Fig. 1 — Variation of pressure drop with increase in fluid velocity.

adoption to the catalytic cracking of petroleum in the United States; its success in this process led to a widespread recognition of the technique as a new method of handling solids. In recent years the technique has been applied to the synthesis of liquid fuels, gasification of coal, washing of coals, purification of town's gas from sulphur, conversion of sulphur dioxide to sulphur trioxide and the oxidation of naphthalene to phthalic anhydride and a score of other processes.

The salient features of the fluidization technique are as follows:

1. The fluidized bed of solid particles acts like a thermal fly-wheel transferring heat from one hot part of the system to the cooler part and so the control of temperature of the reaction is easier. This is due partly to the rapid motion of the solid particles but also due to the fact that powdered solid particles impart heat capacity to the fluid and guard against rapid temperature changes.

2. The rapid motion and frequent collisions of particles prevent the development of abnormal conditions in localized areas. In the usual static bed with a stream of fluid percolating through it, the reaction will have proceeded to a greater extent in the front layers than in the back. But in a fluidized bed, uniform conditions are very quickly established and relatively easy to maintain. Consequently, it is possible to operate with a much deeper bed, and this is a very important practical advantage that may have a deciding influence on the economic possibility of the process. 3. The fluidized bed of solid particles

3. The fluidized bed of solid particles resembles somewhat a boiling liquid; one characteristic which they have in common is a high rate of heat transfer. This is obviously advantageous where heat exchange is a primary object, but it is also a desirable feature when exothermic chemical reactions take place in the fluidized bed because it makes possible the maintenance of a high through-put without fear of temperature becoming excessive.

4. When a chemical reaction is taking place between a gas and a solid, the effect of increase in surface due to the solid particles being in a fluidized state, as compared to the static bed, will lead to a higher reaction rate. This is so because of the continual relative movement of the individual particles whose surfaces are kept fresh by collisions, and the streaming gas which reduces the film resistance between the reactants.

5. And finally, it offers an easy method of moving large quantities of solids from one part of a process to another and is adaptable to continuous as opposed to intermittent or batch operations.

As opposed to these main advantages, there are two outstanding disadvantages of the fluidization technique. The first is that the rapid mixing and turbulent motion of the particles leads to a greater degree of short-circuiting by the solid between its entrance and its exit than in the case of the conventional static or moving bed. It is, therefore, not possible to use this technique to achieve counter-current flow effect between solid and fluid.

The second objection applies to those processes in which the solid is undergoing change during a reaction, that is, in general, to the non-catalytic processes. Here a difficulty is encountered by the need of maintaining always a suitable gradation of particle size in the fluidizing column. The particle size will be reduced during the reaction. For example, in the gasification of caking coals, due to the agglomerating property, the coal particles will stick to one another resulting in the collapse of the fluidized bed. However, this may be overcome by including with the solids certain



FIG. 2 - RELATIONSHIP OF THE BED HEIGHT AND VOIDAGE WITH FLUID VELOCITY.

amount of inert diluent which, by keeping the particles apart, will reduce the agglomerating property.

The first large-scale application of fluidization technique was to the Winkler gas generator<sup>2</sup>. In pre-war Germany the demand for hydrogen for use in the synthesis of ammonia and liquid fuels was so great that it could not be met by errecting the conventional type of water gas generators as the large amount of coke needed by these plants was not available. One of the pressing problems was to obtain hydrogen or synthesis gas by using the brown coals, which were available in large quantities, as the starting material. This was solved by installing the Winkler gas generator.

The generator is shown in Fig. 3. The body of the producer is a tall steel cylinder, 65' high and 18' in dia.; a flat grate forms the bottom of the generator. Coal crushed to 3 mm. dia. is fed through the conical coal bunker through a screw conveyer to the top of the grate to a height of about 3' to 5'. A blast of oxygen and steam (if a water gas of a high calorific value is needed) or air (if producer gas alone is needed) is passed to the bottom of the fuel bed through the slots provided at the bottom of the grate. The blast fluidizes the fuel bed and gives it the appearance of a boiling liquid. The fluidized bed has a temperature of about 900°C. The large amount of space provided over the fuel bed is to facilitate the complete gasification of the carry-over fuel by introducing a stream of secondary oxygen.

The rating, that is the amount of fuel consumed per square foot of grate area, is nearly 1½ to 3 times the normal "blow" and "run" gas, that is, about 40 tons/hr. normal rating for a grate 18' in dia. producing 1,80,000 cu. ft. per hour of water gas. The percentage of oxygen in the blast is usually about 20 but it can be as high as 50. The ash tends to segregate by reason of agitation of the bed and is removed by a conveyer screw.

The next large-scale application of fluidization technique was to the catalytic cracking of petroleum oils whereby some of the high-boiling fractions of the oil are converted to the more valuable motor spirit components.

The original process for cracking of petroleum oils was a thermal process in which gas oil or paraffin oils were partly converted into light oils suitable for motor spirits by subjecting them to high temperature



FIG. 3 - THE WINKLER GENERATOR.



FIG. 4 — SCHEMATIC REPRESENTATION OF THE PLANT EMPLOYING FLUIDIZATION PROCESS FOR THE CATALYTIC CRACKING OF PETROLEUM OILS.

treatment. Later on it was found that the same objective could be achieved yielding a better quality product by passing the vapours of the oils over a suitable catalyst. In the Houdry process the catalyst comprising of activated natural clay or synthetic catalyst of the silicaalumina type was in pellet forms, kept over fixed beds, and the oil vapours were passed But the selective molecular over it. breakdown of oils by catalysis is accompanied by carbonaceous deposits on the surface of the catalyst. This reduces the efficiency or activity of the catalyst and would ultimately inactivate it completely. The successful operation of the catalytic cracking of petroleum oils, therefore, depended on the continuous regeneration of the spent catalyst. This problem was solved by the application of fluidization to the process. The process is schematically represented in Fig. 4. The catalyst in a fine state is entrained by oil vapours and in this condition is conveyed to the reactor. Here the catalyst is maintained in a fluidized state and in this condition the vapours come into better contact with the catalyst. The cracked hydrocarbons are separated from the top. The spent catalyst is drawn from the bottom, entrained by a stream of air and conveyed to the regenerator. Here the carbon on the catalyst is burnt and the regenerated catalyst is ready again to be entrained by a fresh stream of the oil vapours. Thus the cycle of operation is completed.

Although high concentrations of solid are maintained in the reactor and regenerator, the mixture is extremely turbulent and each particle is in rapid motion. It is found that the turbulence results in a very uniform temperature throughout the mass - even in the large-scale reactors the temperature variation between the top and the bottom of the bed is not more than 5°F. in the range 800° to 1,000°F. for the reactor and 1,000° to 1,200°F. for the regenerator. The temperatures are varied by changing the amount of heat applied to the oil vaporizer or by altering the amount of hot catalyst in circulation. The degree of cracking of oil vapours depends on the depth of the catalyst bed and on the ratio of the catalyst to the oil vapours, both of which are controllable. It will be appreciated that the fluidization technique has given considerable flexibility to the cracking operation, a quality which is very important from the

point of view of process adaptation. The fluidized bed in the reaction vessel may contain as much as 75 to 100 tons of catalyst in the reactor and 150 to 300 tons in the regenerator. The rate of flow in the different sections of the plant is controllable by sliding valves.

There are about 40 plants in the United States alone using the fluid-catalyst system for oil cracking and some have a capacity of 7,50,000 gal. per day.

In recent years the fluidization technique has been applied to the synthesis of liquid fuels by the Fischer-Tropsch synthesis. In this synthesis pure carbon monoxide and



FIG. 5 — TYPICAL CATALYST BOX USED IN THE FISCHER-TROPSCH SYNTHESIS PLANT IN GERMANY.

hydrogen in the ratio of one to two are passed over a suitable catalyst kept at a temperature of about 230°C, when the synthesis of liquid hydrocarbon takes place. Since the reaction is an exothermic one, unless suitable provision is made to remove the heat liberated, the reaction products will decompose into methane, carbon monoxide and other less useful products. One of the chemical engineering problems in-volved in the commercial exploitation of the reaction for the synthesis of liquid fuels is to devise a suitable method of dissipating the heat evolved during the reaction. The Germans solved this problem by passing the synthesis gas through a series of boxes packed with a suitable catalyst, in which there was arrangement for circulating water through coils. Fig. 5 shows a typical catalyst box used in Germany. By varying the amount of water in circulation through the coils the reaction was controlled. One catalyst box alone weighed about 47 tons when empty and each box contained about 3 tons of catalyst. In a plant producing 1,80,000 gal. of liquid fuel per day there were some 120 similar catalyst boxes. Despite such elaborate set-up occupying a large area, the conversion of synthesis gas to liquid fuel was only 30 to 40 per cent. The motor spirit that was obtained was also of a very poor quality having an octane number of only 40.

In recent years the Americans have applied the fluidization technique to the Fischer-Tropsch synthesis. In the process they have developed the catalyst is kept



FIG. 6 — FLOW-SHEET OF THE EXPERIMENTAL UNIT SET UP AT THE FUEL RESEARCH STATION, GREENWICH, ENGLAND, FOR FISCHER-TROPSCH SYN-THESIS.



Fig. 7 — The lower portion of the reaction tube in the experimental Fischer-Tropsch synthesis unit at the Fuel Research Station, Greenwich, England.

in a fluidized state by the synthesis gas. Unfortunately no detailed account of the plants is available. According to the little information available, the Kellogg "Synthol" process<sup>4,5</sup> is capable of producing 75 ASTM octane number (83 Research) gasoline for about 5 cents per gal., using natural gas at 5 cents per 1,000 cu. ft. and figuring plant depreciation at 10 per cent per year. According to the Kellogg claims, by keeping an iron catalyst in fluidized state the major product which can be leaded to 80 ASTM octane number by addition of tetraethyllead can be obtained.

Hall<sup>6</sup> at the Fuel Research Station, Greenwich, has been using the fluidization technique in his experiments on Fischer-Tropsch synthesis. The experimental set-up that he adopted is shown in Fig. 6. The reaction vessel employed consisted of a 3.05 metre length steel tube, 2.5 cm. internal dia., fitted with a conical gas inlet at the lower end and with a 30.5 by 7.6 cm. disengaging column at the top to allow entrained catalyst to separate from the gas



FIG. 8 — FLOW-SHEET OF THE HEAVY MEDIA SEPARATION UNIT WHICH IS BEING INSTALLED AT THE CENTRAL RESEARCH LABORATORIES, HYDERABAD (DECCAN).

stream. A central tube, 0.8 cm. external dia., served as a thermocouple pocket and as a support for grids cut from steel gauge (4 mesh/cm.) which were made slightly convex to the gas flow and spaced at 5 cm. interval up the tube.

The reaction tube and the disengaging column were heated electrically, no special heat-dissipating means (aluminium block or liquid jacket) being employed. The lower portion of the reaction tube is shown in Fig. 7.

Another recent application of fluidization technique to exothermic reaction that may be mentioned is the oxidation of naphthalene to phthalic anhydride<sup>7</sup>. Naphthalene is oxidized by air at  $340^{\circ}$  to  $400^{\circ}$ C. in the presence of vanadium peroxide as catalyst. There is a considerable evolution of heat during the reaction — 10,000 B.Th.U./lb. of naphthalene converted — and the advantage that fluidization offers in the dissipation of heat is one of its main advantages. The vaporized naphthalene is mixed with air and flows into a chamber, 5' dia. by 30' high, where it fluidizes the catalyst bed and is converted to phthalic anhydride. In this case the catalyst is not inactivated by any of the reaction products and so no regenerator is required. The product gases, after leaving the dense catalyst phase, pass through a settling zone and cyclone separators to remove entrained catalyst and then to condensers which liquefy the phthalic anhydride. The through-put is 400 lb. of naphthalene per hour and the product is 99 per cent pure which is better than 96 to 98 per cent purity obtained in the conventional processes.

The fluidization technique has been applied to the washing of coal on the sink and float principle in the heavy media separation IYENGAR : FLUIDIZATION - A NEW TECHNIQUE



FIG. 9 - FLOW-SHEET OF A SEMI-TECHNICAL MOVING BURDEN WATER GAS PLANT.

unit developed by the American Cynamide Corporation<sup>8</sup>. The installation of an experimental plant of 500 lb. capacity is being completed at the Central Laboratories, Hyderabad (Dn.). The flow diagram of the unit is shown in Fig. 8. In the unit, finely ground, low cost magnetite (sp. gr. 5.0 to 5.2) is fluidized by a stream of water furnishing a medium that very closely duplicates a true heavy liquid as regards fluidity and stability in a range of gravities from 1.25 to 2.50. Coal of size coarser than  $\frac{3}{32}$ " is fed from sizing screen (1) to the separatory cone (2) through which is flowing the fluidized magnetite. The clean coal separates out at the top and is removed to the draining screen (4) where it is separated from the medium. The impurities of coal sink to the bottom of the medium in the separatory cone, entrained by a stream of air and lifted up to the draining screen where the medium is recovered from the impurities. In the recovery and control of concentration of the medium, advantage is taken of the magnetic property of magnetite.

In the heavy media separation unit, coals of as low a specific gravity as 1.25 and as high a specific gravity as 2.5 can efficiently be treated.

The fluidization technique has been applied in recent years to a number of processes. Dr. Garside, at the University of Leeds, has developed a novel method of purifying town's gas from sulphur. Prof. Mertens, at the University of Louvain, Belgium, has developed an interesting method of converting sulphur dioxide to sulphur trioxide. It is not possible in this paper to go into details about all these processes. However, it may be interesting to describe briefly the pilot plant developed by the I.C.I. for carbonization and gasification of non-caking coals<sup>11</sup> to indicate the recent trends in the development of fluidization technique. The plant is represented diagrammatically in Fig. 9. Coal, as particles of 1.0 mm. dia. or less according to design, is carbonized by direct contact with hot particles in the gas space of the vessel A by injecting it through (1) into a regulated stream of hot burden (coke and ash at 1,100°C.) flowing from the hopper D through valve (2); the bed in this carbonization vessel is fluidized by the carbonization gas produced (3), part of which is recycled through (4) after cooling, cleaning and reheating. The bed level is automatically maintained by a governor tube (5) from the hopper. The burden containing freshly carbonized coal is transferred by a downcomer (6) and a stream of hot nitrogen or blow gas (7) to the combustion tube C, in which it is partially burnt, to raise its temperature to 1,100°C., while being entrained into the hopper D by air (8) preheated to 500°C.; the blow gas produced is drawn off at (9). Water gas production takes place in vessel B, hot

burden from D flowing into B via the governor tube (10) which regulates the level of the bed through which steam is blown (11), the water gas and undecomposed steam leaving by (12). As on the carbonization side, the burden is taken from B by the downcomer (13) and an inert gas stream (14) to the combustion stage where it combines with the carbonization burden circulation. The dimensions of the vessel B, the steam rate and the rate of circulation of the burden are such that the gas contact time is adequate (a few seconds) for economic steam decomposition and that the steam rate will "boil" the bed and achieve good gas-solid distribution. The process as a whole, therefore, consists of two continuous circulations of burden for the carbonization and steaming stages, with a common combustion stage in which the heat required for the endothermic processes is restored by partial combustion of the burden.

It will be seen from the diagram (FIG. 9) that there are many problems to be solved before full-scale process plant can be designed - problems in carbonization conditions, dust and tar removal, control of heat of reaction in blow vessel to prevent slugging, control of flow of powders, design of heat exchangers, etc. The I.C.I. have studied this process on a laboratory scale and on a semi-technical scale. Many of the snags in the detailed operating conditions have been overcome and progress has been sufficiently satisfactory to justify a small pilot plant. This will have a capacity of 500 cu. m./hr. of water gas (about 300 c.f.m.).

### REFERENCES

- 1. MURPHY, W. J.: Ind. Eng. Chem., 1949, 41, 1249.
- 2. WALTER, J. F.: Inst. Petrol. J., 1946, 32, 295.
- 3. BIOS Report No. C 30/364. 4. ROBERTS, G. & PHINNEY, J. A.: Oil Gas J., 1947, 46, 139. 5. КЕІТН, Р. С. : ibid., 1946, 45, 102. 6. HALL, С. С. : "Industrial Chemistry" (1949).

- 7. LEE, J. A. : Chem. & Met. Eng., 1945, 52, 100.
- "Mineral Dressing Notes", American Cynamide 8. Co., Report No. 16, Feb. 1948. 9. KING, J. G. : Inst. Gas Eng., Report No. 315/136.

- 10. BOYLE, J. L. : Int. Ind., April 1948, 181. 11. TOWNEND, D. T. A. : William Young Memorial Lecture, 1947.

# Manufacture of Zinc Oxide from Zinc Sulphide Flotation Concentrates—Literature Report No. 11

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**THE most important use of zinc oxide** is in the pigment industry although it has various other uses also such as in the ceramic and the rubber industries. For white paints, pure zinc oxide is used but for certain other grades, leaded zinc oxide is used. The physical and the chemical characteristics of the oxide particles are of importance, e.g. for paints; acicular or needle-shaped particles (anisotropic) are desirable for giving superior weathering properties. For use in the rubber industry, uniformly sized round (isotropic) particles measuring about 0.22 microns are preferred. This indicates that the process by which the oxide is manufactured has a considerable influence over the quality of the product and, consequently, its suitability for various uses.

In this article, the various methods of manufacture of zinc oxide, as practised in other countries, are briefly described.

In India, zinc oxide is manufactured from imported spelter. This is an indirect method of producing the oxide and is naturally very costly. India has been adopting this process for obvious reasons, because zinc ore was not produced till now in this country. The Metal Corporation of India Ltd., which operates a lead-zinc mine at Udaipur, are shortly expected to bring their ore-dressing plant into production stage when about 12-15 tons of zinc sulphide concentrates will be produced daily. Till a reduction plant is put up in India to utilize these concentrates for production of spelter, perhaps the best possible outlet for these flotation concentrates will be for the manufacture of zinc oxide. This will also eliminate imports of spelter into India for this purpose.

Comparative merits and demerits of various processes have been discussed and their suitability for adoption in India is suggested.

### Manufacture

The three processes employed for the manufacture of zinc oxide are:

1. The "Direct Process" which produces the oxide direct from the sulphide ore or sulphide concentrates. This is achieved by reducing zinc sulphide by means of carbonaceous fuel and immediately burning the evolved zinc vapour to oxide. This process is also known as the "American Process",

2. The alternate method of producing pigment zinc oxide is the "Indirect Process", also known as the "French Process". The raw material is metallic zinc which is volatilized and burnt to oxide.

3. The "Wet Process" precipitates the carbonate or hydrate from zinc-bearing liquors and then calcines the precipitate.

1. Direct Process — Following are the different methods adopted at various places for the manufacture of oxide from ore or sulphide concentrates:

- (a) The Wetherill-grate furnace method
- (b) The travelling-grate furnace method
- (c) The Waelz process
- (d) The electrothermic process
- (e) The electric process
- (f) The flash-fume process

(a) Wetherill Furnace Method<sup>1,2</sup> — Two types of furnaces, called the Eastern and the Western Wetherill furnaces, are used. The essential difference between the two types is that in the Eastern type, zinc fumes from various furnaces are led to a system of combustion chambers for oxidation, whereas in the other type of furnace the fumes are conducted through a common flue to a combustion chamber.

Ore and anthracite coal or any other small-sized smokeless fuel are thoroughly mixed with water. The charge is spread over the grate after the furnace has been properly lighted. Zinc vapour produced is burnt to oxide in combustion chambers.



1. Concentrated unloading station. 2. Roaster. 3. Mixing plant. 4. Charging bin. 5. Furnace block. 6. Bag house. 7. Refinery. 8. Packing house. 9. Warehouse.

FIG. 1 — FLOW-SHEET FOR MANUFACTURE OF ZINC OXIDE BY WESTERN WETHERILL PROCESS (American Zinc Oxide Co.).

The oxide along with gases is passed through bag filters where the zinc oxide is collected. Flow-sheet for this method is given in Fig. 1.

(b) Travelling-grate Method<sup>1</sup>— Ore and coal are used in the form of briquettes. Ore briquette is composed of 80 per cent finely divided ore plus 20 per cent fine-sized anthracite (coal briquette is prepared from low grade fine anthracite). Sulphite liquor is used as binder for coal briquetting as well as for ore briquetting.

The travelling grate, in its forward journey, first picks up coal briquettes which start burning as the grate enters ignition zone. A layer of ore briquettes is then deposited over the incandescent coal bed and the grate enters the reduction zone under a forced draft. Zinc fumes produced are passed on to a combustion chamber and oxide is formed.

A flow-sheet of the process is given in Fig 2.

(c) Waelz Process<sup>1,3</sup>—Ore is subjected to a preliminary roasting if the sulphur content is high (more than 10 per cent). Fine-sized ore (75 per cent) is thoroughly mixed with coke dust (25 per cent) and

water. This charge is fed into an inclined rotary kiln which is heated by pulverized charcoal and is under an induced draft. Zinc fumes produced are passed through the oxidizing chamber and then to bag filters.

(d) Electrothermic Process<sup>1,4</sup>—Ore or sulphide concentrate is first roasted whereby most of the sulphur is eliminated as SO<sub>2</sub>. The roasted concentrate is then thoroughly mixed with a flux, fuel and water and sintered at about 1,600°C. The sintered cake is crushed, screened and combined with carefully sized coke and preheated to about 750°C. The preheated material is then charged into an electric furnace in which the charge itself serves as the resistance in developing temperature. Air is forced inside the furnace which oxidizes the zinc fumes. Oxide is then passed through a cyclone separator to the bag house.

The electrothermic process is adopted by St. Joseph Lead Co., U.S.A., with an ore of the composition: Zn 57 per cent, Pb 0.6 per cent, Fe 6.6 per cent, Cd 0.1 per cent, SiO<sub>2</sub> 1 per cent, CaO and MgO 0.7 per cent. The typical example of the final product is ZnO 99.4 per cent, PbO 0.01 to 0.5 per cent, CdO



Coal bin. 2. Ore bin. 3. Weighing hopper. 4. Elevator. 5. Mixed storage bin. 6. Conveyer.
 Binder. 8. Paddle mixer. 9. Pan mixer. 10. Elevator. 11. Large briquetting press. 12. Briquette pan dryer. 13. Fan. 14. Fire box. 15. Oxide furnace. 16. Blow house. 17. Fan house. 18. Cyclone separator. 19. Bag house. 20. Packing house. 21. Oxide storage.

FIG. 2 — FLOW-SHEET FOR TRAVELLING-GRATE FURNACE METHOD.
0.002 per cent, Fe<sub>2</sub>O<sub>3</sub> 0.002 per cent, total S as SO<sub>3</sub> 0.06 per cent.

Flow-sheet of the process is given in Fig. 3.

(e) Electric  $Process^5$ —Zinc ore or concentrate is roasted with free access of air in an electric furnace whose carbon sides serve as electrodes. A layer of powdered charcoal connects the electrodes before starting. Air is forced in from the top of the furnace. Zinc oxide formed is passed through cyclone separator and bag filters.

(f) Flash-fume Process<sup>6</sup> — Zinc sulphide concentrate, after being completely dried, is dispersed into four or more furnaces by compressed air nozzles and ignition is accomplished by natural gas burners. Sulphur in the concentrate burns like powdered coal or atomized oil. The collected zinc oxide is sent for heat treatment to improve its physical and chemical properties. It is then discharged into a hammer mill to break any agglomerated particles formed during heat treatment. The oxide then passes on to a blending plant.

If leaded zinc oxide is desired, a calculated amount of basic lead sulphate is added to zinc oxide.

2. Indirect Process<sup>7,8</sup> — Oxidation of zinc in crucibles each with its own furnace gives good quality oxide if very pure zinc is used. Zinc is vaporized in a reducing atmosphere at approximately  $1,200^{\circ}$ C. and then passed to the combustion chamber when a forced draft is maintained keeping the temperature at 1,100°C. The gases and oxide are then passed to another chamber where temperature is kept at 850°C. They are passed to the next chamber with gradually decreasing temperature until they reach the bag filters.

The oxide is refined by reheating. If cadmium is present, it is removed by preheating in a reducing temperature.

3. Wet Process<sup>9</sup> — Zinc ores, preferably roasted; are heated with an excess of sulphurous acid solution with the formation of soluble zinc bisulphite which, when heated, separates out as insoluble zinc sulphite. The sulphite is washed, redissolved in excess of sulphurous acid and the liquor is treated with milk of lime, which precipitates zinc hydroxide. The latter is digested with ammonia to form ammonium zincate, from which zinc oxide is obtained by calcination. Ammonia is regenerated.

#### Comparative Merits & Demerits of Different Methods

Among the three grate-type furnaces, the travelling grate is more advantageous to operate than the others. Rate of combustion of the charge has a great influence over certain physical properties of pigment. A free-burning open charge, rapidly worked, produces material different from that produced by a charge that is dense and slowworking. The use of briquetted charge in



Ore storage. 2. Roaster. 3. Hammer mill. 4. Screen. 5. Mixer. 6. Sinter machine. 7. Ignition furnace. 8. Tooth-roll crusher. 9. Smooth-roll crusher. 10. Trommels. 11. Cottrell dust precipitator.
 Preheater. 13. Electric furnace. 14. Fan. 15. Agglomerate trap. 16. Filler bags. 17. Bag house. 18. Blend bin. 19. Packer. 20. Screen. 21. Storage bins.

FIG. 3 — FLOW-SHEET FOR ELECTROTHERMIC PROCESS (St. Joseph Lead Co.).

the travelling-grate method helps to attain the above working conditions.

In the Waelz process, the necessity for pre-roasting is eliminated as ferrites, ferrates, sweet roast and sulphate roast are all easily reduced in the kiln. As the reducing and oxidizing zones are superimposed over the whole length of the reduction zone, the amount of heat consumed by chemical reactions is balanced. A lesser amount of fuel is needed as the burning of zinc vapours gives back a corresponding quantity of energy.

The treatment of slags from lead-smelting blast furnaces for extraction of these zinc contents and recovery as oxidized fume is done on a large scale by this process at the *East Helena Slag-treating Plant of the Anaconda Copper Mining Co.* and elsewhere. Electrolytic back-leach residues need not be dried, for the process will treat slimes running 40 per cent moisture without preliminary drying. Fuel consumption is also low and almost any sort of coke can be used.

St. Joseph Lead Co. of Josephtown is using the electrothermic process. The process requires elaborate equipment and hence is costly.

The success of the electric process depends on the availability of cheap power.

Flash fuming of sulphide ores is the latest development towards the production of pure zinc oxide for use as a pigment. The process is simple and there is a great increase in furnace capacity and perfect control over the desulphurization. But there is the disadvantage of a large amount of dusting. The method is in the experimental stage and its use on an industrial scale has not yet been adopted.

Purification of Zinc Oxide<sup>9</sup> — The principal impurities in zinc oxide are the oxides of Bi, Sb, Cd, Se, Te and basic lead sulphate. Zinc oxide can be purified by digesting it under slightly reduced pressure with 4-N ammonium carbonate at ordinary temperature. All the zinc oxide is dissolved together with Cd and a small quantity of Ag, leaving a residue of basic lead carbonate and other impurities. The filtered solution is passed through a cylinder containing scrap zinc to remove Cd and Ag and then distilled with live steam, whereby zinc is deposited as carbonate which can be converted into oxide by heating at 400°C. Zinc oxide condenses at  $1,370^{\circ}$ C. whereas other volatile oxide impurities condense at  $1,060^{\circ}$ C. By careful control, this difference in condensation temperatures can be utilized for the separation of zinc oxide from its impurities.

Uses of Zinc  $Oxide^{9-13}$  — Various uses are in :

1. Pigment industry

- 2. Rubber industry
- 3. Ceramic industry
  - (a) Glazes
  - (b) Enamel
  - (c) Glasses
- 4. Cosmetics and medicinal purposes.

For white pigment, pure oxide is essential; sometimes leaded zinc oxide<sup>6</sup> is also used. The most common blends for the latter type of pigments are (i) 35 parts sublimed white lead with 65 parts zinc oxide, and (ii) 50 parts of sublimed white lead plus 50 parts of zinc oxide.

In rubber<sup>11</sup>, the oxide acts as an accelerator, activator and imparts high strength to the finished product. As a reinforcing agent, it is second only to carbon blocks. Where high tensile and tear resistance are required and colour is also important, it holds the field. It is used as a compounding ingredient for all synthetic rubber such as Butadiene-styrene rubber, Butyl rubber, Neoprene, Thiokol, etc., and serves as the curing agent. Best results are normally obtained with 5 per cent based on the rubber, but in tyre treads and other such carbon black compounds, 8 or 10 per cent is often used.

In the ceramic industry<sup>12</sup>, it is commonly used in glazes and in many optical glasses to reduce their tendency to crystallize on cooling. In glazes, it acts as a fluxing agent, reduces the expansion, prevents crazing and increases the gloss and improves the whiteness and opacity.

Zinc oxide in sheet-iron enamels<sup>13</sup> increases fusibility, gives a superior gloss, lowers frit solubility and increases acid resistance.

## Specifications for Zinc Oxide for Pigments<sup>14</sup> - 1. Zinc Oxide

#### Composition & Properties

(a) Dry Pigment — The pigment shall be zinc oxide of either the American process type or the French process type to conform to the following requirements:

•	American process type		French process type	
	max.	min.	max.	min.
Zinc oxide		98		99
Total sulphur	0.2	_	0.1	_
Moisture and other volatile matter	0.5	_	0.5	_
Total impurities including moisture and other volatile matter	2.0	_	1.0	
Coarse particles (total residue retained on a				
No. 325-44 micron sieve)	1.0	_	1.0	-

(b) Paste in Oil - The paste shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall conform to the following requirements :

Pigment, per cent	80-16
Linseed oil, per cent	14-20
Moisture and other volatile matter, per cent	0.5 max.
Coarse particles and skin (total residue retained on a No. 325 sieve), per	
cent of dry pigment	1.5 max.

(c) In the event that either an acicular or a nodular type of zinc oxide is desired, the particle shape shall be determined by examining or photographing microscopic mounts (1000X or more) of the samples.

#### 2. Leaded Zinc Oxide

#### Composition & Properties

(a) Dry Pigment — The pigment shall consist of zinc oxide and normal or basic lead sulphate and shall conform to the following requirements :

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Zinc oxide, per cent	62-67
Matter soluble in water, per cent	1.0 max.
Moisture and other volatile matter,	
per cent	0.5 max.
Total impurities including moisture	
and other volatile matter, per cent	2.0
Normal or basic lead sulphate	remainder
Coarse particles (total residue re-	
tained on a No. 325 sieve), per cent	1.0 max

tained on a No. 325 sieve), per cent

(b) Paste in Oil — The paste shall be a dispersion of the specified pigment in linseed oil. As received, it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportion, without curding with linseed oil, turpentine or volatile petroleum spirits or any mixture of these substances. The paste shall conform to the following requirements :

Pigment, per cent	81-87	
Linseed oil, per cent	13-19	
Moisture and other volatile matter, per cent	0 <sup>.5</sup> max.	
Coarse particles of skins (total residue retained on a No. 325 sieve).		
per cent of dry pigment	1.5 max.	

#### Specification for Zinc Oxide from **Messrs** Paint Federation, Calcutta

Description - The material shall consist of zinc oxide in the form of dry powder.

Moisture — Maximum 0.5 per cent.

Composition - After drying at 98° to 102°C., it shall contain not less than 99.5 per cent of zinc oxide. The percentage of lead expressed as PbO shall not exceed 0.2.

Coarse Particles - It shall not contain more than 0.1 per cent of coarse particles when tested in the manner described under the general clause.

Oil Absorption — The oil absorption of the material shall not be less than 12 or more than 20 when determined in the manner described under the general clause.

Colour & Reducing Power - In whiteness and reducing power, the material shall be equal to the agreed sample. The test for reducing power shall be carried out in the manner described under the general clause.

The zinc concentrates that may be available from Zawar Mines is expected to have the following approximate analysis:

Zn	55-60
Pb	up to $5$
Cd	0.2-0.2

#### Conclusion

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The electrothermic process produces high grade of zinc oxide, but the initial cost of installation of the plant is high, so also is the operating cost.

Much is not known either about the flashfume process or the direct electric process.

Out of the remaining, the Wetherill and the Waelz processes are extensively used in the United States. With the Wetherill furnace, it is necessary to counteract the tendency of the blast to break through the charge, making blow-holes and craters. This requires constant attention. These disadvantages were overcome by the New

Jersey Zinc Co. by briquetting the charge made up of ore and coal. This opened the by travelling-grate method which the output per furnace increased, resulting in lower operating and investment cost per pound of product. The quality of product is also under better control.

The Waelz process is simple and relatively cheap for treating low grade ores and for recovery of zinc from lead smelting blast furnaces.

The process suitable for zinc sulphide concentrates appears to be the improved Wetherill process employing the travelling grate.

#### Acknowledgement

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## Nuffield Foundation Travelling Fellowships for India

THE NUFFIELD FOUNDATION, WITH THE object of advancing the interests of India and for further strengthening the academic ties between India and the United Kingdom, has decided to make available to India five Travelling Fellowships from 1951-52. An Advisory Committee consisting of Shri Gaganvihari L. Mehta (Chairman), Shri S. Varadachariar, Dr. C. V. Raman and Shri J. J. Ghandy has been appointed to advise the Foundation on the administration of the scheme. The Fellowships will be awarded by the Trustees of the Foundation on the recommendation of this Advisory Committee.

It has been decided to award Fellowships during 1951-52 in the following subjects :

Two Fellowships in Medical Sciences, preference being given to candidates wishing to study (1) Physiology or Bacteriology, and (2) Industrial Medicine or Public Health. One Fellowship in Engineering, preference being given to candidates wishing to study Electrical Engineering (Generation or Distribution). One Fellowship in Natural Sciences, preference being given to candidates wishing to study Plant Genetics or Soil Science; and one Fellowship in Social Sciences, preference being given to candidates wishing to study Industrial Relations or Personnel Management, and Agricultural Economics.

REFERENCES

- 1. WEATON, G. F.: Trans. Amer. Inst. of Min. & Metall. Engs., 1936, **121**, 599; also Bunce, E. H. & Haslam, H. M.: ibid., 678; Harris, W. E.: ibid., 702.
- 2. EIDE, A. C. & EASLEY, M. K. : Engng. & Min. J., 1938, 139, 67.
- 3. MATHY, C.: Industr. & Engng. Chem., News Ed., 1932, 10, 146.
- Chem. & Metall. Engng., 1935, 42, 598.
   SIMM, W. & SIMM, H.: J. Soc. Chem. Ind., 1907, 26, 1150.
- 6. HALLOWS, R. L.: Engng. & Min. J., 1943, 144, 113.
- 7. MACONOCHIE, H. & DEROS, D. : J. Soc. Chem. Ind., 1918, 37, 622A.
- 8. DANIELS, R. G.: Chem. Abs., 1928, 783.
- Danieles, A. G., Chem. 1926, 1926, 193.
   FERRERE, P. : J. Soc. Chem., 1915, 34, 228.
   WARING, W. G.: Chem. Abs., B, 1926, 95.
   Chem. Age, 1949, 60, 22.
   Cer. Ind., 1948, 50, 132.
   FERREROPE L. D. American Science L. 1926

- 13. FETTEROLF, L. D.: Amer. Cer. Soc. J., 1933, 16, 319.
- 14. A.S.T.M. Standards, 1946, Part II, 862.

The purpose of the Fellowships is to enable Indian graduates of outstanding ability to gain experience and training in the U.K. in their chosen fields, and to make contacts with scholars working in those fields, with a view to the Fellows equipping themselves to take up senior posts in research and teaching in India.

The Fellowships are intended for men or women of first-rate intellectual and personal qualities, who have already shown unusual capacity to advance knowledge and teaching in one of the fields concerned. Candidates must be Indian nationals, normally between the ages of 25 to 40 years, and must be university graduates holding, preferably, a Master's or a Doctor's degree and having subsequently had a year or more of teaching or research experience on the staff of a university or comparable institution.

A Fellowship will normally be tenable for one year, but in exceptional cases may be extended by a few months, the tolal value of the award being (exclusive of travelling expenses)  $f_{2}$  770 to  $f_{2}$  890 a year.

Applications for Fellowships to begin in 1951 should be submitted not later than the 31st March 1951 to the Secretary, Nuffield Foundation Advisory Committee in India, C/o Planning Commission, Government House, New Delhi, from whom copies of the form of application may be obtained.

Organic Chemistry, by Paul Karrer, translated by A. J. Mee (Elsevier Publishing Co., Amsterdam, London, New York & Brussels), 1950, Fourth English Edition, pp. 983. Price 57s. 6d.

KARRER'S "ORGANIC CHEMISTRY", THE first English edition of which was published in 1938, has now established itself throughout the world as one of the standard textbooks on the subject. The first German edition (Lehrbuch der Organischen Chemie) appeared in 1928, and the English translation made in 1938 was based on the fifth German edition. In view of the fact that Karrer is among the greatest organic chemists of all time, this is a safe choice of a text-book giving a clear, comprehensive and balanced account of a vast and rapidly expanding science. Considering the scope and size of the book, it makes very pleasant reading from end to end. Karrer's approach is essentially classical and his treatment lays emphasis on synthetic reactions, experimental methods and the steps by which the structures of naturally occurring compounds are determined. Both synthetic and natural substances of biological interest receive special attention. In revising the third edition, much of the material has been brought up to date. Some of the additions are streptomycin, organic silicon compounds such as the silicones, and organic compounds containing carbon and nitrogen isotopes. The chapters or sections dealing with microanalysis, sex hormones, vitamins and alkaloids have been largely rewritten. The chemistry of chloromycetin (chloramphenicol) was apparently not known in time for inclusion.

Deficiencies in the book are mainly in the sense that for a full course in organic chemistry it must be supplemented by books on the theory of organic chemistry, the technique of organic chemistry and organic chemical technology or, at least, by Bernthsen and Sudborough's text-book which covers a wider range of topics treated in a systematic manner. Karrer's classification according to functional groups and ring systems is generally satisfactory, but leads to such anomalies as the widely separated occurrence of sulphanilamide, sulphathiazole and sulphapyridine. There are no references to the original literature. There are a few references to books, sometimes not to the latest book on the subject, e.g. for diazo compounds, Hantzsch and Reddelien's classical book is rightly cited, but Saunders, 1936 and 1949, might well have been added. The importance of the sulphanilamides would have been demonstrated very simply if a reference to Northey's book of 660 pages had been made.

Although it is obvious that a text-book cannot be an encyclopaedia and omissions are inevitable, there are some which need to be noticed, e.g. preparation of succinic acid by catalytic reduction of maleic acid; chromatography and in particular the separation and identification of amino acids by partition chromatography; lauryl paper al cohol, sodium lauryl sulphate and other wetting agents and detergents ; the oxidation of ethylene to ethylene oxide and the technical importance of the latter; 9:10-dime-thyl-1:2-benzanthracene and 20-methylcholanthrene as most potent carcinogens; Bardhan's synthesis of phenanthrenes; diaminodiphenvlsulphone; trifluoromethylbenzene and its derivatives ; the Raschig process for phenol; technical production of phthalic acid from o-xylene, of benzoic acid by decarboxylation of phthalic acid and of benzovl chloride from benzotrichloride; esters of phthalic acid and their uses as plasticizers; the use of phthalic anhydride in the manufacture of alkyd resins; zinc chloride double salts of diazonium salts (the common type of stabilized diazonium salts); azoic coupling components other than Naphtol AS; formation of complexes by trinitrobenzene; technical preparation of m-nitrotoluene by fractionation of the nitrotoluenes; 4- and 5-aminosalicylic acid; the technical importance of terephthalic acid; selenium dioxide oxidation of o-hydroxychalkones to flavones; copper chromite, Raney nickel and lithium aluminium hydride reductions; preparation of thionaphthene from thioindoxyl (which is superior to the method mentioned); cyanine dyes other ( and far more important ) than the quinoline derivatives; the Pfitzinger, Willgerodt and various other important reactions. The title of chapter 39 ("Quinone Dyes with Closed Rings") is inappropriate. Erroneous structures are given for diazotized sulphanilic acid and Indanthrene Bordeaux B. A new structure is suggested for Hydron Blue with no reference to experimental data. Algol Yellow 3G is mentioned as Algol Yellow G.

K.V.

Progressive Mathematics, by P. Clyne, (Chapman & Hall Ltd., London), 1950, pp. xii + 270. Price 15s. net.

THE AUTHOR, IN HIS PREFACE, HAS EXplained that the object of his book is to develop the mathematics from about the School Certificate stage to Higher National Certificate level, as a serial story, sweeping into appendices all interesting mathematical results which do not fit in the picture. We have the authority of the foreword that the method of the text has worked successfully with many students of our "Responsible Lecturer". The reader is perhaps to be congratulated on his being carried a long way in a short space, though the process may be rather risky. The long way starts from zeroes, infinites and their quotients, deter-minate and indeterminate, and labours through differentiation, maxima and minima, infinite series, integration, conic sections, vectors, Fourier series and differential equa-To the appendices are relegated tions. equations, triangle mensuration, permutation and combination, the binomial theorem, important curves like the catenary, the cycloid and the cardioide, polar co-ordinates and determinants.

The short space in which the author has woven such a variety of topics could have been made even shorter with a little restraint of diction and doubtful humour. What is the use of such a definition as "Successive points are those which are closer together than any specified distance, yet both on a curve" (p. 9)?

The author's humour may be tolerated in a serious text-book if it does not go too far into fields of irrelevancy and coarseness and does not waste the reader's time. Such terms of expression as "integrating the brute" and "formula-fiends" serve no purpose. The dialogues between the student

and the teacher, though sometimes spicy, become of doubtful significance when they are enigmatic (p. 14).

The reviewer is constrained to remark that there are certain definitely wrong teachings in the text. One wonders how intelligent students with adult minds can readily swallow the different-sized zeroes ( p. 21), the proof of the indeterminacy of  $1^{\infty}$  (p. 5), the proof of the area of the circle based on

$$\begin{array}{ccc} \text{Lt} & \underline{\sin\theta} \\ \theta \to 0 & \overline{\theta} \end{array}$$

which is itself proved with the help of the formula  $\pi r^2$  (pp. 41, 42), the mode of gradual distortion of a circle into an ellipse, a parabola and a hyperbola (p. 117) and the conditions for convergence which are half-truths, since convergence and finite oscillations are not clearly distinguished (p. 71). Though the author does not wish himself to be regarded as a creature of tradition, he has thought fit innocently to repeat such traditional mistaken notions as (1) the lesser of the turning value must be the minimum (p. 60) and (2) a parabola is obtained by taking a section plane parallel to one of the generating lines (p. 117).

In spite of all the erratic and unconventional modes of presentation and misplaced rhetoric, there is a certain sincerity of exposition underlying Progressive Mathematics which gives the book its real value. The chapters on integration and Fourier series are particularly well done. There are numerous worked examples with applications to engineering and there is a good collection of exercises at the end of each chapter and answers are also provided for them. Anv student carefully plodding through the book will get a useful mathematical foundation which will stand him in good stead in various practical applications of mathematics.

The printing and get-up of the book are just what may be expected of the well-known publishers, Chapman & Hall. The latter part of the book contains a few printing mistakes which may be rectified in a second edition.

A. A. KRISHNASWAMI AYYANGAR

The Vanderbilt Rubber Handbook, edited by S. S. Rogers (R. T. Vanderbilt Co., New York), 1948, pp. 719.

THIS IMPORTANT BOOK, NOW IN ITS NINTH edition, is based on the formulations for the

compounding of rubber products with the compounding materials marketed by the Vanderbilt Co. True to its tradition, the book admirably maintains the rôle of initiating "new men coming to the rubber industry in the fundamentals of compounding and to provide the more experienced technologists with a fund of reference material in a handy form".

The book is divided into five sections. The articles in these sections are written by specialists and bear their stamp of authority for accuracy. Section I, covering the field of compounding, both of dry rubber and latex, has chapters on types of polymers, both synthetic and natural and their reclaims, rôle of fillers, accelerators, antioxidants, vulcanizing agents, processing aids and the principal processing operations involved in general rubber goods manufacture. Section II gives a tabular summary of the properties and uses of Vanderbilt compounding materials. Under Section III are given test methods and equipment covering essentially the methods which have been developed by the various rubber manufacturing concerns to compare and evaluate the products and have the approval of the trade or of the American Society for Testing Materials. It is rightly emphasized that no laboratory tests can quantitatively predict the service behaviour of the product but are merely a check on the properties of a component which within the experience of the technologist has given a satisfactory product. This section could probably have been made more complete by inclusion of methods for the chemical analysis of rubber products (A.S.T.M., D. 297-43 T).

Section IV gives general articles on subjects like yarns and fabrics, textile testing, rubber derivatives, application of X-ray and electron microscope methods and induction heating in the field of rubber, etc. The treatment of these articles is elementary, but each one is enriched with an excellent bibliography to guide those interested in further information.

In Section V are assembled tables and charts on the statistics of rubber, both natural and synthetic, their consumption, price movements over a number of years, specific gravity of rubber compounding ingredients, gravity and weight charts useful in designing a rubber compound, steam pressure-temperature charts and other valuable conversion data needed by a rubber technologist. The book is a mine of information and the publishers, who have been issuing "Vanderbilt News" and other laboratory reports on rubber materials and processing, have done a signal service in issuing them in the form of the above book. Rubber industry is known for its "bugs" and this book may help to locate these.

M. R. VERMA

Engineering Supersonic Aerodynamics, by E. Arthur Bonney (McGraw-Hill Book Co. Inc., New York), 1950, pp. xxii + 329. Price \$ 3.00.

IF AN AERONAUTICAL ENGINEER IS LOOKING for a book that will introduce him to the basic information on the subject of supersonic aerodynamics, he will find such a book in Arthur Bonney's *Engineering Supersonic Aerodynamics*. The goal set by the author in his preface is fulfilled in an admirable manner. He has endeavoured to present the practical applied information to the engineer engaged in the design of supersonic aircraft.

In order to understand the basic principles outlined in the book, one should have a knowledge of differential and integral calculus, differential equations and a thorough understanding of thermodynamics in addition to simple mechanics. This book, however, cannot be used as a standard text-book for the graduate course, as all mathematical derivations are purposely avoided by the author, whose aim, throughout the book, has been not to disturb the continuity of the physical picture of the supersonic flow.

The book consists of seven chapters, beginning with "Compression and Expansion Waves" and ending with the "Composite Design and Performance". A large number of problems is worked out in the body of the book, and at the end of each chapter many typical examples, emphasizing the principles involved, are set for the students to solve.

A few noteworthy features of this volume are the presentation of the graphical results of the conical shock-wave solution by Taylor and Maccoll, the linearized theory solution for bodies of revolution by von Kārmān, Tsein and Ferrari, and a descriptive introduction to the supersonic wind tunnel construction and experimental procedure. The most recent work on the aerodynamic characteristics of delta wings by Pucket and Stewart is also included. In the last chapter, the author concludes the book by briefly discussing the physical aspects of wing-body-tail interference, propulsion and performance.

It is observed that the discussion on topics like entropy and enthalpy seems to be somewhat brief and inadequate. Today, an aeronautical engineer would better appreciate the physical picture of shock wave if his attention is drawn to the increase of entropy rather than to a decrease of total pressure and hence the reviewer feels that the author should have devoted on the aforesaid topics much more space than what has been done in this book.

Though the mathematical treatment is simple and within the scope of an undergraduate student, the physical picture presented in the book seems to be a bit advanced for him to grasp. In the opinion of the reviewer, one should have a thorough grasp of the subsonic aerodynamics for conventional types before he proceeds to tackle problems on supersonic aircraft, and this knowledge one can acquire only in the graduate school. However, the author is to be congratulated for having written this volume in such a lucid language so as to bring the subject-matter within the grasp of an undergraduate student.

V. CADAMBE

Colours and How We See Them, by H. Hartridge (G. Bell & Sons Ltd., London), 1949, pp. xi + 158. Price 15s.

THE ROYAL INSTITUTION CHRISTMAS LECtures have produced some of the best books on popular science. Dr. Hartridge's fascinating volume is the first book based on these lectures to be published since the war.

The lectures, which have been improved upon to suit the book form, are more exhaustive than its title is likely to suggest and cover a wide range of subjects. They include, amongst others, a very vivid description of several optical phenomena such as diffraction, interference, polarization, etc. One full chapter is devoted to "Some Illusions of Colour " and another to " Colours and Their Uses in Science and Arts". The latter chapter comprises certain details as to how colours are made use of by animals and plants. The dominating chapter is the one wherein the author deals at length with the mechanism of vision and claims the

superiority of the eye over the photographic camera, for he asks "where is a camera the iris of which adjusts itself according to the intensity of light or the photographic plate which works almost as well for the dim interior of the cellar as it does for the most brilliantly lit landscape?"

A prominent feature of the book is the large number of illustrations and plates. The methods which the author presents for projecting Newton's rings, spectra and similar phenomena to a large audience are very interesting and should prove of considerable value to teachers for demonstration purposes.

#### Sohan Singh

Electrical Power System Control, Monographs on Electrical Engineering, Vol. XI, by H. P. Young (Chapman & Hall Ltd., London), 1950, pp. xvi+456, Cloth Bound. Price 28s. net.

THIS IS A VERY USEFUL VOLUME IN THE series of "Monographs on Electrical Engineering" under the editorship of Mr. H. P. Young, and contains valuable information on the operation of generating equipment in large power-stations, regulation of their voltages, synchronizing arrangements and the methods of interconnection with other power-stations. It gives valuable information about the switch-gear and protective equipment of large power-stations.

Chapters I to V deal with parallel operation of generating voltage control, types of automatic regulators and their operation, automatic synchronizing of generators and control of power by reactance. Results of latest investigations and developments have been made clear and understandable by men in charge of power-stations as well as those who teach electrical engineering in technical colleges.

Chapters VI to VIII deal with circuit breakers, their design and principles of operation, rupturing capacity, latest development in low oil content and air blast switch-gear, arrangements of power-stations, switch-gear and short-circuit calculations.

Chapters IX to XI deal with interconnection of power-stations and apparatus for such control. The principles of automatic supervisory control are clearly explained.

Chapters XII and XIII deal with the automatic protective system for feeders, generators, transformers and bus bars. Typical protective systems of the latest design and the principles of operation are clearly explained. The get-up of the book is excellent like other volumes of the series and makes it a very useful addition for the technical library of engineers in the power engineering profession and of colleges.

B. K. R. PRASAD

Elements of Thermodynamics and Heat Transfer, by Edward F. Obert (McGraw-Hill Book Co. Inc., New York), 1949, pp. xiv + 372. Price \$ 4.50.

THE BOOK UNDER REVIEW IS A VERY WELcome addition to thermodynamic literature. While there are excellent treatises on thermodynamics, both on the elementary and the advanced portions of the subject, there would appear to be a real need for books intended mainly for students taking up an engineering course. The aim of the author has been to satisfy this demand. No one who has gone through the pages of this book would deny that the author has succeeded in this purpose remarkably well. He can also justly claim that the presentation of the subject has been throughout " simple and direct without sacrificing rigour or the emphasis on the fundamental aspects and continuity of the Science ".

The book begins with a chapter on units and dimensions. The student should find this particularly useful since the subject has considerable applications in other branches of engineering; and a basic knowledge of it will, therefore, greatly facilitate his work. The second chapter deals with the fundamental concepts of thermodynamics like energy, work, state, etc. The next three chapters are devoted to a treatment of the first and second laws of thermodynamics. The properties of liquids and gases are discussed in chapters VI, VII and VIII. Chapters IX to XIV deal with the application of thermodynamics to fluid flow, power cycles and refrigeration. The last chapter is on heat transfer. A specially noteworthy feature of the book is a selection of fine and wellgraded examples which an engineer often comes across in his everyday work. There are also useful appendices on physical constants for ready reference.

By way of criticism the reviewer feels that the arrangement of the text at certain places could have been a little different, leading to

some gain in logical coherence and elimination of repetition. For example, the definition of heat capacities and specific heats occurs as late as in page 57 in the middle of a discussion on reversibility. Thermodynamical calculations are postponed to chapter XI, thus slightly obscuring their dependence on the first law. Discussion of the boilers, reciprocating piston pumps, etc., immediately after defining the first law in chapter III, looks rather early. The carnot cycle has been virtually discussed thrice, in chapters V, XII and XIII. Incidentally, no mention is made of Hess's law in connection thermochemistry. With regard to with chapter XIV on refrigeration, a fuller discussion of heat pumps and their applications in industry would have been more profitable, as this is a subject which is fast The introduction or at least a developing. brief mention of Maxwell's relationships and their applications would have been welcome in later chapters on account of their importance in thermodynamics.

Apart from these minor points the book will prove to be a valuable introduction to basic thermodynamics for engineers. The get-up of the book maintains the high standard set up by the publishers. The diagrams are excellent and illustrate the text extremely well. P. K. KICHLU

Railway Track, by K. F. Antia (New Book Co. Ltd., Bombay), 1949, pp. xix + 416. Price Rs. 16.

IN BRINGING OUT THE SECOND EDITION OF his book on railway track, which is quite pleasing and attractive, the author has endeavoured to include the latest and more up-to-date methods in the construction and maintenance of railway track. For this purpose he has revised most of the chapters and added a new chapter on track stresses. The author's experience as Deputy Chief Controller of Standardization in the Central Standards Office for Indian Railways has afforded him the fullest opportunities to come into contact with recent investigations and research on tracks in India and elsewhere, and the information he has furnished should be of considerable benefit to those who are engaged in similar investigations.

The book contains very useful information and is a valuable guide to track engineers and inspectors. Its utility would, however, have been enhanced if the fundamental principles underlying the design of track sub-structure and super-structure were treated with greater clarity. As it is, the inexperienced reader may be bewildered with the mass of details and apt to miss the basic principles. This is perhaps inevitable as the book was originally prepared from the notes taken by the author during his work in the railways. His second edition is, however, a great improvement on the first in this respect.

A few statements made by the author are misleading and require correction. In the first chapter, on page 6, the maximum axle loads in India are stated to be 28 tons for broad gauge, 17 tons for metre gauge and 13 tons for narrow gauge. Although at one time these axle loads were contemplated, no locomotive was actually built for Indian railways with such heavy axle loads and the maximum axle loads on Indian railways have so far been limited to 22.5 tons, 12 tons and 8 tons respectively for broad, metre and narrow gauges. In the second chapter, on page 12, it is stated that the approximate permissible axle load in America is taken as 300 times the weight of the rail per yard. This reference should have been the wheel load and not the axle load.

In the chapter on track stresses (page 93) the magnitude of the steam effect is stated to be small. In practice, the steam effect is quite appreciable when compared to the hammer blow due to the overbalance as the figures on page 97 against hammer blow and steam effect reveal. The statement in the seventeenth chapter on page 297 that the wrong type of spring or alteration in the length of the spring hanger might completely unload the wheel is exaggerated. Within limits and provided the compensating beam does not touch the stop, variation in the length of hanger is not considered to be of material importance.

On the whole, track men should feel greatly indebted to Mr. Antia for this book which provides a very handy and useful manual for permanent way.

V.V.

Canning Practice and Control, by Osman Jones (Chapman & Hall Ltd., London), 1949, Third Edition, Revised & Enlarged, pp. vi + 314 and Index. Price 36s. net.

THE GREAT POPULARITY OF THE FIRST TWO editions of the book has led to the publication

of the third edition. Mr. Osman Jones is the sole author of the present edition, Mr. J. W. Jones having resigned his rights of coauthorship.

During the seven years that have intervened between the publication of the second and third editions there have been many developments in the theory and practice of canning and the author has made a thorough revision of the subject-matter in the light of these developments. Particular mention may be made of the recent methods of vitamin assay, examination of suspected materials in food poisoning cases, new culture media, modern sealing compounds, etc. The microbiology of canning is dis-cussed at length and the importance of hygienic conditions in canning practice has been clearly brought out. In this chapter the author has been eminently successful in imparting to his readers, who may not have had any grounding in bacteriology, an understanding of the implications of microbiology as applied to canning.

The book is profusely illustrated with photographs of processing techniques employed in canning and photomicrographs of spoilage micro-organisms. It is regrettable that several misprints have crept in, and we have no doubt that these will be eliminated while reprinting the volume.

S.A.C.

#### **Publications Received**

- PRINCIPLES OF AERODYNAMICS, by James H. Dwinnell, McGraw-Hill Book Co. Inc., \$ 5.50
- CHEMICAL ENGINEERS HANDBOOK (Third Edition), by John H. Perry, McGraw-Hill Book Co. Inc., \$ 12.50
- CHEMICAL AGE, C: 19 Sitaram Buildings, Bombay 1, Rs. 10
- WELDING DESIGN & PROCESSES, by B. Richard Hilton, Chapman & Hall Ltd., 36s.
- PROGRESS IN CHROMATOGRAPHY (1939-1947), by L. Zechmeister, Chapman & Hall Ltd., 45s.
- CELL PHYSIOLOGY & PHARMACOLOGY, by J. F. Danielli, Elsevier Publishing Co. Ltd., \$ 3.00
- DRILLING & SAMPLING OF BITUMINOUS SANDS OF NORTHERN ALBERTA, Vol. 1, Canada Department of Mines & Resources, 25 cents

#### MANUFACTURE OF FILTER PADS FOR STERILIZING LIQUIDS

SPECIAL TYPES OF FILTER PADS ARE required for preparing bacteria-free filtrates in pharmaceutical and biological work. Prior to the World War, the country's requirements of such pads were met by imports of well-known foreign brands such as the Seitz E.K. pads manufactured in Germany. With the cessation of supplies from Germany and other foreign sources during the war, there was an urgent demand for the manufacture of these pads in this country. As a result of the investigations carried out in the laboratories of the Council of Scientific & Industrial Research, a process has been worked out for their manufacture from raw materials readily available in the country. The pads manufactured by this process satisfy all the standard specifications with respect to sterility of the filtrate, rate of filtration, etc.

The process consists of the following steps:

- 1. Preparation of the absorbent.
- 2. Preparation of the pulp medium.
- 3. Incorporation of the absorbent powder in the pulp medium.
- 4. Preparation of the final suspension.
- 5. Preparation of the pads.

The following equipment costing about Rs. 2,500 is required :

- 1. Aluminium or enamelled pans.
- 2. Mechanical shaker.
- 3. Sterilizer to stand a pressure of 50 lb. per sq. in.
- per sq. in. 4. Vacuum pump.
- 5. Hot-air oven.
- 6. Hand press.
- 7. Punches, dies, etc.

#### Manufacturing Cost

	Rs.	as.	p.	
Cost of raw materials and			-	
chemicals per 1,000 pads of				
7 cm. dia	12	0	0	
Manufacturing costs	13	0	0	
Cost per set of 25 pads	0	8	0	

As these pads are to be used for aseptic purposes, it is necessary to pack them in properly sterilized paper or plastic covers.

The demand for these pads was fairly heavy during the last World War and is likely to grow with the development of industries concerned with pharmaceutical or biological processes involving aseptic filtration. The manufacture of these pads can be profitably taken up by surgical goods manufacturers.

#### **PRODUCTION OF CITRIC ACID**

CITRIC ACID AND CITRATES ARE IMPORTANT chemicals with a variety of industrial applications. They are used in pharmaceutical preparations, in the food industries and, to a lesser extent, in dyeing and calico printing, silvering, engraving and in ink manufacture, Citric acid and citrates also find application in the plastics industry.

Citric acid is now manufactured almost entirely by the mould fermentation method. India's requirements of citric acid, averaging to 250 tons per annum, are met entirely by imports. The panel on Fine Chemicals, Drugs and Pharmaceuticals, in fixing a target of production for citric acid, assessed the country's annual requirement at 500 tons by 1957. In view of the rapid growth of the pharmaceutical industry and of the aerated waters' and soft drinks' industry in India, the target may be exceeded in the near future.

Investigations on citric acid fermentation carried out under the auspices of the Council of Scientific & Industrial Research have resulted in the isolation of a giant strain of *Aspergillus niger* with outstanding citric acid producing ability, and in establishing the conditions under which this fungus can be employed for the commercial production of citric acid. An important feature in the use of this organism is that no oxalic acid is produced during fermentation. The fungal mycelia, obtained in the process, has been used in the production of calcium gluconate.

The method consists essentially in fermenting technical glucose or cane sugar under controlled conditions, employing cultures of the special strain of *A. niger* mentioned above. Conversion of glucose to citric acid takes place in 9 to 10 days, after which the fermented liquor is filtered, concentrated and the citric acid allowed to crystallize out. The yield in terms of the weight of raw material used up is 70 per cent with glucose and 50 per cent with cane sugar. When molasses are used, the yields are not consistent.

#### RAW MATERIALS

- 1. Technical glucose or cane sugar.
- 2. Ammonium nitrate, potassium dihydrogen
- phosphate and magnesium sulphate. 3. Slaked lime and sulphuric acid (if alternative recovery process via calcium citrate is adopted).

#### EQUIPMENT

- 1. Fermentation chamber of cement concrete for one ton plant,  $112\frac{1}{2}' \times 70' \times 16'$ .
- 2. Fermentation trays of aluminium or sheet iron or wood suitably coated.
- 3. Sterilization vessel, wood.
- 4. Sedimentation wooden tank.
- 5. Filter presses (plate and frame type and rotary type).
- 6. Vacuum evaporators, either lead-lined or of stainless steel.
- 7. Settling and decolourization wooden tanks.
- 8. Jacketed enamelled iron crystallizing vessel.
- 9. Basket centrifuge.
- 10. Air-conditioner, and
- 11. Boiler.

CAPITAL OUTLAY ( for 1 ton of citric acid per day ) Re

1	Fermentation chamber @ Rs 10/-	1101
1.	per sq. ft.	1,00,000
2.	Air-conditioning equipment	40,000
3.	Evaporators	80,000
4.	Trays and other equipment	80,000
5.	Boiler	15,000
	TOTAL	3.15.000

#### **PRODUCTION COSTS**

#### (a) Glucose as Raw Material —

	ILD.	as. p.
1. Technical glucose, 1.43 tons, @		
Rs. $70/-$ per cwt.	2,002	0 0
2. Other chemicals	400	0 0
3. Power @ -/1/- per kW.	50	0 0
4. Steam, 17 tons, @ Rs. 10/- per ton	170	0 0
5. Depreciation @ 15 per cent	150	0 0
6. Insurance @ 2 per cent	20	0 0
7. Salaries and wages	160	0 0
Cost of 2,240 lb. of citric acid	2,952	0 0
Cost per lb.	1	50
(b) Using Cane Sugar as Raw Material -	-	
1. 2 tons of cane sugar @ Rs. 40/-		
ner cwt	1 600	0.0

2.	Other expenses [ as in (a) 2-7 ]	950	0 0
	Cost of 2,240 lb. of citric acid Cost per lb.	2,550	0 0

(The estimates are based on laboratory experiments.)

#### MANUFACTURE OF CALCIUM GLUCONATE

AS A RESULT OF THE WORK CARRIED OUT IN the laboratories of the Council of Scientific & Industrial Research, a simple enzymic process has been developed for the production of calcium gluconate. The method which is covered by Indian Patent No. 39441 and corresponding foreign patents, has the following advantages over the older fermentation methods: (i) avoids the use of live cultures and, consequently, no special precautions or equipment are required for sterilizing media, etc.; (ii) the equipment required is simple; (iii) fungal mycelia from citric acid or penicillin production are utilized in the process; and (iv) side reactions are avoided and, consequently, the purity and yield of gluconic acid are improved. Technical glucose or starch hydrolysate is employed as raw material.

#### **RAW MATERIALS**

- 1. Technical glucose or starch hydrolysate.
- 2. Calcium carbonate.

#### EQUIPMENT

- 1. Shallow pans ( total area, 50 sq. ft.) for culturing the organism.
- 2. Refrigerator (household) for storage of culture.
- Laboratory autoclave.
- 4. Aeration vessel made of wood with stirrers.
- 5. Filter press, wooden frames and plates.
- 6. Vacuum evaporator of stainless steel.
- 7. Shallow hemispherical crystallizing vessels with enamelled stainless steel anchor stirrers.
- 8. Centrifuge.
- 9. Storage vessel made of wood ;
- 10. Drving oven.
- 11. Grinder, and
- 12. Compressed air plant.

Labour and supervision

#### CAPITAL COSTS

Do oo o

	-	
<ol> <li>Cost of equipment for a plant capable of producing 250 lb. of crystalline calcium gluconate in two days</li> <li>Cost of building 2 000 so ft @</li> </ol>	1,0	0,000
Rs. 10/- per sq. ft.	20	0,000
Total capital cost excluding cost of land	1,2	0,000
WORKING EXPENSES	Rs.	as. p.
Cost of 500 lb. of technical glucose @		-
Rs. 70/- per cwt.	310	00
Cost of calcium carbonate	21	00
Sundry chemicals	12	00
Electricity and fuel	30	00
Depreciation @ 15 per cent	120	00
Insurance @ 2 per cent	16	00
Labour and supervision	60	0 0

Rs

Cost of 250 lb. of calcium gluconate 569 0 0 Cost per lb. of calcium gluconate 4 0

(The estimates are based on laboratory experiments.)

# NOTES & NEWS

#### Scientific Supplies Stores in India

THE LEADING ARTICLE PUBLISHED in a recent issue of Current Science puts forth a plea for the establishment of scientific supplies stores in India (1950, 19, 167). At present essential supplies, research equipment, glassware and chemicals are obtained by Government departments, research institutes and the universities through: (1) reputed commercial concerns, or (2) some government agency such as the Indian Stores Department, Director-General of Industries and Supplies, Trade Commissioners, Scientific Liaison Officers and Supply Missions attached to the Indian Embassies, etc. It is estimated that the cost of articles obtained through commercial concerns is  $2\frac{1}{2}$  to 5 times more than when the stores are directly imported. This is attributed to the absence of any central organizations for obtaining these supplies and arranging for their storage and distribution.

The establishment of regional scientific supplies stores, at im-portant centres, to be either directly in the charge of the Department of Scientific Research, Government of India, or to be managed by a private firm under the control of the Central Government, is suggested for speedy and economic procurement of scientific supplies. Research institutions. university laboratories, etc., within the orbit of each of these centres would be served effectively from Each of these these centres. centres would collect the requirements for the next two years of various institutions in their zones, secure currency releases for articles to be imported, call for quotations, arrange for inspection and stock the material. The laboratory or the department concerned would then merely arrange to draw from these stores its requirements as and when needed.

This procedure would save much unnecessary expense and delay in procurement of supplies and would effect a substantial saving in cost of materials as also in packing, freight, insurance costs, etc. More-

over, research and other institutions would be saved a lot of administrative work in arranging for these supplies.

#### Exhibition of Scientific Instruments

A WIDE VARIETY OF SCIENTIFIC instruments and associated apparatus designed for industrial and power application and scientific process control in industry were on display at the 34th Physical Society Exhibition held in April 1950 at the Imperial College, London.

Among the pick-up, or initial measuring devices, the emphasis was on instruments measuring radioactivity, e.g. an instrument measuring material thickness by radioactive means. Radiation measurement detection and apparatus, varying from simple Geiger-Muller counters to radioisotope analysers, was demonstrated. Among the amplifiers, 12 different designs of d.c. or a.c. amplifiers suitable for application to the measurement of small currents or potentials were incorporated. Some examples of new miniature electrical instruments covering all types of movements were shown and also a neat and small frequency meter with 5 vibrating reeds for use on 50 c./s. A new compact dynamometer has been produced. A small compact moving coil grapher in a metal case, a 3 in. wide chart with two paper speeds, was the most prominent of the graphic recording instruments. A compact single-pen operation grapher or marker, with an eight-day mains-driven drum chart, was on show. This was developed for statistical investigation of consumer demand when using water-heaters and similar appliances and records the time and duration of the load.

Three new instruments for process control were exhibited. The largest has a 10 in. wide chart with self-contained motor-driven pen and integral potentiometer balance. Full-scale deflection is given for 10 mV. For portable use, one type was shown which could be servo-operated. The whole equipment could be accommodated in a portable case with a disc chart record. The use of electro-sensitive paper was a feature of several other recording instruments.

Improved forms of voltage regulators and transformers were exhibited. A transformer for high frequency use, which can deal with loads of 500 VA., was notable for its small size. An equipment, designed for stabilization of a.c. voltages for accurate laboratory work, has a short-term stability within two parts in 10,000. A valuable accessory for control purposes was the vacuum switch. Two new magnetically operated models work with 15A a.c. or d.c. at 600V. respectively and the same current at 1,500V. in 10 milliseconds.

A powerful miniature selfstarting motor, having a torque of 0.75 in. per oz. at 3,000 r.p.m., was shown. Its larger prototype produces 6 in. per oz. at the same speed. Other designs with various terminable shaft speeds and torques were also available.

Some new portable test instruments for laboratory and industrial use were put on show. One portable wattmeter was capable of measuring power without breaking the circuit and is available in ranges from 50 kW. upwards; a plug-in ammeter movement was also available.

New additions to the wide range of insulation testers were made. One of these, hand operated, had a maximum test pressure of 2,500V. d.c. and six ranges from 50,000 megohms downwards, while another with a test pressure of 5,000V. d.c. had ranges down from 1,00,000 megohms.

Many demonstrations were suggestive of the future trends in development. The use of the Hall effect in germanium crystal was one example. Two prototype equipment was operated to show how accurate measurements of magnetic flux may be made using a tiny probe, 3 mm. square; this would be of immense help in investigations of complicated or small magnetic circuits (*Electrical Review*, 1950, No. 4).

#### Mercerization of Jute

THE TRANSFORMATION OF NATIVE cellulose (Cellulose-I) into the hydrate modification (Cellulose II) by the action of caustic soda on raw jute has been studied at the Textile Physics Laboratory, University of Leeds (Nature, 1950, **165**, 818).

The soda-cellulose obtained by treating raw jute without tension in caustic soda solutions of various concentrations was examined for the native modification which was found to be absent for concentrations greater than 10 per cent (w/w) but reappeared when alkali was washed off. The incomplete transformation, therefore, is not due to the failure of the alkali to penetrate into the cellulose crystallites but to at least partial reversibility of the nativesoda-cellulose reaction.

In ramie the conditions for a partial transformation of this kind are much more critical and appear to be such that only part of the cellulose has been acted upon by alkali. The differential behaviour from jute is ascribed to the effect of lignin in the latter, when perhaps by forming cross-linkages between the cellulose chains near the boundaries of the crystallites. it introduces an element of stability absent in the purer fibres. Another effect, which is observed only in jute, for a narrow range of concentrations of the alkali (about 10 per cent), is the formation of an abnormal soda-cellulose-I and is perhaps the consequence of extra crystallite cellulose-lignin linkages.

#### **Direct Plating of Aluminium**

A NEW PROCESS FOR DIRECT plating of aluminium with the help of a suitable etching agent has been developed at the Food Investigation Organization of the D.S.I.R., London (J. Electrodepositors' Tech. Soc., 1950, 26).

The relative efficiency of various etching agents was determined by immersing etched and washed specimens in a solution of iodine (containing 0.1 N iodine and 0.13N sodium iodide).

Etching solutions containing acid in combination with the chloride ion, viz. sulphuric ( $H_3O_4$ -HCl), hydrochloric (HCl NaCl, HCl AlCl<sub>3</sub>), acetic (CH<sub>3</sub>COOH-NaCl) and trichloracetic (CCl<sub>3</sub>-COOH), were the most effective in dissolving the surface film. Trichloracetic acid etches best taking only one or two minutes and leaving a very smooth surface free from pits. The addition of a hydroxy organic substance, e.g. dextrin to the etchant forms a soluble complex salt with alumi-

nium and prevents the precipitation of aluminium hydroxide or basic salts of aluminium. The best etching solution found so far consists of dextrin 50 gm., water 150 c.c., trichloracetic acid (melted crystals) made to 1 litre. The average loss of aluminium (99·3 per cent ) incurred during etching at 95°C. for 1 to 2 min. was 0·14 gm. per cm.<sup>2</sup> (1·3 gm. per sq. ft.) of surface area, equivalent to removal of 0·0005 cm. (0·0002") thickness.

The disadvantages of this etching agent are : (1) the cost is higher than that of the usual etchants; (2) the solution is rather viscous and the loss through drag-out is likely to be appreciable; (3) the fumes which arise at  $95^{\circ}$ C. are objectionable and steps must be taken to reduce or eliminate them; and (4) the operator needs to wear rubber gloves.

The activity of the aluminium at the time of plating has to be maintained by washing in an inert liquid. such as acetone, or it can be reduced by means of a controlled wash in water or a mixture of acetone and water. The results of tests on the effect of varying the time of immersion of etched aluminium in cold or boiling water on the electrodeposition of nickel (from nickel sulphate solution) showed that a brief rinse in boiling water, or 30 min. in cold water, permitted a good deposit. Cold water treatment gave thinner and better conductive films than those formed in boiling water. Further washing should be done with minimum exposure of the etched surface to air and the metal should not be allowed to dry before immersion in the plating solution.

The following have given the best results for plating copper and nickel as aluminium after the preliminary etching treatment in the trichloracetic-dextrin solution.

Chemical Displacement: Copper-The etched metal is washed in two separate lots of acetone, rinsed in a mixture of equal parts of acetone and water and immersed in a saturated solution of copper sulphate for 3 to 10 min. at room temperature. Nickel - The etched metal is treated as above and immersed in the following solution for about 15 min. at 95°C. : nickel sulphate 250 gm. ; nickel chloride 50 gm., ammonium citrate 100 gm.; Perminal BX 3 gm., made to 1 litre with water. The coatings thus produced have a good appearance and permit a fairly

strong soldered joint but are porous and may possibly serve as undercoats for an electro-deposited metal.

Electro-deposition : Copper-The etched metal is treated as above and plating is done in an agitated solution of the following composition for 2 min. at 50 amp. per sq. ft. and then 13 min. at 25 amp. per sq. ft. at room temperature, using a copper anode and a bath of copper sulphate 300 gm., boric acid 20 gm., Perminal BX 2 gm., made to 1 litre with water. Nickel-Treated as for copper and plating is done in the following solution for 30 min. at 50 amp. per sq. ft. at room temperature, using nickel anode and a bath of nickel sulphate 100 gm.; ammonium sulphate 50 gm.; boric acid 2 gm., made to 1 litre with water.

#### Stainless Steel Linings

THE COMPARATIVE MERITS OF stainless steel lined, stainless steel clad and solid stainless steel equipment and the welding techniques employed in these vessels are discussed (*Chem. Eng.*, 1950, **57**, 117).

Considering the wall thickness, the following linings are recommended as most economical : 0.25in. or under, solid stainless steel, a careful comparison of costs is necessary ; 0.25 to 0.5 in. solid stainless steel and stainless steel clad ; 0.5 to 1.0 in. stainless steel clad and stainless steel lined ; 1.0in. or more stainless steel lined ; 0.75 in. or below in conditions severely corrosive, solid stainless steel; 0.75 in. and above, conditions severely corrosive, either clad or lined construction.

Plug welding, strip welding, spot welding and poke welding are commonly used. The choice of a particular method is determined by the vessel design, operating conditions and skill of workmen.

Plug Welding — The method is easier to test for leaks and is found satisfactory in many cases. There is little opportunity for crevice corrosion if the plugs are properly sealed. Comparison of plug welding costs with those for strip welding method is of the order of 10 to 30 per cent more expensive than 2-bead butt weld, 5 to 15 per cent more expensive than 3-bead butt weld and lap weld with formed edges and about 15 per cent less expensive than the butt strap arrangement. The method, however, does not necessarily afford stronger attachment to the shell than the strip method; in fact there is more possibility of harmful dilution of weld metal in plug welds than in any of the strip methods except the single-bead butt weld.

Strip Lining-The major variations of method are : three-bead, two-bead and single-bead butt welds, butt straps, lap welds with formed edges and shingle method. In three-bead butt weld, laying of the cover bead is not easy and fit-up is rather exacting. It is slightly more expensive than lap weld with formed edges, and less expensive than butt strap. Twobead butt weld offers the best all-round combination of freedom from dilution and crevice corrosion and ease of fit-up at moderate cost. Single-bead butt welds have been successfully used in lining work but require the highest welding skill to securely attach the liner and seal the joints with one bead. Even then dilution and crevice corrosion are more likely and unsound welds may occur. Use of a butt strap is the most satisfactory although it is the most expensive on account of four welds required for each joint. Lap weld with formed edges is as satisfactory as the butt strap. Cost of forming is more than overcome by using only two beads per joint instead of four. The ridged effect, however, may cause some inconvenience in cleaning. Shingle method is a variation of lap weld with formed edges. The method is seldom used because it is difficult to make the lapping edge meet properly the flat surface of the lapped strip with the result that the welds are unsound and not crevice-free.

Spot welding methods have been quite successful, especially when the vessel interior is new and smooth. The method and the equipment are patented.

The poke welding gun, making use of the inert gas shielded arc principle, is a recent development. The gun fusion welds two lapped pieces of metal without the necessity of making holes in either piece. It is applied to only one side of the joint. The equipment is patented.

#### Petroleum from Oil Sands

THE DEMONSTRATION PLANT AT Bitumount, Canada, the site of Alberta's Oil Sands Project on the Athabaska river, is the only plant of its kind in the world extracting

petroleum from Alberta's oil-rich sands. These deposits constitute the largest single reservoir of petroleum in the world and are estimated to contain 100 billion to 500 billion gallons of oil. The separation and refining processes employed are described (*Chem. Eng.*, 1950, 57, 103).

The oil sand deposits consist of unconsolidated sand, clay and silt containing varying amounts of heavy asphaltic oil, 4 per cent to 16 per cent by weight. The sand particles are 50 to 200 mesh and the oil is largely in the form of thin films surrounding the sand grains.

The sand is fed to the plant through steam-jacketed screw conveyers after injecting it with high-pressure steam. Hot water is added to adjust the gross water content of the feed to about 12 per cent by weight. A rotary screen removes limestone and other foreign bodies. The oil sand passes through the screen as a heavy pulp containing about 25 per cent by weight of liquid. Adjusting a weir at the discharge end regulates retention time in the mixer. During mixing the oil film surrounding each sand grain is broken and the sand is wetted with water. The tiny air particles so formed coagulate to form droplets. These are aerated by agitation in the mixer.

In the distributor, the oil, water and sand are mixed with 200°F. recycle water circulating at 400 gal. per min. The sand enters the separation cell as individual grains and oil inclusion is prevented. Conditions in the cell are quiescent, so all but the finest mineral matter settles rapidly and is carried along through the bottom to the foot of a twin-screw dewatering conveyer which lifts the sand above the liquid level in the cell before discharging into the tailings receiver. The oil-water interface in the separation cell is maintained slightly below the reach of three sets of rotating paddles that carry the oil out of the cell. A conventional thickener removes almost all fine clay particles mixed with the oil sand.

Oil leaving the separation cell contains 25 to 30 per cent water and 4 per cent mineral matter. This is cleaned up by diluting, setting and flash vaporizing the water. Diluent is a  $30^{\circ}$ -API distillate produced in the refinery. Water in the diluted crude is removed by heating to  $400^{\circ}$ F.

under 20 to 30 p.s.i. back pressure in a conventional tube still and flashing. Distillate flashed with water is recombined with dry crude from the bottom of the flash water.

Refining is restricted to a mild vis-breaking operation (1) recovery of diluent added to facilitate cleaning of the crude; (2) production of new diluent to balance production losses; and (3) production of fuel oil for the power house, refinery heater and dehydration heater.

Dry diluted crude oil is heated to 825°F. under 50 p.s.i. back pressure and flashed. Overhead from the flash tower is fractionated into naphtha, distillate, light gas oil and heavy gas oil. Heavy gas oil is recombined with the flash tower bottoms and the blend used as fuel.

Pilot-plant trials of a cold water separation process and a fluidizedbed operation are under investigation.

#### Recovery of Stainless Steel Scrap

A NEW TECHNIQUE, OXYGEN LANCing, which speeds up the production, improves the quality and reduces the cost of stainless steel, was discussed at a recent meeting of the British Iron & Steel Federation (*Chem. Age*, 1950, **62**, 819). The technique is regarded as an important development in the manufacture of stainless steel since its initial discovery. The new process makes possible the utilization of stainless steel scrap.

The scrap is melted in an arc furnace at 3,000°C. and pure oxygen is blown into the steel through a long, thin pipe thrust through the door of the furnace. The oxygen oxidizes silicon and manganese which tend to increase the temperature of the molten steel. Subsequently, carbon ( contamination from the electrode ) is burnt or oxidized and its content can be brought down to 0.05 per cent. During oxygen injection some chromium is oxidized, thereby generating more heat, and this chromium mainly passes into the slag. At a later stage, this chromium is made to pass back into the molten steel to the extent of about 96 per cent. Thus fourfifths of the chromium in the metal is retained when a stainless steel with 18 per cent chromium is made, and all of it is retained when processing a "rustless" steel with 12 per cent chromium, thereby effecting a considerable saving of the expensive metal and obviating the necessity of rebuilding the chromium content. Great saving in electric current is also effected by this method; 10 minutes of oxygen lancing completes the operation which wouldnormally take 30 to 45 minutes by the old method. The steel made this way takes high polish quickly.

#### **Rapid Analysis of Silicates**

THE STANDARD METHODS OF ANAlysis of clays and other silicates are time-consuming and are not very accurate. Colorimetric and spectrographic methods developed and perfected at the Royal Institute of Technology, Stockholm, and at the University of Ghent respectively (*Research*, 1950, 3, 74) offer rapid and simplified analytical techniques.

Colorimetric Method — Three solutions of the clay sample are prepared. The first solution, for determination of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> is obtained by fusion of 0.2 gm. of clay with sodium carbonate, precipitation of silica by hydrochloric acid and separation from chlorides of iron, aluminium and titanium, and dissolution of silica in sodium hydroxide to form sodium silicate. Each filtrate is made up to 1 litre. Silica is determined colorimetrically in this solution by the yellow colour produced on forming silicomolybdic acid, the AlgO3 by the yellow colour formed with "aluminon", the Fe<sub>2</sub>O<sub>3</sub> by the red colour formed on addition of potassium thiocyanate and the TiO<sub>2</sub> by the yellow produced by hydrogen peroxide.

The second solution for the determination of calcium and magnesium oxides is obtained by the fusion of 0.5 gm. clay with sodium carbonate, precipitation of silica with hydrochloric acid, addition of hydrogen peroxide to dissolve MnO<sub>2</sub> as the chloride, precipitation of iron, aluminium and titanium as their hydroxides by ammonia, and filtration of the solution into a 250 c.c. graduated flask. The precipitate is re-dissolved in hydrochloric acid, reprecipitated by ammonia and washed with four portions of 2 per cent ammonium chloride solution. The washings are added to the filtrate in the 250 c.c. flask, the whole acidified with hydrochloric acid and made up to 250 c.c.

Calcium oxide is determined by precipitation as calcium oxalate and titration with decinomal potassium permanganate. Magnesium oxide is determined colorimetrically by means of titanium yellow which, on co-precipitation with magnesium hydroxide, produces a red colouration.

The third solution, for determination of alkalies, is obtained by treating 2 gm. of clay with hydrofluoric and sulphuric acids, followed by neutralization with ammonia to pH 5 and washing of the precipitate with hot water. The filtrate is made of 200 c.c. in a graduated flask. Sodium oxide is estimated by precipitation with uranyl acetate and estimated colorimetrically by the brown colouration formed with uranium in presence of hydrogen peroxide and ammonium carbonate. Potassium oxide is estimated by precipitation with chloroplatinic acid and addition of potassium iodide, when a red colouration is

formed due to the PtI<sub>6</sub>2-ion.

Complete analysis of clay with this method can be finished within 8 hr. The results are comparable in every way with the results obtained by gravimetric methods. The method is applicable to the analysis of mica, feldspar, sands, slags from blast furnaces and cement. It requires a combined colorimeter and nephelometer with two photoelectric cells of a selenium barrier layer type, connected by a compensating circuit to a galvanometer as null point indicator.

Emission Spectrographic Method - A master solution for determination of  $Al_2O_3$ ,  $Fe_2O_3$ ,  $TiO_2$ , CaO and MgO is prepared by dissolving 1 gm. clay in hydrochloric and sulphuric acids in a platinum crucible so as to volatilize the silica. After evaporation to dryness the residue is taken up in hydrochloric acid and warmed to dissolve the salts completely. This is again evaporated to dryness and redissolved in 10 c.c. of a standard copper sulphate containing 2.5 gm. copper in 1 litre of 2 N HCl. 0.02 c.c. is evaporated at 90°C. on a Hilger graphite electrode of 5 mm. diameter with a flat surface, previously cleaned by a preliminary discharge for 1 min. The upper electrode is also of graphite and the excitation is by intermittent arc. The intensity of the lines is compared with that of the copper which furnishes

an internal standard. The curves produced permit the determination of  $Al_{9}O_{8}$ ,  $Fe_{3}O_{3}$ ,  $TiO_{8}$ , CaO, MgO and Na<sub>2</sub>O in various concentration rays. Very low concentrations are obtained on further simple treatments of the master solution. Silica is determined by difference.

More rapid determinations may be carried out by working directly on a specimen of finely powdered clay. 0.1 gm. clay, 0.4 gm. copper oxide and 0.5 c.c. alcohol are mixed in an agate mortar and ground finely until the alcohol has completely evaporated. The mixture is dispersed in 1 c.c. alcohol and, in order to deal with the whole of the suspension, it is withdrawn by means of a glass tube of diameter less than that of the flat electrode and placed on the electrode; the alcohol disappears by absorption and evaporation. Except sodium and potassium oxides (which cannot be determined by this method ), the other oxides can be determined in concentrations of 1 per cent and below. A maximum of 15 to 20 min. is required for the determination of the principal constituents. Reproducibility is excellent, with a mean error of 3 to 5 per cent.

#### **New Rabies Vaccine**

A NEW VACCINE FOR PROTECTING dogs against rabies is reported (*Sci. Newsletter*, 1950, **57**, 279).

The vaccine is made from a live rabies virus modified by growing in chick embryos and, unlike the older vaccines, does not contain any mammalian brain or spinal cord tissue. The vaccine provides a high degree of immunity. Dogs inoculated with a single injection were found to be completely resistant to virus infection for 5 to 27 weeks. The chick embryo vaccine is highly stable and maintains its immunizing capacity for at least 18 months.

#### **Improved Metal Cleaner**

A NEW AND SIMPLE METHOD FOR making and using di-phase metal cleaners with very good detergent properties is based on a system in which the water and solvent remain as separate layers.

The solvent consists of a chlorinated 'solvent such as trichlorethylene to which is added mineral spirits to give a final specific gravity greater than the water layer which remains on top of the solvent. Phosphatesare frequently added to the water to help in removing water-soluble dirt from the metals.

The efficiency of this system depends on the addition of polyethylene glycol 400 (di, tri) Ricinoleate S-556U (*Glyco Products Co. Inc.*, U.S.A.) about 0.5 per cent to the solvent. S-556U acts as an organic solvent wetting agent for the metals, helping to remove grease and oils from the metals rapidly. Not only does it act as a preferential wetting agent in the solvent for metals, but also as an emulsifying agent when the metal parts are raised through the water layer, causing more effective removal of trace grease and other adhering dirt.

Cleaning by this di-phase system is done simply by dipping the metal parts through the water layer into the solvent and then, after a short time, bringing them up through the water layer with a minimum of agitation. Heating is not resorted to at any stage, thus eliminating toxic fumes, fire hazard, loss of solvent and change in composition of the two phases.

#### Sheep-marking Composition

A NEW SHEEP-BRANDING FLUID, which eliminates special processing and does not harm the sheep or the wool, has been produced by the Australian Commonwealth Scientific & Industrial Research Organization after seven years of research and investigation (*Agr. Newsletter*, Australia).

The composition is prepared as under: Lanolin (26 lb.), resin (10] lb.), stearic acid (7 lb.) and tallow (3] lb.) are melted at 230°F. The molten mixture is poured slowly into an ammonia solution (sp. gr. 0.88) with slow mechanical stirring. A preservative such as toluene or oil of eucalyptus phellandra (1 lb.) is then added with stirring and a suitable pigment (5 lb.), e.g. Monolite Scarlet R.N.S., Monastrol Blue B.S. weak, or carbon black is mixed in. The product keeps well without any sedimentation for at least three years and its marks on sheep are readable for as long as 12 months.

#### High Frequency Induction Furnace

A HIGH FREQUENCY 5 kW. INDUCtion furnace for metallurgical research has been designed at the Research Laboratories of the *General Electric Co.* Two types are



FIG. 1 — HIGH FREQUENCY INDUCTION FURNACE.

illustrated (Fig. 1). The 5 kW. one is the smaller of the two.

The furnace is housed in a sheet metal cabinet with a top of heatresisting material. A lever operating through a notched gate is used to raise or lower the crucible and during operation the furnace is covered with a lid which has a small hole to allow for the observation of the charge or for the insertion of a pyrometer. The lid, table top and removable rear panel are all fitted with safety switches to prevent power being switched on when they are not in a position. A graphite and refractory crucible is used with the equipment. Power for the furnace is supplied from a 5 kW. high frequency heating generator and the overall consumption, when the equipment is in full operation, is 10 kW. from a 400 to 440V. three-phase supply. Typical performance figures for the plant are: 2 kg. of copper can be melted in about 15 min. and 1 kg. of steel in 12 to 15 min.

#### Chemical Survey of Australian Plants

MORE THAN 750 AUSTRALIAN plants have been examined as sources of drugs and chemicals in a recent survey conducted jointly by the C.S.I.R.O. and the Universities of Melbourne, Sydney and Queensland. The survey has been specially initiated to search for plants which might provide a starting-point for the manufacture of cortisone, which shows promise in the treatment of rheumatoid arthritis. Plant saponins, it is suggested, may make possible the large-scale production of this substance.

One hundred and forty-five of the plants examined seem likely to contain alkaloids which are being identified. Eleven plants have been found to contain prussic acid and may be dangerous to stock under certain conditions of growth.

#### **Timber for Packing Cases**

A PRELIMINARY SURVEY OF INDIAN timbers suitable for the manufacture of packing cases has been made in a pamphlet (*Indiam Forest Leaflet No.* 106), recently issued by the Forest Research Institute, Dehra Dun. The survey shows that there are extensive possibilities for the manufacture and supply of box shooks from indigenous timbers.

Besides listing the various timbers which are already in use for making packing cases, names and descriptions of other woods which are considered suitable but have not been tried so far are given. In all there are about 125 species distributed over various regions as under: (1) North-west Region (East Punjab, Himachal Pradesh, etc.), 8; United Provinces Region, 15; East Indian Region (including Bengal, Assam, Andamans, Bihar and Orissa), 50; Central Indian Region (including the Madhya Bharat, the Deccan and Deccan States), 10; South-eastern Region (including the Western Ghats, Malabar, Mysore, Coorg, Cochin and Travancore), 40.

Three classes of timbers have been considered : woods with an air-dry weight of 20 to 30 lb. per cu. ft., 30 to 35 lb. per cu. ft. and 35 to 40 lb. per cu. ft. The methods of seasoning and protective measures against attacks from insect pests are described.

#### ISI Committee on Weights & Measures

THE SPECIAL COMMITTEE ON Weights and Measures set up by the Indian Standards Institution in 1949 to recommend ways and means for standardization of the numerous systems of weights and measures prevalent in the country has recommended the adoption of the metric system in its report recently published by the ISI.

Several committees since 1801 have been appointed by the central and provincial governments but the results achieved did not go beyond the Regulation VII of 1833 which fixed the weight of the Furruckabad rupee at 180 grains English weight, the Government of India resolution of 1854 which fixed the standard railway maund at 40 seers ( $82_1^2$  lb.); and the Measure of Length Act of 1889 which fixed the imperial yard with its subdivisions in feet and inches as the primary unit of length. The report of the ISI. Committee, which is based on a countrywide consensus of opinion of scientists, technologists industrialists and Government departments, recommends the adoption of the metric system as the standard system. The Committee have outlined detailed programmes for systematic and gradual adoption of the metric standards in everyday trade activities, a complete change-over being extended over a period of 15 years in three distinct stages, viz. a preparatory stage of 3 to 5 years for intensive dissemination of information on

the new system, followed by a change-over stage of about the same period and a final stage for overall transition. After that period, the only system accorded legal recognition would be the metric system.

The introduction of decimalized currency, in which the weights and dimensions of coins should be related to the metric system, is also recommended in the early stage of the change-over.

The Committee endorses the decision of the ISI to accept the recommendation of the Empire Scientific Conference of 1946 that whenever units are expressed in scientific literature in systems other than the metric, metric equivalents should always be indicated. This practice should be immediately adopted as a general rule in all technical publications issued in India. With regard to the nomenclature of the new system, the use of international nomenclature is recommended. For the implementation of the Committee's recommendation, the report stresses the need for an early central legislation for the recognition of the metric system and the creation of an agency to co-ordinate all related measures for the change-over.

#### Spun Silk Industry in India

THE DEVELOPMENT OF THE SPUN silk industry in India and its manufacture from silk waste are reviewed in a bulletin (No. 8) issued by the Central Silk Board, India.

In India, the production of spun silk commenced in 1939 with the establishment in Mysore of the Mysore Spun Silk Mills Ltd. The annual production of silk waste in India is estimated at about 11,00,000 lb. of which Mysore contributes nearly 75 per cent. The factory is the only silkspinning factory working in the whole of India and South-east Asia. The annual consumption of silk waste by the factory has also gone up to 6 lakh lb. from 4 lakh. Steps are now being taken in pursuance of the recommendations of the Silk Panel appointed by the Government of India to establish another spun silk factory in Bihar.

The cost of production of spun silk in India, however, continues to be high. This is accounted for primarily by the poor quality of silk waste which cannot withstand

the severe combing operations of the waste-silk spinning machinery and also by the coarse denier waste. For this, it is, therefore, necessary that production of higher grade silk (which is conducive to the production of high grade waste) is expanded as rapidly as possible to displace the crude *charka* silk. A blending of superior long staple foreign waste with the short staple Indian waste is capable of producing the best spun silk yarn, but this is not possible at present owing to difficulty of imports from abroad. In this connection the analogy of the Indian woollen industry is cited. There is no import duty on import of raw wool for manufacture of fine quality wear. In order to help the Indian spun silk industry to similarly produce better quality goods, it is neces-sary to allow the mills to import superior silk waste from abroad and remove the present productive duty imposed thereon. This course is recommended only until larger supplies of filature waste become available in the country itself. It is possible to improve the quality of *charka* waste, to a certain extent, by removing at the time of its preparation the pupal and extraneous matter from the waste and drving it on bamboos or other hangers instead of on the ground. It is also necessary to study and take note of the advances made in the technique by other sericultural countries like Japan and Italy which are the main competitors in the field and get suitable machinery which would help to bring down the cost of production and at the same time increase the yield and better the quality of the goods.

#### **Protective Duties**

BASED ON THE RECOMMENDATIONS of the Indian Tariff Board, the Government of India have revised the duties with respect to certain industries. The Indian Tariff Board has been examining the progress of several industries with special reference to the renewal of protection. The Board has also undertaken an inquiry into the claims of some industries for protection. The following table summarizes the recommendations of the Board (as accepted by the Government of India) with respect to certain industries formulated during recent surveys.

### NOTES & NEWS

INDUSTRY	TARIFFS RECOMMENDED
Antimony	Protective duties of 30 per cent and 20 per cent <i>ad valorem</i> on antimony and crude antimony respectively, up to March 1952
BICHROMATES	30 per cent ad valorem protective duty up to March 1952
Calcium Chloride	Protective duty of Rs. 3/4 and Rs. 4/14 per cwt. on British and non- British manufactures respectively and 10 per cent <i>ad valorem</i> on imports from Burma up to March 1952
Cocoa Powder & Chocolate	Protective duty of 30 per cent ad valorem up to March 1952
Cotton & Hair Belting	Protective duty of $7\frac{1}{2}$ per cent ad valorem up to March 1952
Cotton Textile Machinery	Protective duty of 10 per cent up to March 1953
Dry Battery	Protective duty of 30 per cent ad valorem on imports up to December 31, 1951
ELECTRIC MOTORS	Protective duty of 10 per cent ad valorem on motors up to 20 h.p. up to March 1953
Ferro-silicon	Duty of 184 per cent ad valorem up to March 1951
Glass & Glassware	Protective duty of 45 per cent ad valorem on sheet glass of all gauges up to March 1952
Glucose	Protective duty of 30 per cent ad valorem to continue up to March 31, 1951
NON-FERROUS METALS	Existing protective duty on various items to continue up to March 31, 1951
Phosphates & Phosphoric Acid	Revenue duty of 64 per cent ad valorem on phosphoric acid and 37 per cent ad valorem on sodium phosphate from April 1950
Plastics .	(i) Protective duty of 30 per cent ad valorem on phenol-formaldehyde moulding powder; (ii) refund of import duty on phenol formal- dehyde and hexamine to moulding powder manufacturers producing 200 tons or more per annum; (iii) 20 per cent ad valorem duty on cellulose nitrate, rennet casein and polyvinyl chloride resin; (iv) protective duty of 30 per cent on electrical accessories imported from U.K.; (v) 20 per cent duty on raw celluloid up to March 1953
Potassium Permanganate	Protective duty is replaced by a revenue duty with effect from April 1, 1950
Sewing Machines	Protective duties of 24 per cent and 30 per cent <i>ad valorem</i> on imports of British and non-British manufactures respectively up to March 1953
SMALL TOOLS	5 per cent ad valorem duty on special steels of British manufacture and 15 per cent ad valorem on steels other than British manufacture
Soda Ash	Protective duty of 30 per cent <i>ad valorem</i> preferential and 40 per cent standard granted Duty of 40 per cent <i>ad valorem</i> preferential and 50 per cent standard. A subsidy at Re. 1 per cwt. to be granted to soda ash produced in the country. Protection up to March 1953
Starch	20 per cent ad valorem protective duty up to March 1952
Stearic & Oleic Acids	Protective duty of 30 per cent ad valorem up to December 31, 1951
STEEL BELT LACING	Protective duty discontinued
Sugar	Protection withdrawn
Wood Screws	30 per cent ad valorem duty to continue up to March 1952

#### Conservation of Museum Objects

THE INTERNATIONAL INSTITUTE for the Conservation of Museum Objects recently established with its headquarters in London fulfils an urgent need for an organization aimed at setting and maintaining a high standard of skill and competence in the conservation of all kinds of valuable and artistic material and national treasures. Its office-bearers are authorities of international reputation in their respective fields and Dr. G. L. Stout, formerly Head of the Department of Conservation and Research, Fogg Art Museum, Harvard University, is its first President.

The Institute will help to bring about collaboration between experts of different nations and those intimately interested in conservation of museum objects. The organization will pay particular attention to the training of personnel in the theory and practice of conservation and co-operate with all institutions providing for such instruction. The Institute proposes to publish from time to time articles on technical studies in the field.

A booklet giving full details of the Institute's aims and regulations is in preparation (*Nature*, 1950, **165**, 903).

#### **Indo-Pacific Fisheries Council**

AT ITS SECOND ANNUAL MEETING at Cronulla (Australia) in April 1950, the Indo-Pacific Fisheries Council elected Dr. J. D. F. Hardenberg of Indonesia as its Chairman and Dr. A. V. Villaotolid of the Philippines as its Vice-Chairman for 1950-51. Technical committees and various ad hoc sub-committees were set up to carry out investigations on Tunas, neritic-pelagic group of fish, fish culture, transplantation of fish, planktology, hydrology, taxonomy and survey of fish industries in this region.

One of the Council's major projects is the compilation of a register of the projects, institutions, and personnel concerned with fisheries work in the Indo-Pacific region.

#### Australian Journal of Marine & Freshwater Research

THE COMMONWEALTH SCIENTIFIC and Industrial Research Organization has decided to publish the Australian Journal of Marine and Freshwater Research as a medium for the publication of research papers on the results of original investigations on sea, estuarine and freshwater fisheries and cognate subjects. It is expected that two issues of the journal will be published each year; each issue will cost 7s.~6d.

#### The New Publication — "Chemical Age"

"CHEMICAL AGE", SERIES I (TECHnical Press Publication, Bombay, April 1950, Price Rs. 10), is the first of a series of comprehensive biennial issues covering the chemical process industries of India. The inaugural number features a wide range of review articles dealing with various developmental and technical aspects of the heavy chemical, fine chemical, synthetics and fertilizer industries. Special emphasis is laid on artificial manures and pesticides, both vital to the agricultural economy of the country.

The articles are contributed by eminent scientists and writers all specialists in their respective fields. The annotated bibliographies, graphical flow sheets, production charts and abundant illustrations of plant and equipment have increased the usefulness of the volume.

#### **Board of Engineering Research**

THE PRESIDENT, INDIAN COUNCIL of Scientific & Industrial Research, has constituted a Board of Engineering Research to function as an Advisory Body to the Governing Body of the Council. The following have been nominated to serve on the Board up to March 1953: Shri A. N. Khosla, Chairman, Shri Kasturbhai Lalbhai, Shri S. Moolgaokar, Shri S. L. Malhotra, Shri Ramesh Chandra or alternately Shri J. M. Trehan, Shri F. C. Budhwar, Shri Jang Bir Singh, Shri B. S. Puri, Dr. S. S. Bhatnagar, Shri G. D. Birla, Shri K. D. Jalan, Sardar Teja Singh Malik, Shri D. V. Joglekar, Prof. C. A. Hart, Prof. M. S. Thacker, Prof. A. Viswanathan, Director, National Metallurgical Laboratory, Dr. K. S. Krishnan and an engineer as Secretarv.

The Board's main functions will be to initiate and co-ordinate research work in various branches of engineering.

#### Government of India to Purchase Uranium Stocks

THE GOVERNMENT OF INDIA HAVE decided to purchase all stocks of uranium available with dealers and mine-owners. A minimum uranium content equivalent to 10 per cent by weight of uranium oxide is required in the ores or concentrates which will be paid for at a minimum rate of Rs. 9 per lb. of contained uranium oxide. Uranium dealers and mine-owners should contact the Secretary, Atomic Energy Commission, Department of Scientific Research, New Delhi.

#### Announcements

Prof. P. Maheshwari, Head of the Department of Biology, Delhi University, has been elected Chairman of the Plant Embryology Section of the International Union of Biological Sciences for a period offive years. Prof. Maheshwariwill prepare a directory of the scientific institutions and individuals carrying on research work in plant embryology and will also conduct a census of recent research in this field.

Dr. R. C. Sahney of the National Physical Laboratory, New Delhi, has been granted the 1851 Empire Exhibition Scholarship for 1950. The scholarship awarded to him is for research in Physical Chemistry at Cambridge University.

The Lady Tata Memorial Trust have announced the following awards of Scholarships and Grants for the year 1950-51 on the occasion of the 1950 death anniversary of Lady Meherbai Dorabji Tata. The International Awards totalling  $f_3,000$  for research in diseases of blood with special reference to Leucaemias are made to Dr. Edith Paterson (England), Dr. Pascou Atanasiu (France), Dr. J. Bichel (Denmark), Dr. E. Kelemen (Hungary), Dr. Charles Oberling (France), Dr. Günther Shallock (Germany), Dr. Astrid Fagraeus and Dr. Bo Thorell (jointly) (Sweden), Dr. M. Henri P. L. Febure (France) and Dr. George Discombe (England).

Indian Scholarship's of Rs. 250 per month each for one year are awarded to Mr. P. R. Srinivasan (Coonoor), Mr. Siva Sankar (Madras), Dr. Dharmendra, Dr. Pal Singh (Agra), Dr. Srinivasa Sriramacharyulu (Andhra) and Dr. (Miss) Sunita Popatlal Bharani (Bombay).

#### INDIAN CENTRAL COTTON COMMITTEE

THE ANNUAL REPORT OF THE TECHNOLOGICAL Laboratory of the Indian Central Cotton Committee ending 31st May 1949 records steady progress in technological, applied and fundamental research. Some of the investigations completed during the year have been of direct benefit to the cotton breeder.

One of the important items of work completed during the period under review relates to the estimation of spinning values of Indian cottons from a knowledge of their chief fibre properties. Regression equations and correlation coefficients have been worked out for G. hirsutum, G. herbaceum L. Var frutescens (Delile) H & G., G. arboreum Var neglectum forma indica and G. arboreum Varneglectum forma bengalensis. By using these formulae a cotton breeder can now easily estimate the spinning value of any new strain with a fair degree of accuracy from its fibre properties. The partial correlation coefficients have also been calculated to assess the relative importance of the fibre properties.

Pre-cleaning and ginning tests have been carried out on Mathio cotton with saw gin, single roller gin and double roller gin and the lint obtained was tested for fibre length and spinning value. Saw gin gave about 1.50 per cent lower ginning percentage than either single roller or double roller gin. The power consumed in double roller gin was practically the same as that in the saw gin for an equal weight of seed cotton, while in single roller gin it was about twice as much. Yarns spun from samples ginned in the saw gin were, on the average, stronger than those spun from the other two gins. An apparatus specially designed and calibrated in the laboratory was used for the purpose of deter-mining the strength of attachment of fibres to the seed. These studies showed that the strength of attachment of the fibres to the seed was very much greater at the micropylar end than those attached to other regions.

A new gin suitable for small samples has been designed in the Laboratory. This gin is of the Macarthy type having a roller length of 7" with a chain drive on ball bearings and is designed both for hand and for electrical drive. The machine, even in its wooden model, has been found to be quite satisfactory and can conveniently be used in place of Platt's hand gin recommended in the standard ginning technique for small samples.

Studies on the effect of agronomical factors on the fineness of cotton were completed during the year. Ammonium sulphate applications have been found to lower the mean fibre length, fibre maturity, swollen wall thickness, bundle strength and intrinsic strength. Application of manure just before sowing proved more useful to fibre length. 40 lb. of nitrogen gave the highest bundle strength value when broadcast 3 weeks before sowing on high fertility soil. On the whole, doses of nitrogen over 20 lb. are harmful to fibre properties.

A systematic study of the incidence of structural deformities such as binds and spurs and apical ends on the standard Indian cottons was made. Observations on the incidence of (i) hairiness of leaf-bits found in raw cottons, (ii) immature and insect-attacked seeds in the Kapas and (iii) neps at various stages of yarn manufacture were included in the investigation. A significant positive correlation between the percentage by weight of neps in the sliver and the number of neps per gm. of the yarn, counted according to either of the two methods, shows that the quantity of neppy material taken out while preparing the sliver can be taken as an index of the neppiness of the cotton.

A study on the causes of abnormal spinning performance in Indian cottons is being conducted at the Laboratory. It has been found that the roving generally has a mean fibre length less than that of the raw cotton and possesses a greater fibre length irregularity percentage and is about 10 per cent stronger. The decrease in length and regularity in the roving is pronounced in the case of Indian cottons whose staple length is about 0.93" and over.

The use of polarized light for evolving a quick method of determining fibre maturity was investigated. Under the polarized light the mature fibres appear yellow and green, while the immature ones appear pink and blue. Based on this observation an efficient and a quick method has been worked out.

Routine and other tests were carried out on 1207 cotton, yarn and cloth samples received from cotton breeders and the cotton trade and industry. Comparative mill and laboratory tests were carried out on Jayawant and Jaydhar varieties as regards their mean fibre length and spinning performance. The examination of the results obtained for the three seasons, 1944-47, showed that Jaydhar yields distinctly stronger yarns than Jayawant in all localities. The mill tests also gave the same result except in the case of Transition Tract and Eastern Tract. Comparative tests on Ladog 1 and Lazmi (9-3) showed that Lazmi yields stronger yarns.

Showed that Laxmi yields stronger yarns. Standard Indian cottons of the 1947-48 season were subjected to detailed fibre and spinning tests and a bulletin containing the results of these tests was published during the period under review. Four cottons, Jayawani, V 434 (Akola), Gaorani 6 and Cambodia Co. 32, showed an improvement over their performance of last season.

#### **IRRIGATION RESEARCH IN MADRAS**

THE ACTIVITIES OF THE IRRIGATION RESEARCH Station, Poondi, Madras, reviewed in its annual report for 1948, show considerable progress in a number of research projects investigated during the year. The studies have contributed to increased efficiency in the design of works and considerable reduction in their cost has been effected.

In river control studies, experiments on a 1/150 scale model of the Godavari for testing the alignments of the cellular dams for the Rampadasagar Project were pursued with a tilted bed and data obtained for application to bed movement in the proto-type. A large number of anicuts was designed and tested. Novel features were introduced in these designs with a view to increase their efficiency in performance and economy in construction. Savings of Rs. 12,000 and Rs. 20,000 were effected on the Shambipettai odai and Uttiramerur anicuts respectively. Another saving of nearly a lakh of rupees was made on the Jedarpalayam anicut. The design of stilling basins with one or two rows of floor blocks and with or without buttresses evolved at the station has proved very efficient as a dissipating arrangement for a spillway dam. Adjustability to variations in discharge and tail water levels to a very wide range is particularly satisfactory in this design. Considerable extension of irrigated area was effected by remodelling the existing sluices so as to increase the discharge drawn through them. As a result of tests on earth dam models, a large portion of the Lower Bhavani Dam, originally proposed to be constructed in masonry, is now proposed to be constructed in earth. The reduction in cost due to this change-over is likely to be of the order of Rs. 50 lakhs.

Studies conducted on building materials and construction led to the adoption of cheap and easily provided substitutes in place of the conventional materials that have become costly and difficult to procure. Soil-cement blocks, cement-concrete slates and tiles, reinforced cement concrete frames for doors and roof trusses, etc., have been satisfactorily used for many buildings in the project camps with great economy.

#### SCIENTIFIC ACTIVITIES OF THE NATIONAL METALLURGICAL LABORATORY

A FEW OF THE PROBLEMS UNDER INVESTIGATION IN the National Metallurgical Laboratory are: Electrolytic polishing and etching of ferrous and nonferrous metals and alloys and the detection of temper brittleness; a study of austenitic grain size control of nickel-chrome high alloy steels; anodizing of aluminium and its alloys, and the production of titanium tetrachloride from Indian ilmenite. The characteristics of Indian moulding sands are being studied.

Experiments carried out to convert low-grade manganese ores (from Madhya Pradesh) into manganese sulphate have yielded encouraging results. Attempts have been made to prepare electrolytic manganese and high purity manganese dioxide from manganese sulphate.

A scheme on the production of beryllium from beryl on pilot-plant scale, based on a process developed by the *Beryllium Corporation*, has been prepared.

Investigations have been carried out on the corrosion of stainless steels in solutions containing nitric and sulphuric acids, electrolytic recovery of nickel, from silver refinery waste and electro-refining of copper from cuprous chloride solutions.

The Refractories Division has initiated investigations on the production of sillimanite, zircon and carbon refractories. Preparation of carbon electrodes is also being investigated.

Work on the measurement of wear resistance of steels under varying service conditions will be undertaken shortly. Preliminary work on the production of iron-aluminium-cobalt sintered magnets by the powder metallurgy method is in progress. For this purpose experiments are being conducted for preparing an iron-aluminium master alloy by the sinter method.

Ore-dressing Laboratory problems under investigation are: (i) separation of cryolite from carbon dust produced in the aluminium reduction furnaces at Jakaynagar, (ii) concentration of ilmenite sands, and (iii) beneficiation of low-grade chrome ore from Dodkanya, Mysore, and other sources.

#### DICTIONARY OF ECONOMIC PRODUCTS & INDUSTRIAL RESOURCES OF INDIA

THE FIRST VOLUME OF THIS ENCYCLOPAEDIA COVERing letters A and B, which was published in January 1950, has been very favourably reviewed in many of the leading scientific journals of the world. The second volume covering the letter C is now in the press and will be published in about a month. Like the first volume it is divided into two parts : one dealing with "Raw Materials" and the other with "Industrial Products". The raw materials volume consists of about 300 main articles, including tea, coffee, coconut, citrus, cinchona, coal and clay and is profusely illustrated with text illustrations and plates. The part dealing with the industrial products is also profusely illustrated. This part includes articles on canning, cotton textiles, ceramics, coir, etc.

The Office of the Dictionary of Economic Products is now busily engaged in compiling articles for the third volume which will be published in 1951. In addition to the main work of compiling the Dictionary, this office has been acting as a clearing house of scientific information on raw materials and industrial products of India.

# 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 July 1950.

#### Paints, Varnishes & Lacquers

- 41456. Extraction of titania from titanium-bearing materials: Treating the raw material with sulphuric acid and a bivalent metal or its oxide or sulphate and thereafter igniting the double sulphate to obtain titania therefrom — MEHTA & PATEL
- 41457. Titanium pigments: Double sulphate of titanium and of a bivalent metal is treated with an alkali and thereafter calcined at 700°-800°C. — MEHTA & PATEL

#### **Inorganic Chemicals**

- 41005 & 41006. Sodium bicarbonate : Adding 0.1 to 2 per cent of very finely powdered calcium hydroxy phosphate or basic magnesium carbonate to sodium bicarbonate powder : Adding 0.1 to 2 per cent of potato starch powder to sodium carbonate powder — I.C.I. LTD.
- 41755. Method of extracting potassium from dilute solutions: Precipitating polassium aminate with a suspension of highly nitrated secondary aromatic free amine in presence of a base capable of forming soluble salt with the amine — NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB
- 42886. Manufacture of manganous sulphate: By heating pyrolusite with iron pyrites — MOORTHY, DATAR & ZAHEER

#### Metals & Metal Products

- 41821. Manufacture of cast irons: Cast iron with boron and aluminium as alloying elements — SINGH
- 42221. Treating ores bearing chromium or titanium: Establishing and maintaining a bed of material containing iron compound with titanium or chromium oxide in finely divided condition and passing gases containing chlorine and carbon monoxide — THE DORR Co.
- 43046. Manufacture of cast irons with improved nodular structure and malleable characteristics : Molten cast iron containing magnesium, nickel and/or copper — SINGH
- 41838. Distillation of aluminium from aluminiumbearing material: Including the circulation of

the halide vapour in the system by the use of an injector — INTERNATIONAL ALLOYS LTD.

43095. Desulphurization of metals and alloys by silicon: Desulphurizing by heating at reduced pressure with silicon — ELECTRIC FURNACE PRODUCTS CO. LTD.

#### Rubber & Rubber Products

42672. A process for spinning solutions of natural rubber: Solution of rubber plasticized to standard viscosity lower than 10 poises in toluene is used for obtaining threads in a bath saturated with  $SO_2$  — N. V. ONDERZOEKINGSINTITUUT RESEARCH

#### Stone, Clay & Glass Products

- 41342. Foam glass: Powdered glass consists of glass powder of comparatively large grain size to which has been added fine grain glass powder — RAM, VARSHNEY, RAO & SHARMA
- 42403. Grinding of granulated slag or similar materials: Feeding a grinding mill with variable specific gravity and hardness without previous homogenization — TRIEF & TRIEF

#### Miscellaneous

- 42441. Treatment of tars and pitches: Dissolving in volatile solvent separating solid from liquid, removing solvent from filtrate, again distilling the secondary tar and repeating the whole process with the residual secondary pitch — C. D. PA-TENTS LTD.
- 42450. Manufacture of moulded carbon articles from pitch: "Free carbon" obtained from pitch by treating with a solvent is moulded and fired to 550°C. in a non-oxidizing atmosphere — C. D. PATENTS LTD.
- 42760. Converting a petrol-operated internal combustion engine into a kerosine oil-operated engine : Combustion air and fuel independently preheated by exhaust of engine before entering carburettor, air heating chamber has pipes heated by the exhaust, fuel heated by contact with surfaces of fuel-heating chamber heated by the exhaust — BHARGAVA

#### **PATENTED INVENTIONS OF THE C.S.I.R.** — Continued from page 348

#### No. 39427

#### B. B. DEY, R. K. MALLER & B. R. PAI

2, 4-Diaminophenol is obtained by the electrolytic reduction of 2, 4-dinitrophenol in strong sulphuric acid at  $118^{\circ}$ C. in an yield of 94.4 per cent. No attempt appears to have been made to reduce 2, 4-dinitrophenol in dilute sulphuric acid at a lower temperature and obtain the compound in good yields.

According to this process, by electrolysing an emulsion of 2, 4-dinitrophenol in 30 per cent sulphuric acid at a copper cathode, in the absence of a catalyst at a current density of 1.99 amp./sq. dm. and  $90^{\circ}$ C., 2, 4-diaminophenol has been obtained in a pure condition (as the sulphate) in 81 per cent yield.

#### COMPOSITION FOR THE MANUFACTURE OF PATENT LEATHER

#### No. 38063

#### U.S.A. & British Patents pending S. SIDDIQUI & D. C. DHAR

A SUITABLE AND ECONOMICAL COMPOSITION FOR the manufacture of patent leather has been prepared from easily available and, as far as possible, indigenous ingredients. An oil resin is first prepared, as described in patent No. 38064, by copolymerizing vegetable oils with bhilawan shell liquid and turpentine or rosin ( or like oleoresinous materials ), by treatment with mitric acid ; the said oil resin is dissolved, along with film scraps, in butyl acetate and solvent naphtha. The ingredients are mixed by vigorous agitation in a shaking machine.

In respect of successive coatings, variations may be made in the ingredients as illustrated below: (a) The following further ingredients are added

(a) The following further ingredients are added to constitute the first coating composition : butanol or fusel oil; methanol; turpentine; and dibutyl phthalate;

(b) the following further ingredients are added to the first coating composition to constitute the second coating composition : bhilawan enamel; castor oil;

(c) the third coating composition is composed of butyl acetate; butanol or fusel oil; methanol; solvent naphtha; film scraps; dibutyl phthalate and acetone.

The proportions of the various ingredients are described in the patent. Bhilawan enamel is added to the composition for obtaining a fine glossy jet black film.

The coating compositions are applied to leather (preferably chrome-tanned) in the manner widely known in patent leather trades.

#### CONVERSION OF BHILAWAN SHELL LIQUID

#### No. 40752

#### S. SIDDIQUI, K. K. SARIN & A. SINGH

THE INVENTION RELATES TO THE CONVERSION OF bhilawan shell liquid to a non-vesicating drying product for the manufacture of lacquer varnishes, stoving enamels or water-resistant and flameresistant paints. It consists in treating bhilawan shell liquid with chlorine until the chlorination has proceeded at least up to 25 to 30 per cent. The chlorine treatment is carried out in a solvent

The chlorine treatment is carried out in a solvent medium such as kerosene or white spirit at temperatures ranging from  $30^{\circ}$  to  $70^{\circ}$ C. After chlorination, the solvent is partially driven off by heating the chlorinated product at temperatures between  $80^{\circ}$  and  $100^{\circ}$ C. The chlorine-treated product, which is a viscous mass at this stage, is converted to a non-vesicating, semi-solid to solid, drying resin on removal of the solvent, excess chlorine and hydrochloric acid formed during the reaction, at temperatures ranging up to 150°C. This resin which has chlorine content up to 55 per cent by weight is soluble in benzene, turpentine or the like hydrocarbon solvents. For the manufacture of paints, pigments such as zinc carbonate and magnesium carbonate are worked into the varnish. The paint is applied by brushing or spraying to fibres, fabric or like materials to render them water resistant and flame resistant.

#### **PRODUCTION OF 2, 4-DIAMINOPHENOL**

#### No. 35328

R. B. DEY, T. R. GOVINDACHARI & H. V. UDUPA

2, 4-DIAMINOPHENOL SALTS ARE USED EXTENSIVELY as photographic developers. The chemical is commonly prepared by reducing 2, 4-dinitrophenol with a metal and acid, or electrolytically. It has also been obtained by the direct reduction of *meta*dinitrobenzene or *meta*-nitraniline in concentrated sulphuric acid, electrolytically. In the present invention, the substance is produced by the electrolytic reduction of *meta*-dinitrobenzene in dilute acid emulsion.

Example — 40 gm. meta-dinitrobenzene, 400 c.c. of sulphuric acid of 40 per cent strength, 1 gm. mercuric sulphate at copper cathode, current density 7 amp./sq. dm., temperature 95°C. gave 24.4 gm. of 2, 4-diaminophenol oxalate (48 per cent yield).

#### No. 37566

#### B. B. DEY, H. V. UDUPA & B. R. PAI

In the present patent 2, 4-diaminophenol is produced by the electrolytic reduction of m-nitraniline in dilute sulphuric acid emulsion. By electrolysing a solution of m-nitraniline in 30 per cent sulphuric acid at a monel cathode, using 0.2 gm. of mercuric sulphate for every 400 c.c. of the sulphuric acid at a current density of 2.3 amp./sq. dm. and 80°C., 2, 4diaminophenol has been obtained in 50 per cent yield. The 2, 4-diaminophenol is first isolated as the sulphate and then in a pure condition as the oxalate.

#### No. 39426

B. B. DEY, R. K. MALLER, B. R. PAI & H. V. UDUPA

The yield of 2, 4-diaminophenol is improved by the reduction of m-nitraniline in dilute sulphuric acid solution at a copper cathode in the presence of copper sulphate.

Example — By electrolysing a solution of mnitraniline in 40 per cent sulphuric acid at a copper cathode, using 1.0 gm. of copper sulphate for every 400 c.c. of the sulphuric acid at a current density of 1.0 amp./sq. dm. and  $80^{\circ}$ C., 2, 4-diaminophenol has been obtained in 69 per cent yield.

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B

## Ultrasonic Studies in Electrolytes: Part I-Alkali Halides

### BH. KRISHNAMURTY Department of Physics, Andhra University, Waltair

Ultrasonic methods have been employed for studying the influence of concentration on the viscosity and surface tension of aqueous solutions of 4 alkali halides, KI, KBr, KF and NaF. The viscosity-concentration curves show a maximum at 0.3 mol. concn. in all the 4 cases. The surface tension increases with concn. in all the cases. In the range of concns. studied, the following empirical relationships hold good: (1)  $\eta = 2.0 \times 10^8 \beta$  and (2) T =  $1.2 \times 10^{-6} \rho_v^{\frac{3}{7}}$ , where  $\beta$  represents adiabatic compressibility;  $\rho$ , density; and v, ultrasonic velocity.

**THE** addition of an electrolyte to water is found to influence some of its molecular properties like the adiabatic and apparent molar compressibilities, viscosity, surface tension, etc. Investigations relating to the variation of these properties with different concentrations of the electrolyte, especially utilizing ultrasonic methods, have been few in number, and do not give any definite idea of the nature and extent of influence, especially in regard to the latter two. The variation of the adiabatic compressibility was, however, found to conform to a relation of the type  $\beta = \beta_{o} + Ac + Bc^{\frac{3}{2}}$ , deduced by Bachem<sup>1</sup>, where  $\beta$  is the adiabatic compressibility of the solution at the molar concentration c,  $\beta_{\circ}$  is that of water, A and B are constants, A being large and negative and B small and positive. According to this equation, therefore, the adiabatic compressibility decreases continuously with increasing concentration and remains always below that of water. An explanation for this lowering of the adiabatic compressibility can be sought in Debye-Huckel theory. The electrostatic field around each ion in solution exerts a large electrostatic pressure which, just like an enormous external pressure, lowers the compressibility.

Similarly, the apparent molar compressibility  $\varphi$  (which is a function of  $\beta$ ) of the electrolytes is found to follow the linear relation  $\varphi = a + b\sqrt{c}$ , obtained by Gucker<sup>2</sup>, where a and b are constants, and  $a = \varphi_{\alpha}$ , the apparent molar compressibility at infinite dilution (i.e. c = 0). Gucker calculated the values of  $\varphi$  for many electrolytes using the compressibility data obtained by Mair and Lanmann<sup>3</sup>, Perman and Urry<sup>4</sup> and others by piezometer measurements. He had shown that the apparent molar compressibility follows the linear relation set forth above. Also Falkenhagen and Bachem<sup>5</sup>, and Bachem<sup>6</sup> independently studying solutions of NaCl, KBr, BaCl<sub>2</sub>, Na<sub>2</sub>Co<sub>3</sub>, MgSO4, etc., by ultrasonic methods had shown that Gucker's relationship is followed by all the electrolytes. Ultrasonic velocity measurements in solutions of electrolytes were carried out by Hubbard and Loomis7, and Szalay<sup>8</sup>, and they obtained linear graphs between velocity and concentration. Some of the alkali halides like KI and NaI and KNO<sup>9</sup><sub>3</sub>, however, show a departure from such a linear relationship, especially at lower concentrations.

In case of viscosity and surface tension, no such simple relationships have been proposed.

In the present investigation, 4 alkali halides, KI, KBr, KF and NaF, have been chosen to test the linear relationships in these cases, especially at close concentrations in the low concentration range (up to 0.5 mol.) where departures have been reported for some electrolytes. Under the same conditions of temperature and atmospheric pressure, the viscosity and surface tension determinations have also been made. A correlative study of these different properties may give a better insight into the cause and extent of their variation.

Also for each electrolyte, at all concentrations, the values of

 $\frac{\alpha_r}{N^2}$ 

where  $\alpha_r$  is the absorption coefficient of sound at the frequency N, have been calculated using Stoke's<sup>10</sup> formula

$$\frac{\alpha_r}{N^2} = \frac{8}{3} \frac{\pi^2 \eta}{v^3 \rho}$$

where v,  $\rho$ ,  $\eta$  are the ultrasonic velocity, density and viscosity at the corresponding concentration.

#### Experimental

The ultrasonic velocities have been determined by the well-known Debye-Sears<sup>11</sup> arrangement for the diffraction of light by ultrasonics. The densities are determined by a sensitive Bunge balance and the adiabatic compressibility is calculated using the equation

$$\beta = \frac{1}{v^2 \rho}$$

The apparent molar compressibility is calculated using the relationship

$$\varphi = \frac{1000\beta}{c} - \frac{\beta_1}{d_1} \left\{ \frac{1000d}{c} - M_2 \right\}$$

where  $\beta$  is the adiabatic compressibility at the molar concentration c,  $\beta_1$  and  $d_1$  are adiabatic compressibility and density of water, and  $M_2$  is the molecular weight of the electrolyte.

The viscosity  $\eta$  is determined by the syphon method. The surface tension T is determined by observing the rise in a capillary tube. Curves representing the relation between (1)  $\beta$  and c, (2)  $\varphi$  and  $\sqrt{c}$ , (3)  $\eta$  and c, (4) T and c, and (5)  $\frac{\alpha r}{N^2}$  and c, have been drawn. Empirical relationships between (1)  $\eta$  and  $\beta$ , and (2) T, v and  $\rho$  have been proposed. The results are given in Table I

and also graphically represented in Figs. 1-5. Accuracy of Results — The density determinations are accurate up to one part in 1000. The accuracy of the fringe width measurements is estimated as 2 parts in 1000. The wavemeter (Philips heterodyne type) used to fix the frequency of the oscillator measures with an accuracy of 0-2 per cent. Hence the estimated accuracy of the velocity determinations is about 2

c, mol./lit.	ρ	v, meters	$\beta \times 10^{6}$	$\phi \times 10^{s}$	$\eta \div 10^{s}$	$\frac{\alpha r}{N^2} \times 10^{17}$	$\eta/eta \div 10^{\circ}$	т	$\frac{T}{\rho v^{\frac{3}{2}}} \times 10^6$
KI, Temp. 3	1°C.								
$ \begin{array}{c} 0 \cdot 1 \\ 0 \cdot 2 \\ 0 \cdot 3 \\ 0 \cdot 4 \\ 0 \cdot 5 \\ 1 \cdot 0 \end{array} $	$1 \cdot 012$ $1 \cdot 022$ $1 \cdot 036$ $1 \cdot 045$ $1 \cdot 059$ $1 \cdot 114$	1538 1528 1517 1526 1523 1541	$\begin{array}{r} 42 \cdot 29 \\ 42 \cdot 38 \\ 42 \cdot 45 \\ 41 \cdot 60 \\ 41 \cdot 23 \\ 38 \cdot 25 \end{array}$	$ \begin{array}{r} -17 \cdot 2 \\ -6 \cdot 7 \\ -3 \cdot 7 \\ -4 \cdot 1 \\ -3 \cdot 9 \\ -3 \cdot 6 \\ \end{array} $	$8 \cdot 230$ $8 \cdot 578$ $8 \cdot 326$ $8 \cdot 104$ $8 \cdot 021$ $7 \cdot 416$	$5 \cdot 896 \cdot 206 \cdot 065 \cdot 755 \cdot 654 \cdot 79$	$2 \cdot 0$ $2 \cdot 0$ $2 \cdot 0$ $2 \cdot 0$ $2 \cdot 0$ $1 \cdot 9$	71.6173.0274.5474.1674.8575.15	$1 \cdot 2 \\ 1 \cdot 1$
KBr, Temp.	29°C.								
$\begin{array}{c} 0.1 \\ 0.2 \\ 0.3 \\ 0.4 \\ 0.5 \\ 1.0 \\ KF, Temp. \\ 0.1 \\ 0.2 \\ 0.3 \\ 0.4 \\ 0.5 \\ \end{array}$	$\begin{array}{c} 1\cdot005\\ 1\cdot015\\ 1\cdot023\\ 1\cdot029\\ 1\cdot040\\ 1\cdot078\\ 28^{\circ}C.\\ 1\cdot002\\ 1\cdot005\\ 1\cdot009\\ 1\cdot014\\ 1\cdot018\\ \end{array}$	$1549 \\ 1534 \\ 1526 \\ 1532 \\ 1545 \\ 1547 \\ 1541 \\ 1523 \\ 1519 \\ 1549 \\ 1569 \\ $	$\begin{array}{c} 41 \cdot 96 \\ 42 \cdot 37 \\ 42 \cdot 47 \\ 41 \cdot 88 \\ 40 \cdot 71 \\ 39 \cdot 18 \\ \end{array}$ $\begin{array}{c} 42 \cdot 50 \\ 43 \cdot 40 \\ 43 \cdot 46 \\ 41 \cdot 57 \\ 40 \cdot 37 \end{array}$	$ \begin{array}{r} -21 \cdot 5 \\ -8 \cdot 3 \\ -4 \cdot 6 \\ -4 \cdot 3 \\ -5 \cdot 7 \\ -3 \cdot 4 \\ -18 \cdot 6 \\ -4 \cdot 2 \\ -2 \cdot 2 \\ -6 \cdot 4 \\ -7 \cdot 2 \\ \end{array} $	8.079 8.364 8.874 8.925 8.572 8.668 8.470 8.952 8.872 8.918 9.069	$5 \cdot 696$ $6 \cdot 013$ $6 \cdot 427$ $6 \cdot 348$ $6 \cdot 878$ $5 \cdot 711$ $6 \cdot 078$ $6 \cdot 640$ $6 \cdot 640$ $6 \cdot 6226$ $6 \cdot 074$	1 · 9 2 · 0 2 · 1 2 · 1 2 · 2 2 · 2 2 · 0 2 · 1 2 · 0 2 · 1 2 · 0 2 · 1 2 · 0 2 · 1 2 · 0	$\begin{array}{c} 73 \cdot 98 \\ 75 \cdot 77 \\ \\ 77 \cdot 59 \\ 77 \cdot 41 \\ 77 \cdot 23 \\ \hline \\ 75 \cdot 04 \\ 75 \cdot 18 \\ 76 \cdot 93 \\ 76 \cdot 60 \end{array}$	$1 \cdot 2 \\ 1 \cdot 3 \\ 1 \cdot 3 \\ 1 \cdot 3 \\ 1 \cdot 2 \\ 1 \cdot $
1.0	1.037	1572	39.50	- 4.1	9.625	6.292	2.4	75 95	$1 \cdot 2$
NaF, Temp	$28^{\circ}C.$								
$ \begin{array}{c} 0.1 \\ 0.2 \\ 0.3 \\ 0.4 \\ 0.5 \\ 1.0 \end{array} $	1.003 1.008 1.013 1.015 1.021 1.036	$\begin{array}{c} 1538 \\ 1526 \\ 1561 \\ 1531 \\ 1531 \\ 1531 \\ 1545 \end{array}$	$\begin{array}{r} 42 \cdot 64 \\ 43 \cdot 06 \\ 41 \cdot 03 \\ 42 \cdot 57 \\ 42 \cdot 34 \\ 40 \cdot 90 \end{array}$	$ \begin{array}{r} -18 \cdot 2 \\ -7 \cdot 2 \\ -11 \cdot 7 \\ -4 \cdot 7 \\ -4 \cdot 3 \\ -3 \cdot 4 \end{array} $	8.624 8.760 8.498 8.258 8.754	6 · 222 6 · 440 5 · 813 5 · 977 6 · 302	2.0 2.0 2.1 1.9 2.1 	75 · 48 75 · 51 75 · 56 76 · 58  77 · 02	1·3 1·3 1·2 1·3 

TABLE I



Fig. 1 — Adiabatic compressibility ( $\beta$ ) vs. molar concentration (c).



Fig. 2 — Apparent molar compressibility ( $\varphi$ ) vs. molar concentration ( $\sqrt{c}$ ).

parts in 1000, and this gives an equivalent accuracy to the factors calculated from the values of the velocity.

#### Discussion

Adiabatic Compressibility — The adiabatic compressibility for the 4 electrolytes chosen, of course, remains at all concentrations below the corresponding value of water at the temperature at which the experiments were carried out as required by Debye's theory, but departs from Bachem's linear relation; for the  $\beta$  instead of falling down gradually, slightly rises up to a concentration of very nearly 0.3 mol. where it shows a maximum, and then falls off linearly. It is interesting to note that the maxima correspond to a reduction of  $\beta$  of approximately 4 per cent in the case of KI and KBr, and 3 per cent in the case of KF and NaF. This considerably erratic variation of  $\beta$  at very low concentrations lying between 0 to 0.3 may, perhaps, also be due to the large osmotic activity evidenced at these very low concentrations, being superposed on the Debye effect. At comparatively large concentrations, say, above 0.3 mol. the osmotic activity may lessen, and hence the Debye effect is the only influencing factor, so that  $\beta$  goes on gradually falling.

Apparent Molar Compressibility — The apparent molar compressibility is negative, as is the case with electrolytes in general.



Fig. 3 — Viscosity ( $\eta$ ) vs. molar concentration (c).



Fig. 4 — Surface tension (T) vs. molar concentration (c).



FIG. 5 —  $\frac{\alpha_r}{N^4}$  vs. molar concentration (c);  $\alpha_r$  is the absorption coefficient of sound at the frequency N.

But the interest is that all the 4 halides show a departure from the linear relation of Gucker. Though no maxima and minima are shown,  $\varphi$  first rises with concentration steeply up to a value of 0.5 of  $\sqrt{c}$ , and then rather flatly. It is interesting to note that in the case of all the 4 electrolytes studied, the curves are almost parallel to each other, which shows that the nature and extent of variation of  $\varphi$  are the same.

Viscosity & Sound Absorption — The viscosities mostly remain above the value of water except in the case of KI where, after showing a maximum, it gradually decreases and goes below that of water. An interesting feature shown by the 4 halides is that the curves show a maximum at a concentration of very nearly 0.2 mol. The values of  $\eta$  at these points of maxima correspond roughly to 8 per cent rise in case of KI and KBr and 6.5 per cent rise in case of KF and NaF (analogous to the case of fall in  $\beta$ , where KI and KBr show the same percentage of fall and KF and NaF a different percentage, slightly lower). After the maximum, the curves for KI and KBr go on gradually falling off whereas the curves of KF and NaF show a minimum and begin to rise again. However, no simple relationship between  $\eta$  and c is possible.

It is interesting to note that the curves (Fig. 4) representing the relation between calculated values of  $\frac{\alpha_r}{N^2}$  (N = 2.5 megacycles per second in these measurements) and c are closely similar to the respective viscosity curves in all the 4 cases.

Surface Tension — The surface tension of the solutions of the 4 electrolytes goes on increasing with increasing concentration, though no simple relationship exists between the two. The rise is rather steep at first and flat afterwards (between 0.5 and 1 mol. concentrations) in case of KI, and reverse is the case of NaF. The KBr and KF curves (FIG. 5) first rise steeply up to a maximum at 0.4 mol. and then show a tendency to decrease slowly.

Empirical Relationships proposed in this Investigation — This correlative study of the adiabatic compressibility, ultrasonic velocity, viscosity and surface tension has afforded the following empirical relationships at the concentration range chosen.

(a) Between Viscosity & Adiabatic Compressibility ( $\eta & \beta$ ) — In all the 4 electrolytes, the ratio of  $\eta$  to  $\beta$  is a constant, quite independent of the concentration and temperature, as can be seen from the values in column 8, Table I. The constant is very nearly  $2 \cdot 0 \times 10^2$  in almost all the cases, so that the relationship  $\eta = 2 \cdot 0 \times 10^2 \beta$  exists. (b) Between Surface Tension, Ultrasonic Velocities & Density — The simple relationship  $T = 1 \cdot 2 \times 10^{-6} \rho v^{\frac{3}{2}}$  is found to exist between T, v and  $\rho$  at all concentrations in all the electrolytes, so that

 $\frac{T}{\rho v^{\frac{3}{2}}}$ 

is a constant,  $1.2 \times 10^{-6}$ , independent of concentration and temperature, as can be seen from column 10, Table I.

#### Summary

The adiabatic compressibility in the 4 halide solutions though remains always below the corresponding value of water, yet shows a departure from Bachem's linear type of relationship, in the sense that it shows maxima at a concentration of 0.3 mol. Above this concentration only it falls off gradually. The large osmotic activity evidenced at very low concentrations, it is suggested, may have something to do with the cause of the departure.

The linear relationship of Gucker,  $\varphi = a + b \sqrt{c}$ , between  $\varphi$ , the apparent molar compressibility, and  $\sqrt{c}$ , is not followed by these halides. Though no maxima and minima exist, the  $\varphi$  rises steeply first up to a value of 0.5 of  $\sqrt{c}$  and is then rather flat.

The curves are closely parallel to each other, showing that the nature and extent of variation of  $\varphi$  with  $\sqrt{c}$  is the same in all the halides.

The viscosity curves show a maximum at 0.2 mol. concentration nearly in all the 4 cases. The curves of KI and KBr run down after the maximum, whereas KF and NaF show a minimum and rise up again continuously.

The surface tension, in the 4 cases, gradually increases with increasing concentration.

The following empirical relationships have been proposed in the range of concentrations chosen :

(1)  $\eta = 2.0 \times 10^2 \beta$  and

(2)  $T = 1.2 \times 10^{-6} \rho v^{\frac{3}{2}}$ 

REFERENCES

- 1. BACHEM : Z. f. Physik., 1936, 101, 341.
- 2. GUCKER : Chem. Reviews, 1933, 13, 111.
- 3. MAIR & LANMANN: (Unpublished) Quoted by Gucker in Ref. 2.
- 4. PERMAN & URRY : Proc. Roy. Soc. London, 1929, A126, 44.
- 5. FALKENHAGEN, H. & BACHEM : Z. Electrochem., 1935, 41, 570.
- 6. BACHEM : Vide Ref. 1.
- 7. HUBBARD & LOOMIS: Phil. Mag. (VII), 1928, 5, 1177.
- 8. SZALAY : Phys. Z., 1934, 35, 639.
- 9. DUTT & GHOSH : Ind. J. Phys., 1942, 17(1), 19.
- STOKES, G. G. : Cam. Trans. Phil. Soc., 1845, 8, 287.
- 11. DEBYE & SEARS : Proc. Nat. Acad. Sci. Wash., 1932, 18, 410.

### The Elastic Properties of Single Jute Filaments: III — Nature of the Torsional Rigidity Modulus

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The nature of the torsional rigidity of single jute filaments, both in the raw and batched conditions, has been studied by using the method of the torsion pendulum. A procedure for comparing different jute varieties for twist resistance is indicated.

PPLICATION of twist is of prime importance in the formation of a spun textile yarn. It is immaterial whether the filaments are derived from plant or animal sources, or are regenerated from cellulosic or protein bases, or derived from man-made high-polymers. It also does not make any difference, except in respect of degree, as to whether the filaments are long and continuous, or cut to short lengths from such material. Some twist, either a few or even, as for cotton, more than a score over a couple and a half centimetres, needs must be applied in making a usable thread.

The purpose of applying twist may, of course, differ. In the case of the continuous filaments like rayon, nylon, etc., which may be made to have any desired length, a few twists only are imparted with a view to make the otherwise slippery materials easy to work with or handle. On the other hand, for the relatively much shorter natural fibres, twist happens to be an essential factor for building up of a compact and continuous yarn structure.

Twist always involves a shearing stress. So, whenever a twist is applied to a textile filament the rigidity of the filament introduces an opposing force of restitution to counteract the shear that follows the application of torsion. The magnitude of this force of restitution varies with the amount of shear imposed, i.e. the magnitude of the angle through which the material is deformed by the applied twist. If, therefore, a torsion pendulum is constructed using the textile fibre for suspension of a suitable "bob", then, between the force of the applied torsion and the proportionally generated one of restitution, a uni-planar periodic oscillation of the bob of the simple harmonic type can be set up. The period of oscillation of such a torsion pendulum will depend on the moment of inertia of the oscillating bob and the cross-section of the suspending fibre. A very simple relation exists between the modulus of rigidity of the suspending fibre and the period of oscillation of such a pendulum.

It is intended in the present article to study the nature of the torsional rigidity of single jute filaments both in the raw and the batched conditions (the filaments being derived separately from the middle and the base of the plant) by using the method of the torsion pendulum. The first attempt to measure the torsional quality of a textile filament by this method was by Peirce<sup>1</sup> for cotton. Recently Lochner<sup>2</sup> determined the rigidity modulus of wool and other fibres by a similar method which was also adapted to measure the damping capacity.

The rigidity of a textile fibre sets a limit to the amount of twist which can be tolerated in a unit length of the yarn of a particular diameter, without detracting from its quality. If jute filaments, for example, were less rigid, it would be possible to apply more twist to any particular yarn, within certain limits, without imparing its strength or increasing the stiffness. A larger number of twists in a unit length would, under the circumstances, result in greater binding effect and, probably, less hairiness<sup>3</sup> of the yarn. The importance of the study of the rigidity of the fibres, in view of its effect on both the utility and the appearance of the yarn, is, therefore, obvious. Since the rigidity of a textile fibre may even be varied by appropriate chemical treatment, the importance of the study of this property is further accentuated from the standpoint of reducibility by such treatment.

#### Experimental

Details of the Method — For the purpose of the experiments under consideration, a jute filament was regarded as possessing an *elliptical* cross-section. Such an assumption was necessary to make the incidental calculations easy to execute. The true shape of the cross-section of a jute filament is, however, irregularly polygonal. The approximation of the actual polygonal structure to an ellipse is not considered likely to have any significant effect on the precision of the measurement; for, as Smith<sup>4</sup> pointed out, the *shape* of the cross-section of a textile filament is much less important



FIG. 1 - TORSION PENDULUM.

AB, filament; l, length of the filament; D, brass rod; d, aluminium rod; L, length of the aluminium rod; 2R, the diameter of aluminium rod.

for the purpose of calculating the elastic modulus than its *size*.

The construction of the filament pendulum is shown in Fig. 1. Here AB represents a single filament, f, of a definite test length, One end, A, of the filament is fixed with l. shellac to a brass rod D. During an experiment, D is held by a clamp  $(a_1, a_2, \text{ etc.},$ in PQ, FIG. 2), such that the end holding the filament is sufficiently projected forward. The other end, B, of the filament is attached to the mid-point of an aluminium rod d, of known mass m, and length L and diameter 2R. The filament, AB, is required to be mounted accurately at right angles to the lengths of the rods D and d respec-When, therefore, D is clamped in tively. a horizontal position, d can oscillate freely in a horizontal plane.

The rod d is made to oscillate freely by means of a small, carefully executed push



FIG. 2 - PENDULUM HOUSE.

GK and RN, glass sides (RN, sliding door); PQ, horizontal rod provided with holes; a<sub>1</sub>, a<sub>2</sub>, etc., to insert D's; VX, sliding horizontal brass rod mounted with the vibration inducer Z. with the sliding vibration inducer Z (FIG. 2). Great care is necessary in executing the push so that the oscillation takes place in the horizontal plane and is of a purely simple harmonic type. Of course, the angular displacement is immaterial; for, even when this is large, the oscillation of a torsion pendulum is accurately simple harmonic<sup>5</sup>.

In order to determine with sufficient accuracy the period T of one complete oscillation, it is necessary to note the time taken by 2 to 5 successive oscillations, depending upon the rigidity of the filament. For exact measurement of T, the transit of an edge of the oscillating rod across the vertical cross-wire of a telescope was noted.

In one set of experiments  $\hat{m}$ , l, L and R are kept constant while the semi-axes of the elliptical cross-sections, a and b, and the period of oscillation, T, vary from filament to filament. The rigidity modulus, n, of a filament is derived from a consideration of the moment of inertia of the oscillating bob, the laws of simple harmonic motion, and the physical definition of the modulus of rigidity in relation to the applied torque. The formula employed is:

$$n = 4\pi \ ml \left[ \frac{L^2}{12} + \frac{R^2}{4} \right] \quad \left[ \frac{a^2 + b^2}{T^2 a^3 b^3} \right]$$

For a circular cross-section, a = b = r (radius), and so

$$n=\frac{8\pi \ I \ l}{T^2 r^4}$$

for, the moment of inertia of the rod d, or

$$I = m \left( \begin{array}{c} \frac{L^2}{12} + \frac{R^2}{4} \end{array} \right)$$

Peirce has defined this quantity

$$nr^4 \left( = \frac{8\pi^3 Il}{T^2} \right)$$

as the rigidity of the fibre.

For the present experiments, a batch of rods, d, was at first numbered serially and kept in separate, correspondingly numbered receptacles. The mass and the dimensional particulars, m, L and 2R for each rod was determined. The values were:  $m \leftarrow 0.398$  gm.;  $L \leftarrow 1.88$  cm.; and  $2R \leftarrow 0.313$  cm.

The length, l, of all the jute filaments examined was maintained at 5 cm.

The single filaments were obtained by the method described in an earlier paper<sup>6</sup>. The middle point of each rod, d, was marked beforehand by a sharp thin scratch for

correct mounting. The mounting was done by means of a suitable mixture of good quality red sealing wax and paraffin. The test filaments, after mounting, were conditioned overnight inside a desiccator in an atmosphere of about 75 per cent relative humidity (R.H.), maintained by using a saturated solution of ammonium chloride. Slight variations in R.H. occurred due to the fluctuation of the room temperature. Filaments to be tested were taken out of the conditioning desiccator and placed in position inside the test chamber (FIG. 2) in which also the same R.H. as in the desiccator was maintained, and the period of oscillation. T, determined. Later, all the tested filaments were measured for their width with the help of the apparatus described earlier<sup>7</sup>. Α similar procedure to the previous one reported, was, mutatis mutandis, adopted for mounting on the apparatus and also for measuring the width. The larger values of width  $(w_1)$  were pooled together, as also the smaller ones  $w_2$ .  $w_1$  and  $w_2$  corresponded to the axes 2a and 2b of the elliptical section mentioned above. One hundred filaments were examined for each mean value recorded here.

Material — The fibre from the crop, the middle and the base portions of the reeds of a single variety, in both the raw as well as the batched conditions, were tested separately. The batching and the binning were carried out as for the standard spinning procedure followed up in these laboratories<sup>8</sup>. The reference numbers of the tested varieties with the respective measures of yarn quality are given in Table I. In the present investigation 5 varieties of jute with widely divergent quality measure were tested.

#### Results

The test material was obtained by sampling as described in an earlier investigation<sup>2</sup>.

The arithmetic mean values of the rigidity moduli for the filaments are given in Table II.

TABLE I - THE V	ARIETIES	OF JUTE TESTED
REF. No.	VARIETY	QUALITY MEASURE*
J 1760 (W) J 1763A (W) J 1736 (T) J 1749 (T) J 1751 (T)	Capsularis Capsularis Olitorius Olitorius Olitorius	77 64 85 108 97
• The ratio, $\frac{bre}{gr}$	aking load in ist in lb./spin	$\frac{1 \text{ lb.}}{\text{ adle}} \times 100$

The arithmetic mean values of the greater  $(w_1)$  and the smaller  $(w_2)$  widths measured at right angles in a cross-section of the tested filaments are noted for the different test samples in Table III.

#### Discussion

Koshal and Turner<sup>9</sup> found from their studies on cotton that the distribution of fibre rigidity was "extremely asymmetrical". They found the coefficients of asymmetry to vary from +0.096 to +0.351. Individual values, which were sometimes as much as ten-fold the arithmetic mean, were observed. These two authors followed Peirce's technique in their measurements and did not take into account the actual dimensional measures of the cross-section of

the fibres. However, the general conclusions reached by Koshal and Turner seem to apply to jute filaments also.

It may be pointed out here that since the occurrence of extreme individual values affect the arithmetic mean, so far as the filament rigidity of a textile material is concerned, the arithmetic mean cannot be taken as an efficient criterion of the overall quality of the material. It is obvious. therefore, that the most important thing about the rigidity of jute filaments is the actual distribution of the various rigidity grades in a sample. It is possible that this feature characterizes particularly all fibres of natural origin.

The distributions obtained from the present experiments are shown graphically

JUTE REF. NO.	TABLE II Condition during test	- ARITHMETIC MEAN OF 7 Crop			THE RIGIDI	TY MOD	ULI OF	FILAMENTS * Base			
		RIGIDITY MODULUS		TEMP.	RIGIDITY MODULUS		TEMP.	RIGIDITY MODULUS		TEMP.	
		Mean (10 <sup>9</sup> dynes/ sq. cm.)	S.E.	( <sup>-</sup> F.)	Mean (10° dynes/ sq. cm.)	S.E.	(°F.)	Mean (10 <sup>o</sup> dynes/ sq. cm.)	S.E.	(°F.)	
J1760(W)	Raw	4.24	0.235	78	4.07	0.222	84	3.41	0.187	78	
	Batched	4.64	0.235	81	3.97	0.209	79	3.53 †	0.224	78	
J1763A(W)	Raw	3.11	0.284	74	3.20	0.243	83	5.05	0.267	87	
	Batched	2.80	0.289	86	2.81	0.172	82	3.03	0.253	75	
J1736(T)	Raw	5.42	0.332	87	4.34	0.245	82	4.92	0.293	81	
	Batched	5.21	0.308	86	5.95	0.359	85	6.32 +	0.350	86	
J1749(T)	Raw	2.28	0.163	86	2.32	0.152	89	3.21	0.205	87	
	Batched	2.22	0.179	87	2.38	0.185	86	2.91 +	0.194	91	
J1751(T)	Raw	3.37	0.248	77	2.92	0.212	84	3.01	0.195	87	
	Batched	<b>3</b> ∙00 †	0.209	72	3.57 †	0.244	79	2.70 †	0.121	86	

 R.H. varied between 77 and 79 per cent.
 † These were tested within 2 or 3 days after batching and binning. Others were tested after an interval of more than 10 days after batching.

TABLE III - MEAN WIDTHS AT RIGHT ANGLES OF THE CROSS-SECTION OF FILAMENTS

JUTE REF. NO.	Condition during test	Скор			MIDDLE			BASE		
		Width (10.3 cm.)		11'1 12's	Width ( 10 <sup>-3</sup> cm.)		<u><i>w</i></u> <sup>1</sup>	Width ( 10 <sup>-3</sup> cm.)		$\frac{w_1}{w_2}$
		w	w	(10-1)	$w_1$	ws	(10-1)	w	w	( 10-1)
J1760(W)	( Raw	7.218	4.513	6.25	7.287	4.832	6.63	7 . 950	$5 \cdot 191$	6.53
	Batched	7.191	4.475	6.22	7.300	4.674	6.40	8.001	4.996	6.24*
J1763A(W)	Raw	7.908	5.338	6.75	7.622	5.396	7.08	7.456	4.699	6.30
	Batched	7.769	5.712	7.35	7.298	5.273	7.23	7.828	5.824	7.44
J1736(T)	Raw	6.843	3.807	5.56	7.274	4.409	6.06	6.687	4.124	6.17
	Batched	7.076	3.878	5.48	6.545	3.798	5.80	6.634	4.028	6.07*
J1749(T)	Raw	6.925	4.754	6.86	6.559	4.808	7.33	6.234	4.909	7.87
	Batched	7.011	5.014	7.15	7.511	5.356	7.13	5.968	4.659	7.81*
J1751(T)	Raw	6.876	5.194	7.55	6.725	4.734	7.04	6.541	5.138	7.86
	Batched	7.402	5.698	7.70*	5.990	4.417	7.37*	6.992	5.142	7.35*
* These after batchin	were tested wit	hin 2 or 3 days	after batch	ning and bi	nning. Oth	ers were tes	sted after a	n interval of	more than	10 days

in Fig. 3. The extreme asymmetry of the distributions of the rigidity of jute filaments is striking. The statistical coefficient of asymmetry, measured by the method used by Koshal and Turner (loc. cit.), is found to range between +0.027 and +0.325. The range for jute is apparently a little wider than that of cotton. To illustrate the similarity of the nature of actual distributions of rigidity of the jute and the cotton fibres, typical cases of cotton can be seen from Koshal and Turner's report. The types for cotton compare favourably with those in Fig. 3.

Koshal and Turner (loc. cit.) have explained the observed asymmetry of distribution for cotton fibres in the following words: "...as the rigidity of the fibre depends upon the square of the area of the cross-section, the observed rigidity will be largely dependent upon the existence of thin places in the fibre; and the effect of such thin places will be exaggerated out of all proportion to their frequency and extent". This view is supported by the fact that jute, despite differences in structure and composition from cotton, exhibits the same kind of asymmetry.

It may be noted here that Koshal and Turner presumably assumed circular section for the cotton fibre and thus ignored the possibility of unequal inter-fibre or intervarietal differences in the dimensions of the cross-widths. If the cross-sections are circular, equal amount of variation, when any larger or smaller cross-section is encountered. must occur in the case of all the diameters within it. The possibility of variation of cross-sectional dimensions is thus very much limited. For an elliptical cross-section, on the other hand, the variation may occur in either or both the dimensions and the latter simultaneously, in the cross-sections measured for the same or different filaments ; and the extent of variation may be also the same or different. There is thus a very much wider possibility of variation in the cross-sectional areas of filaments of elliptical section than in those of the fibres of circular section. This greater possibility of variation in sectional area induces greater liability to produce extremely variable values of the rigidity modulus in the case of jute than in that of any fibre with circular section.

The graphs given in Fig. 3 show that the batching treatment often fails to effect any

significant change in the distribution of the rigidity modulus of jute. If reference is made to the mean moduli reported in Table II, it is observed here also that while, in some cases, the batched fibre records a lower value of the arithmetic mean, in others, similarly treated, an increase in value over that of the raw fibre occurs. Of course, as indicated before, the arithmetic mean provides no precise measure of the quality.

But referring to the ratio  $w_1/w_2$ , given in Table III, it is found that in most cases a lowering of the ratio to a notable extent results from the batching treatment. This indicates the probability of the widths along the two different axes of a cross-section changing differently under the treatment of an oilwater emulsion. This may then be regarded as providing more or less an evidence of the constitutional anisotropy of the jute filament.

Table IV provides the data for the coefficient of asymmetry calculated for the different test samples.

It is observed from this table that while in some cases batching induces greater asymmetry of distribution, in others the asymmetry in the raw state is higher. Further, one cannot also say that the degree of asymmetry is affected in a definite way by a particular part of the plant stem, viz. the crop, the middle or the base.

Now, in a yarn it is the integrated rigidity values of the component filaments present in a region which determine the stability of structure in that region. There the characters of all the relevant individuals exert *collectively* their full share of influence. vis a vis the distribution curve, in bringing about the twist-resisting effect observed in the yarn. The smoothed out average, such as one obtains by testing a bundle, even if it be of a known number of individuals, is hardly a suitable measure for comparing the structural qualities of yarns. It is to be noted also that in testing a bundle of separate entities, as a unit, what is measured is not the pure torsional rigidity, but merely a complex quantity, more or less of an uncertain nature, and determined by the torsional cum flexural rigidities of the constituent entities. Moreover, the dimensional specifications of such a bundle can only be rather uncertain.

From the above discussion, one may safely conclude that whether a jute will be, on the whole, more twist-worthy by batching, or by



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JUTE NO	Скор		MID		BASE		
REF. NO.	Raw	Batched	Raw	Batched	~	Raw	Batched
J1760(W) 11763A(W)	+0.104 + 0.182	+0.041 +0.413	+0.155 + 0.239	+0.115 + 0.226		+0.050 +0.107	+0.104 + 0.325
J1736(T) 11749(T)	+0.114 + 0.237	+0.027 +0.239	+0.131 + 0.149	+0.152 + 0.258	•	+0.165 + 0.168	+0.157 + 0.218
J1751(T)	+0.139	+0.548	+0.144	+0.091		+0.128	+0.175

TABLE V — APPROXIMATE PROPORTION (PER CENT) OF FILAMENTS HAVING MODULI OF RIGIDITY NOT EXCEEDING THE ARITHMETIC MEAN

JUTE	CROP		MI	DDLE	BASE		
REF. NO.	Raw	Batched	Raw	Batched	Raw	Batched	
J1760(W) J1763A(W) J1736(T) J1749(T) J1751(T)	72 67 62 81 67	64 74 56 81 71	75 60 72 83 70	75 73 71 80 57	58 59 67 63 67	58 71 62 71 72	

sorting out the material from a particular part of the plant, is uncertain.

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An approximate proportion of filaments whose moduli of rigidity do not exceed the mean value appreciably is given for each sample in Table V.

An important point to be noted here is that a majority, and very often a large majority, of filaments considering all the different cases examined, seems to possess a rigidity, the modulus of which does not much exceed the arithmetic mean. Therefore, there is a useful application of the mean rigidity modulus also, for example, in the matter of showing up the limit of rigidity within which most filaments may be found to occur. The arithmetic mean of the rigidity moduli may thus provide a rough basis of of comparing different jute varieties for twistresistance quality.

### Conclusions

1. The rigidity modulus of jute filaments is distributed with a distinct bias in favour of the small values. The distribution possesses features similar to those of cotton.

2. The procedure of batching or of sorting from a particular part of the stem does not seem to systematically affect either the mean rigidity modulus or even the distribution of the moduli in one particular direction. In other words, batching does not necessarily make a jute more twistworthy.

3. The extreme departure of the distribution of the rigidity moduli from normality precludes any effective study of the character by normal statistics. The different varieties may, however, be more or less roughly compared by the arithmetic mean value of rigidity, because, generally a large majority of the filaments possess a rigidity modulus not much exceeding this limit.

4. The variable effect of batching on the mutually perpendicular directions of a crosssection of a jute filament provides an evidence of its probable anisotropic nature.

The present work forms the third part of a series of studies undertaken in respect of the elastic properties of single jute filaments. The first two parts deal with the flexural rigidity<sup>2</sup> and the creep quality<sup>10</sup> of jute fibres.

### Acknowledgement

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### REFERENCES

- 1. PEIRCE, F. T.: J. Text. Inst., 1924, 14, 1. 2. LOCHNER, J. P. A.: ibid., 1949, 40, T220.
- 3. SEN, K. R. : J. Sci. Club, 1947, 1, 34.
- 4. SMITH DEWITT, H.: 19th Edgar Marburg Lecture, U.S.A., 1944.
- 5. ALLEN, H. S. & MOOR, H.: A Text Book of Practical Physics (Macmillan & Co.), 1934, p. 161
- 6. SEN, K. R. : Sci. & Cult., 1949, 15, 210.
- J. Hem.: J. Text. Inst., 1948, 39, 339.
   NODDER, C. R. & GILLIES, A. S.: Tech. Res. Mem., Indian Centr. Jute Comm., No. 1, 1940.
- 9. KOSHAL, R. S. & TURNER, A. J. : J. Text. Inst., 1930, 21, T325.
- 10. SEN, K. R. : Under publication.

### Studies in the Chemotherapeutic Derivatives of Acridine Series: Part II – Some Derivatives of 2-Iodo-7-ethoxy-9-substituted Aminoacridine

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2-Iodo-7-ethoxy-9-chloroacridine has been synthesized from two different routes, and condensed with o- and p-anisidines, toluidines, phenetidines, p-chloro-, bromo-, iodo-anilines, 2-aminopyridine and m-xylidine. 2-Iodo-7ethoxy-9-aminoacridine shows marked bacteriostatic action against Vibrio cholera, B. shigae, B. flexner, B. coli, B. paratyphosum B and Staph. aureus.

'N a previous communication<sup>1</sup> it was reported that some derivatives of 2iodo-7-methyl-9-aminoacridine showed distinct bacteriostatic action. The present communication deals with the synthesis of allied compounds in which the methyl group in position 7 has been replaced by ethoxy group, keeping the iodo group fixed in position 2. Ethoxy group is known to confer high antiseptic properties to this class of compounds and the findings of Morgenroth<sup>2</sup> that ethoxy group was the best substituent at position 2 or 7 (both being interchangeable) in the acridine nucleus for enhanced therapeutic properties supports this view. The well-known drug "Rivanol" also has an ethoxy group in this position.

Singh *et al.*<sup>3</sup> have reported that some N-substituted compounds having *p*-anisidine, *p*-phenetidine, *p*-toluidine and *p*chloroaniline substituents at position 9 in the acridine series possess considerable antiseptic activity and in some cases these compounds showed preferential action against gram-negative organisms.

In view of the above findings, it was of interest to study the influence of ortho substitution in some of the radicals which have been found to impart a fair degree of antiseptic activity to the parent compound. Ortho anisidine, phenetidine and toluidine have been condensed with 2-iodo-7-ethoxy-9chloroacridine with this idea in view. It may be mentioned that the solubilities of these compounds are considerably more than their p-isomers. Other substituents at position 9 include p-toluidine, p-anisidine, p-phenetidine, p-chloro-, bromo- and iodoanilines, 2-aminopyridine and m-xylidine.

2:5-Di-iodobenzoic acid was condensed with p-phenetidine in the usual way and the resulting 5-iodo-N-5'-ethoxy-N-phenylanthranilic acid on ring closure gave 2-iodo-7-ethoxy-9-chloroacridine. A confirmatory\* synthesis of the above product has also been achieved by the condensation of *m*-ethoxy-6-bromobenzoic acid with p-iodoaniline. The resulting 5-ethoxy-N-5'-iodophenylanthranilic acid, on ring closure, gave 2-ethoxy-7iodo-9-chloroacridine which was identical with the product already described. 2-Iodo-7ethoxy-9-aminoacridine was obtained from it by adding ammonium carbonate to the hot phenolic solution according to the procedure of Albert and Gledhill<sup>4</sup>.

2-Iodo-7-ethoxy-9-aminoacridine has been found to possess high inhibitory action against Vibrio cholera in a dilution of 1:3,20,000 and is active against Staph. aureus, B. coli, B. paratyphosum B, B. shigae and B. flexner in a dilution of 1:80,000. It shows moderate activity against B. typhosum A, and Proteus X-19. 2-Aminopyridine derivative has also shown moderate activity. Other compounds are inactive. The results of the assay have been communicated elsewhere<sup>5</sup>.

### Experimental

5-Iodo-N-5'-ethoxyphenylanthranilic Acid— This compound was obtained by the condensation of 2:5-di-iodobenzoic acid with p-phenetidine by the same procedure as reported earlier in the case of p-toluidine<sup>1</sup>. The reprecipitated acid, on crystallization from glacial acetic acid, came out in the form of faint-green rhombohydrons melting \*This part of the work was done in the National Chemical Laboratory of India, Poona 7. at 208°-9°C. (yield 80 per cent). Found, N, 3.65; M.W., 381;  $C_{15}H_{14}O_3NI$  requires N, 3.68 per cent; M.W., 383.

2-Iodo-7-ethoxy-9-chloracridine — 15 gm. of the above acid were refluxed with excess (50 c.c.) of phosphorus oxychloride for 4 hr. at 120°C. in an oil bath. Excess of phosphorus oxychloride was removed under reduced pressure and an ice-cold solution of dilute ammonia was added to the contents with constant stirring. The liberated base was filtered and washed with water and dried in vacuum. On crystallization from absolute benzene, it came out in fine silky yellow needles which melted at 170°-72°C. Found N, 3.72; C<sub>15</sub>H<sub>11</sub>ONCl requires N, 3.68 per cent.

3-Éthoxy-6-bromobenzoic Acid — 20 gm. (1 mol.) of *m*-hydroxy-6-bromobenzaldehyde were dissolved in 60 c.c. of 10 per cent sodium hydroxide (1.5 mol.) and heated gently under reflux. 20 gm. (1.4 mol.) of freshly distilled ethyl sulphate were slowly added to the above mixture and heating continued for 2 hr. The ethylated product separated out as an oil was not purified further but was used as such for oxidation.

17 gm. of potassium permanganate and 10 gm. of sodium carbonate dissolved in hot water were added in the course of 1 hr. to m-ethoxy-6-bromobenzaldehyde heated on a water bath. Heating of the reaction mixture was continued for 2 hr. more, after which the precipitated manganese dioxide was filtered off and excess of potassium permanganate removed by the addition of a small amount of sodium bisulphite. On acidification with dilute hydrochloric acid, 3-ethoxy-6-bromobenzoic acid came out as a fluffy precipitate which was filtered and washed with cold water. It crystallized from 60 per cent alcohol in colourless slender needles, m.p. 115°-16°C. Found, C, 48.5; H, 3.5;  $C_9H_9O_3Br$  requires C, 47.7; H, 3.67 per cent.

5-Ethoxy-N-5'-iodophenyl-anthranilic Acid-A mixture of 14.2 gm. (1 mol.) of potassium salt of m'-ethoxy-6-bromobenzoic acid, 16 gm. (1.5 mol.) of p-iodoaniline, 7 gm. (1 mol.) of anhydrous potassium carbonate and 20 c.c. of iso-amyl alcohol was refluxed in an oil bath at  $140^{\circ}$ -45°C. for 4 hr. A pinch of copper powder was added as a catalyst. At the end of this period, steam was passed through the reaction mixture to remove iso-amyl alcohol, filtered hot and acidified with dilute hydrochloric acid. The acid was purified through its ammonium salt which, on crystallization from absolute alcohol, yielded green needles, m.p. 208°C. Found N, 3.55; C<sub>15</sub>H<sub>14</sub>O<sub>3</sub>NI requires N, 3.68 per cent.

2-Ethoxy-7-iodo-9-chloroacridine was prepared by heating 5-ethoxy-5'-iodophenylanthranilic acid with phosphorus oxychloride at 120°C. for 4 hr., removing the excess of phosphorus oxychloride under reduced pressure and working out in the usual way. It crystallized from absolute benzene in the form of fluffy yellow needles, m.p.  $170^\circ$ -71°C. It showed no depression in its melting point when mixed with a sample of 2-iodo-7ethoxy-9-chloroacridine prepared earlier.

2-Iodo-7-ethoxy-9-aminoacridine was prepared in the usual way by adding ammonium carbonate to the hot phenolic solution of the corresponding 9-chloro derivative. The hydrochloride, on crystallization from a mixture of absolute alcohol and methanol, came out in the form of deep-yellow needles, m.p.  $302^{\circ}-4^{\circ}$ C. (decomp.). The base was liberated from it by warming with 2 per cent sodium hydroxide which, when crystallized from absolute alcohol, formed light-yellow needles, m.p.  $244^{\circ}-46^{\circ}$ C. Found N, 7.82; C<sub>15</sub>H<sub>13</sub>N<sub>2</sub>OI requires N, 7.69 per cent.

2-Iodo-7-ethoxy-9-chloroacridine was further condensed with *ortho* and *para* toluidines, anisidine and phenetidines, p-chloro-, bromoand iodo-anilines, *m*-xylidine and 2-aminopyridine as described in the general procedure below. Analytical data for the bases and their hydrochlorides are reported in Table I.

### **General Procedure**

The condensation was carried out according to the method of Gilman et al.6. 2-Iodo-7-ethoxy-9-chloroacridine (1 mol.) was dissolved in phenol at 80°C. and 1.1 mol. of the required amine was added to it and the contents heated for 3-4 hr. in a boiling water bath. When the reaction did not start at this temperature the mixture was heated in an oil bath at 130°-40°C. for 3 hr. A deep red to scarlet colour change indicates that the condensation has proceeded to completion. Ether was added after cooling the contents, when the base hydrochloride separated out. It was filtered and washed with ether and hydrochloride crystallized acetone. The from glacial acetic acid or absolute alcohol. The free base was obtained by treating the

SUBSTITUENT AT POSITION 9		BASE	М.Р., °С.	Base hydrochloride	М.Р., °С.	BASE FORMULA	ANALYS	IS OF BASE : ROGEN, %
							Found	Calculated
1.	<i>p</i> -Toluidine	Yellow prismatic plates	206-207	Golden-yellow slen- der needles	307-308	C22H10N2I	6.3	6.18
2.	<i>p</i> -Anisidine	Reddish-violet minute needles	234-235	Orange needles	296-297	C22H19N2O2I	6.1	5.96
3.	<i>p</i> -Phenetidine	Bright-scarlet stout needles	183-184	Yellow fluffy silky needles	302-303	C22H21O2N2I	5.7	5.77
4.	p-Chloroaniline	Chrome-yellow prismatic rods	191-192	Slender yellow needles	300	Ca1H18ON2CII	5.9	6.09
5.	p-Bromoaniline	Greenish-yellow plates	172-175	Silky yellow needles	298-299	C <sub>21</sub> H <sub>10</sub> ON <sub>2</sub> Br. I	5.35	5.50
6.	p-Iodoaniline	Yellow needles	186-187			C <sub>11</sub> H <sub>10</sub> ON <sub>1</sub> I <sub>2</sub>	4.9	5.09
7.	o-Toluidine	Yellow needles	171-172			CHON.I	6.25	6.18
8.	o-Anisidine	Yellow slender silky needles	164-165	Ruby-red prisms	278	C <sub>22</sub> H <sub>10</sub> O <sub>2</sub> N <sub>2</sub> I	5.8	5.96
9.	o-Phenetidine	Yellow slender silky needles	168-169	Ruby-red prisms	268-269	C <sub>23</sub> H <sub>21</sub> O <sub>3</sub> N <sub>2</sub> I	5.65	5.77
10.	m-X vlidine	Greenish-vellow plates	176-178			CH., N.I	6.3	6.20
11.	2-Aminopyridine	Yellow needles	176			CroH10ON,I	9.65	9.52
						21		

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TABLE I - 2-IODO-7-ETHOXY-9N-SUBSTITUTED AMINOACRIDINES

hydrochloride with 5 per cent sodium hydroxide in the hot. After drying it could be crystallized from absolute alcohol or suitable mixed solvents. Both the base and the hydrochlorides are not very soluble in organic solvents and solubility in water is poor.

### Acknowledgement

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#### REFERENCES

- 1. SURJIT SINGH & MAHAN SINGH : J. sci. industr. Res., 1950, 9B, 27.
- MORGENROTH : Chemistry of Chemotherapy (Ernst Benn Ltd., London), p. 169.
   SINGH et al.: Ind. J. med. Res., 1948, 36,
- 3. SINGH et al.: Ind. J. med. Res., 1948, 36, 397.
- 4. Albert & Gledhill : J. Soc. Chem. Ind., 1945, 69, 166.
- 5. SURJIT SINGH : Ind. J. med. Res., under publication.
- 6. GILMAN et al.: J. Amer. Chem. Soc., 1946, 68, 326.

### Nitration of 4-Chloro-6-methoxy-quinaldine

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The preparation of 4-hydroxy-6-methoxyquinaldine and its nitro derivative is described. Starting with the nitro derivative, a number of amino and chloro derivatives have been prepared.

**T**N our attempts to prepare 8-aminoquinoline derivatives (which might find use as possible anti-malarials), it was considered desirable to introduce certain substituents in the pyridine nucleus as it is found that the quinine molecule undergoes biological oxidation at 2-position, and that some of the 4-substituted quinoline derivatives have been found to exhibit pronounced anti-malarial activity. Attempts to prepare 8-nitro-4-hydroxy-quinaldine from o-nitroaniline and ethylacetoacetate by the method of Conrad and Limpach<sup>1</sup> were unsuccessful, probably due to the presence of o-nitro group in the aromatic amine. We have also experienced the same difficulty with regard to 4-methoxy-o-nitroaniline.

Halcrow and Kermack<sup>2</sup> have shown that when 4-chloro-quinaldine is nitrated with potassium nitrate and sulphuric acid (100 per cent) the product obtained contains mainly 4-chloro-8-nitro derivative along with some of the 5-nitro and 6-nitro isomers. Hence it would be of interest to study the nitration of 6-methoxy-4-chloro-quinaldine which can be prepared by the condensation of p-anisidine and ethylacetoacetate.

4-Hydroxy-6-methoxy-quinaldine was prepared by condensing p-anisidine with acetoacetic ester according to the method of Conrad and Limpach. This was converted to the 4-chloro derivative and nitrated when the 8-nitro derivative was obtained as the sole product. The orientation of the nitro group has been established by reducing it to 8-amino derivative and then to 4:6dimethoxy-8-amino-quinaldine. Employing Gattermann reaction it was then converted to 4:6-dimethoxy-8-chloro derivative. Its identity was established by comparing it with an authentic sample of the same compound prepared by an alternative route, i.e. by subjecting 2-chloro-4-methoxy-aniline (obtained by the simultaneous reduction and chlorination of p-nitro anisole<sup>3</sup>) to Conrad and Limpach reaction. The 4-hydroxy-6methoxy-8-chloro-quinaldine so obtained was converted to 4: 6-dimethoxy-8-chloro-quinaldine.

### Experimental

4-Hydroxy-6-methoxy-quinaldine was prepared by condensing p-anisidine and ethylacetoacetate in the presence of an acid catalyst. The resulting ester was cyclized in diphenyl ether to get 4-hydroxy-6methoxy-quinaldine which crystallized from ethanol as shining plates; m.p. 317°-18°C. (decomp.).

4-Chloro-6-methoxy-guinaldine-Tohydroxy quinaldine (46 gm.) was added phosphorus oxychloride (75 c.c.) and refluxed at 120°-25°C. After one hour another lot of phosphorus oxychloride (75 c.c.) was added and refluxing continued for another two hours. Excess of phosphorus oxychloride was removed by distillation under reduced pressure. The white residue was then mixed with 700 c.c. of iced water and well stirred. The clear solution obtained was treated with charcoal twice and filtered and the filtrate made alkaline with 20 per cent sodium hydroxide. The precipitate was filtered, washed free of alkali and dried. It crystallized from ethanol as shining crystals, m.p. 92°-93°C. Found N, 6.69; C<sub>11</sub>H<sub>10</sub>ONCl requires N, 6.75 per cent.

Nitration of 4-chloro-6-methoxy-quinaldine — The base (45 gm.) was dissolved in sulphuric acid (100 per cent, 300 gm.) and the

solution cooled in a freezing mixture and stirred with a mechanical stirrer. Finely powdered potassium nitrate (35 gm.) was added gradually over a period of one hour. Stirring was continued for two hours and the mixture kept overnight at 15°C. The mixture was poured into 600 c.c. of iced water and made alkaline with 20 per cent sodium hydroxide. The precipitate was filtered, washed and dried (58 gm.). It crystallized from ethanol as shining prisms, m.p. 119°-20°C. Found N, 10·98;  $C_{11}H_9O_3N_2Cl$  requires N, 11·09 per cent.

4-Chloro-6-methoxy-8-aminoquinaldine—The nitro compound (50 gm.) was dissolved in concentrated hydrochloric acid (330 c.c.) and to the solution was added a solution of stannous chloride (160 gm.) in concentrated hydrochloric acid (250 c.c.) and water (250 c.c.). The mixture was refluxed for two hours, charcoaled and the solution filtered hot. The filtrate was made alkaline with 20 per cent sodium hydroxide when shining yellow crystalls separated out; yield 25 gm. A portion crystallized from ethanol melted at 100°-101°C. Found N, 12·48; C<sub>11</sub>H<sub>11</sub>ON<sub>2</sub>Cl requires N, 12·58 per cent.

4:6-Dimethoxy-8-aminoquinaldine — 4-Chloro-6-methoxy-8-aminoquinaldine (2 gm.) and sodium pellets (0.4 gm.) in absolute methanol (20 c.c.) was refluxed on a steam bath for two hours. Most of the methanol was then removed and the residue was diluted with water and the precipitate filtered, washed and dried. The residue (2 gm.) crystallized from ethanol as shining plates, m.p. 227°-28°C. (decomp.). Found N, 12.65;  $C_{12}H_{14}O_{2}N_{2}$  requires N, 12:84 per cent.

4: 6-Dimethoxy-8-chloro-quinaldine — The aminoquinaldine (1.5 gm.), water (5 c.c.) and conc. hydrochloric acid (5 c.c.) were cooled in a freezing mixture and diazotized with a solution of sodium nitrite (0.75 gm. in 4 c.c. of water). To the diazotized solution was added copper bronze powder (1 gm.) gradually with stirring when a light-yellow precipitate separated out. After the evolution of nitrogen had stopped, 20 c.c. of water were added and the mixture heated on a water bath when the yellow precipitate went into solution. It was filtered and the filtrate made alkaline with ammonia. The precipitate formed was filtered, dried (yield, 1.1 gm.) and crystallized from water (charcoaled) when white shining plates, m.p.

95°-98°C., were obtained. Found N, 6.08; C12H12O2NCl requires N, 6.29 per cent.

2-Chloro-4-methoxy aniline was prepared according to the method of Hurst and Thorpe<sup>3</sup> by the simultaneous reduction and chlorination of p-nitroanisole with concentrated hydrochloric acid and tin.

4-Hvdroxv-6-methoxv-8-chloro-guinaldine — 2-Chloro-4-methoxy aniline (4.3 gm.) was mixed with ethyl acetoacetate (3.6 gm.), dilute hydrochloric acid (3 drops) added and the mixture left overnight. The resulting ester was separated and dried over anhydrous magnesium sulphate. The dried ester (6.5 gm.) was added to boiling diphenyl ether (30 c.c.) and refluxed for 15 min. The crystals which separated out on cooling were washed with acetone, m.p. 300°-301°C.; yield 1.1 gm. Found N, 6.1; C11H10O2NCI requires N, 6.26 per cent. The compound was found to be insoluble in common organic solvents.

4:6-Dimethoxy-8-chloro-guinaldine - To 4-hydroxy-6-methoxy-8-chloro-quinalding (0.5 gm.) dissolved in 10 per cent sodium hydroxide (10 c.c.) was added distilled methyl sulphate (2 c.c.) and the mixture well shaken for half an hour, after which it was heated on a water bath for another half an hour. Finally it was cooled, filtered and the residue washed and dried (vield, 0.45 gm.). After crystallization from water it was obtained as shining plates, m.p. 95°-96°C. This sample, when mixed with that prepared by the previous procedure, melted without any depression in the melting point.

### REFERENCES

- 1. CONRAD & LIMPACH : Ber., 1884, 21, 1981.
- 2. HALCROW, B. E. & KERMACK, W. O. : J. Chem. Soc., 1945, 415. 3. HURST & THORPE : ibid., 1915, 934.

### Chemical Examination of the Seeds Cucumis utilissimus of Roxb.

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A detailed chemical examination of oils from seed kernels of two varieties (Lucknow and Saharanpur) of Cucumis utilissimus Roxb. has been carried out. The oils from both varieties are of the semi-drying class and are almost identical in composition.

UCUMIS utilissimus Roxb. (Hindi: kakri) belongs to the natural order Cucurbitaceae. Its seed kernels are edible and are used in confectionery in India; a drink made from the ground kernels has a refreshing and cooling effect. A detailed chemical examination of the seed kernels of two varieties (Saharanpur and Lucknow) has been undertaken and the results obtained are recorded in this paper. The Saharanpur variety is about 1' to 1.5' long and is of green colour, while the Lucknow variety is only 9" to 12" in length and is of pale-green colour.

The seed kernels of both the varieties contain about 43.75 per cent oil, having the following fatty acid composition :

Acids	Saharanpur variety %	Lucknow variety %
Lauric	0.12	
Myristic	0.20	2.05
Palmitic	12.8	11.14
Stearic	7.88	3.03
Oleic	27.45	31.18
Linoleic	50.40	52.60

The kernels contain about 37.1 and 35.6 per cent proteins respectively. The kernels of both varieties are rich in phosphate  $(1.02 \text{ and } 1.13 \text{ per cent } P_2O_5 \text{ respectively}).$ 

The unsaponifiable fractions of the oils of both the varieties are partly soluble in hot alcohol; this portion proved to be sitosterol (m.p. 140°C.; acetate, m.p. 130°-31°C.). The major portions of the fraction, however, crystallized from glacial acetic acid in lamina-like plates melting at 51°C. and proved to be cetyl alcohol (mixed m.p. with the authentic specimen,  $50^{\circ}-51^{\circ}C.$ ). On oxidation with chromic acid in glacial acetic acid solution, palmitic acid (m.p. 62°C.) was obtained.

The oils from both varieties belong to the semi-drying class and are of almost identical composition.

### Experimental

The seeds of Saharanpur and Lucknow varieties contain about 70 and 67 per cent kernels respectively. The powdered kernels were extracted 4 times with petroleum ether (b.p.  $40^{\circ}-60^{\circ}$ C.). After removing the solvent,  $43\cdot75$  per cent of clear, light-yellow bland oils were obtained. They had the following characteristics:

	Saharanpur variety	Lucknow variety
Sp. gr. at 40°C.	0.9145	0.9135
Viscosity at 40°C.	39.32 c.s.	37.15 c.s.
Ref. index at 25°C.	1.4730	1.4738
Acid value	5.58	5.54
Sap. value	193.2	189.5
Iodine value (Wijs)	118.5	123.3
Thiocyanogen value	74.95	76.55
Acetyl value	14.9	18.79
Hexabromide value	nil	nil
Unsaponifiable matter, %	0.85	1.83
Hehner value	94.7	93.97
Soluble fatty acids cal- culated as butyric acid	nil	0.3
Saturated fatty acid, % (modified Bertram method <sup>3</sup> )	20.4	16.4

250 gm. of the oil were saponified with alcoholic potash, the alcohol was distilled off and the residual soap dissolved in water. The unsaponifiable matter was removed with ether and the soap solution was decomposed with dilute sulphuric acid. When heated on the water bath, the fatty acids formed a clear oily layer at the top. They were removed with ether and dried in vacuum at 100°C. The acids (200 gm.) were dissolved in 95 per cent alcohol (1,000 c.c.), the solution boiled and mixed with a boiling solution of lead acetate (150 gm.) in 95 per cent alcohol (1,000 c.c.), the alcohol

in both cases containing 1.5 per cent glacial acetic acid. The insoluble lead salts obtained on cooling overnight at  $15^{\circ}$ C. were crystallized from alcohol containing 1.5 per cent glacial acetic acid when a pale-yellow crystalline material was obtained. The solid acids were regenerated from the lead salts. The liquid acids were recovered from the lead salts left over on evaporation of the mixed alcohol mother liquors. The quantities of soluble and insoluble lead salts of fatty acids from both the oils and some of their characteristics are given in Table I.

### Soluble Lead-salt Fatty Acids

The soluble lead-salt fatty acids, on oxidations with potassium permanganate (modified Bertram method<sup> $\hat{s}$ </sup>), gave 1.0 and 2.5 per cent saturated acids respectively.

The liquid acids were converted into methyl esters and a known weight was fractionally distilled under reduced pressure<sup>4</sup>. The results obtained are given in Table II.

The losses in distillation (1.26 and 1.92 respectively) were proportionately divided and added to each fraction; the saponification and iodine values were determined and the amounts of individual esters in each fraction were calculated. These results are given in Tables III and IV respectively.

The acids from L-3 and L-6 from both the varieties were oxidized with dilute alkaline potassium permanganate<sup>5</sup> when tetrahydroxy stearic acid (m.p. 173°C.) and dihydroxy stearic acid (m.p. 130°C.) were isolated from both the fractions. A product melting at 155°-66°C. was also obtained which may be an eutectic mixture of  $\alpha$ and  $\beta$  sativic acids<sup>6</sup>. The compositions of the soluble lead-salt fatty acids are as follows :

	Saharanpur variety %	Lucknow variety %
Lauric acid	0.16	J
Myristic acid	0.64 > 0.95	2.04 > 2.44
Palmitic acid	0.15	0.40
Linoleic acid	64.43	61.69
Oleic acid	34.62	35.87

The bromine addition derivatives of the soluble lead-salt fatty acids from both the varieties were prepared according to the method of Eibner and Muggenthalor<sup>7</sup> as

				TA	BLE						
FATTY ACID SAHAR		SAHARANPUR	VARIETY				L	UCKNOW	VARIETY		
		Quantity, %	Neutraliza- tion value	Iodine value	Thiocyar valu	ogen e	Quantity, %	Neut tion	raliza- value	Iodine value	Thiocyanogen value
Total Soluble lea Insoluble l	ad salt lead salt	79·35 20·65	$202 \cdot 8$ 201 \cdot 5 208 \cdot 4	$122 \cdot 0$ $148 \cdot 0$ $4 \cdot 9$	77 · 1 91 · 9	3 96	85·9 14·10	20 20 20	04 · 1 02 · 8 08 · 3	128·2 145·0 6·73	82·42 90·04
A				TA	BLE I	r					
		WEIGHT OF	SAHARANPUR THE ESTERS DIS	VARIETY	05·83 см.		WEIGHT	L OF THE	UCKNOW ESTERS	VARIETY : DISTILLED	, 96·75 см.
		Temperatu range, °C.	re Pressu mm.	re,	Wt. of fraction, gm		Tempera range, °C.	ture	Press	ure, 1.	Wt. of fraction, gm.
L1 L3 L4 L4 L4 L4 L4 L4 L4 L4 L4 L4 L4 L4 L4		180-195 205-207 208 209-210 213-214 215 217-218 220 Residue	$1 \cdot 0$ $1 \cdot 5$ $1 \cdot 5$ 1		$7 \cdot 69 \\ 11 \cdot 50 \\ 10 \cdot 17 \\ 12 \cdot 11 \\ 12 \cdot 04 \\ 14 \cdot 49 \\ 15 \cdot 34 \\ 10 \cdot 15 \\ 11 \cdot 08 \\ 104 \cdot 57 \\ 104 \cdot 57$		190-19 195-19 208-21 208 205 215-21 210 Resid	5 6 0 6 ue	1. 1. 2. 2. 1. 2. 1.	5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	$ \begin{array}{c} 11 \cdot 37 \\ 13 \cdot 50 \\ 7 \cdot 36 \\ 10 \cdot 05 \\ 11 \cdot 62 \\ 14 \cdot 13 \\ 14 \cdot 13 \\ 12 \cdot 67 \\ \dots \\ 04 \cdot 82 \\ \end{array} $
					104.57						94.83
		-	TABLE	III — SA	HARANP	UR V.	ARIETY				
C	ORRECTED WT.,	IODINE VALUE	SAPONIFICA- TION VALUE	MEAN 1 W1	MOL.	Mathul	Mot	WEIGH	IT OF ES	TERS	1 Methyl
-					i	aurate	myris	tate p	almitate	linolea	te oleate
L <sub>1</sub> L <sub>2</sub> L <sub>4</sub> L <sub>6</sub> L <sub>6</sub> L <sub>7</sub> L <sub>8</sub> L <sub>9</sub>	7.78 11.64 10.29 12.26 12.18 14.67 15.53 10.27 11.21	$132 \\ 137 \cdot 1 \\ 140 \cdot 4 \\ 142 \cdot 2 \\ 144 \cdot 3 \\ 145 \cdot 4 \\ 147 \cdot 7 \\ 148 \cdot 5 \\ 125 \cdot 4$	19593.0192.1190.3190.1190.0190.2190.4189.5	287 290 292 294 295 295 295 294 294 294	·7 ·7 ·0 ·7 ·1 ·2 ·9 ·6 ·1	0·18     	0. 0. 0.	82 82 05  	0 · 10 0 · 07   	$\begin{array}{r} 4.95\\ 6.97\\ 6.61\\ 7.99\\ 8.23\\ 10.09\\ 11.10\\ 7.43\\ 5.16\end{array}$	$2 \cdot 69$ $4 \cdot 25$ $3 \cdot 56$ $4 \cdot 27$ $3 \cdot 95$ $4 \cdot 53$ $4 \cdot 43$ $2 \cdot 84$ $6 \cdot 05$
TOTAL	105.83					0.18	0.	69	0.17	68·17	36.62
			TABI	LE IV-	LUCKNO	W VA	RIETY				
ESTER FRACTION	CORRECTED WT.,	IODINE VALUE	SAPONIFICA- TION VALUE	Mean W	MOL.			WEIGHT	OF ESTI	ERS	3
	gm.					Methy myrist	/l ate j	Methyl palmitat	e li	Methyl noleate	Methyl oleate
L1 L2 L3 L4 L5 L6 L7 L8 Total	$     \begin{array}{r}       11 \cdot 6 \\       13 \cdot 77 \\       7 \cdot 51 \\       10 \cdot 25 \\       11 \cdot 85 \\       14 \cdot 42 \\       12 \cdot 93 \\       96 \cdot 75 \\     \end{array} $	128.9135.1137.5140.6143.3144.8145.7119.0	196 · 8 195 · 0 194 · 3 194 · 5 190 · 4 190 · 2 190 · 0 189 · 8	285 287 288 288 294 294 295 295	9-1 9-7 9-7 9-5 1-6 1-9 9-2 9-6	1.1 0.6 0.2    2.0	5 7 2 2	0.01 0.09 0.08 0.20    0.38		6 · 94 8 · 60 4 · 79 6 · 69 7 · 88 9 · 81 9 · 98 4 · 97 59 · 66	$     \begin{array}{r}       3 \cdot 50 \\       4 \cdot 41 \\       2 \cdot 42 \\       3 \cdot 36 \\       3 \cdot 97 \\       4 \cdot 61 \\       4 \cdot 44 \\       7 \cdot 96 \\       \overline{34 \cdot 67}     \end{array} $
-				ТА	BLE	v					
Ester Fraction	v	Sahar. vt. of ester	ANPUR VARIETY DISTILLED, 35	: 35 см.		-	WT. 0	LUCE f ESTER	NOW VA	RIETY: 2D, 27.76	GM.
	Tempera	ture ,	Pressure, mm.	Wt. of fr	action,	Ten	nperature range, °C		Pressure mm.	e, V	Wt. of fraction, gm.
51 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	170-18 185-18 187-18 195-20 Residu	81 37 90 95 96	$1 \cdot 5$ $1 \cdot 5$ $1 \cdot 5$ $2 \cdot 0$ 	4 · 5 7 · 8 5 · 7 9 · 4 7 · 0 <b>34</b> · 8	26 35 70 44 82 87	1 1 1	80-182 84 85-186 Residue		1.0 1.5 1.5 		4 · 47 5 · 31 6 · 27 5 · 26  21 · 31
	1971 - 1972 - 19	0									

described by Jamieson and Boughmann<sup>8</sup>. The results are as follows :

			Saharanpur variety	Lucknow variety
Soluble acids ta	lead-salt aken for ar	fatty	3·2860 gm.	4•2334 gm.
Saturated tram's	l acids from method	n Ber-	0.0328 gm.	0·1058 gm.
Linoleic soluble (m.p.	tetrabromi in petrol 113°-14°C.	de in- ether	2·1609 gm.	2·9340 gm.
Residue dibrom acids)	( tetrabro ide and sat	omide, urated	4·1802 gm.	5·2050 gm.
Bromine residue	contents o	f the	45·35 %	43.44 %
Linoleic the res	tetrabromi sidue	de in	2·3067 gm.	2·428 gm.
Total tet	rabromide	found	4.4674 gm.	5.362 gm.
Linoleic to tetr	acid equi	valent	2.0850 gm.	2.5015 gm.
Saturated	1 acid		1.0 %	2.5 %
Oleic aci	d		35.55 %	38.4 %

The mean values obtained for these acids from iodine value, distillation and bromination results are :

	Saharanpur variety	Lucknow variety
Saturated acids	0.99	2.48
Linoleic acids	64-21	61.23
Oleic acid	34.80	36.29

### **Insoluble Lead-salt Fatty Acids**

The insoluble lead-salt acids, after conversion into methyl esters, were distilled under reduced pressure. The fractions obtained are given in Table V.

The losses in distillation were added proportionately to each fraction. The iodine and saponification values and mean molecular weights of all the fractions were calculated according to the method of Jamieson and Boughmann<sup>8</sup> (TABLES VI and VII).

The acids from fraction  $S_2$  from both the varieties, on repeated crystallization from

alcohol, gave palmitic acid, while those from  $S_4$ , on repeated crystallization from alcohol and acetone, gave a product melting at 68°-70°C. showing it to be principally stearic acid.

The amounts of individual saturated components in the insoluble lead-salt fatty acids are :

	Saharanpur variety	Lucknow varietv
	%	%
Myristic acid		1.95
Palmitic acid	61.52	76.51
Stearic acid	38-48	21.54

From the above analysis, the total fatty acid constituents of the oils from the seed kernels of two varieties of *Cucumis utilissimus* Roxb. are :

	Saharanpur variety	Lucknow variety
	%	%
Lauric acid	0.12	
Myristic acid	0.20	2.05
Palmitic acid	12.83	11.14
Stearic acid	7.94	3.03
Linoleic acid	50.98	52.6
Oleic acid /	27.63	31.18

The composition of the two oils, therefore, are :

	Oil from Saharanpur varietv	Oil from Lucknow variety
Glycerides		
Lauric acid	0.12	
Myristic acid	0.50	2.04
Palmitic acid	12.80	11.00
Stearic acid	7.88	2.98
Linoleic acid	50.40	51.48
Oleic acid	27.45	30.67
Unsaponifiable matter consisting of sitosterol and cetyl alcohol	0.82	1.83

### Seed Kernel Cake

The cakes obtained from the seed kernels, after complete removal of the petrol ether

		TA	BLE VI — SAHARA	NPUR VARI	ETY		
	CORRECTED WT.,	TERS,					
	gin.			w1.	Methyl palmitate	Methyl stearate	Unsaturated esters
S1 S3 S3 S5 S5	4 · 32 7 · 96 5 · 78 9 · 57 7 · 72	2·7 3·15 3·27 4·09 6·8	$204 \cdot 6$ $203 \cdot 7$ $203 \cdot 5$ $199 \cdot 2$ $189 \cdot 5$	$\begin{array}{c} 274 \cdot 3 \\ 275 \cdot 4 \\ 275 \cdot 7 \\ 281 \cdot 6 \\ 296 \cdot 1 \end{array}$	3 · 72 6 · 50 4 · 65 5 · 68 0 · 60	0.52 1.28 1.00 3.61 6.75	0.08 0.18 0.13 0.28 0.37
	35.35				21.15	13.16	1.04

			TABLE VII — L	UCKNOW	VARIETY			
	CORRECTED WT.,	IODINE VALUE	SAPONIFICATION VALUE	MEAN MOL.	WEIGHT OF ESTERS, GM.			
	Бш.			wr.	Methyl myristate	Methyl palmitate	Methyl stearate	Unsaturated esters
S S S S	$     \begin{array}{r}             4 \cdot 57 \\             5 \cdot 42 \\             6 \cdot 40 \\             5 \cdot 37 \\             \hline             21 \cdot 76         \end{array}     $	$2 \cdot 2$ $3 \cdot 32$ $4 \cdot 58$ $13 \cdot 45$	$209 \cdot 2 \\ 206 \cdot 1 \\ 202 \cdot 5 \\ 195 \cdot 4$	$\begin{array}{c} 268 \cdot 2 \\ 272 \cdot 2 \\ 277 \cdot 0 \\ 287 \cdot 2 \end{array}$	0·41  	$4 \cdot 09$ $5 \cdot 04$ $4 \cdot 81$ $2 \cdot 04$ $15 \cdot 08$	$     \begin{array}{r}                                     $	$ \begin{array}{r} 0.07\\ 0.12\\ 0.2\\ 0.50\\ \hline 0.80 \end{array} $
	21.70				0.41	19.98	44.8	0.89

soluble fraction, were analysed according to the method described in the Official & Tentative Methods of Analysis. The following results were obtained :

	Saharanpur variety %	Lucknow variety %
Moisture	11.56	9.52
Proteins ( $N \times 6.25$ )	66.00	63.28
Ash	9.12	9.77
Crude fibre	4.03	4.72
Carbohydrates (by dif- ference)	9.29	12.71

The ash from both the cakes, on analysis, gave 19.79 and 20.52 per cent phosphate calculated as P<sub>2</sub>O<sub>5</sub> respectively.

### REFERENCES

- 1. DHINGRA, D. R. & NARAIN, P. J.: Indian Chem. Soc., 1945, 22, 123. 2. Soni, (Miss) Padmini, Gupta, S. C. & Aggar-
- WAL, J. S.: J. sci. industr. Res., 1949, 8B, 210.
- 3. BERTRAM, S. H.: Z. Utersuch Lebensum, 1928, 55, 179.
- 4. HILDITCH, T. P.: The Chemical Constitution of Natural Fats (Chapman & Hall, London), 1947, p. 476.
- 5. SULLIVAN, B. & BAILEY, C. H. : J. Am. Chem. Soc., 1936, 58, 383.
- 6. JAMIESON, G. S. : Vegetable Fats & Oils (Reinhold Publishing Corp., New York), 1943, p. 348.
- 7. EIBNER & MUGGENTHALOR : Chemical Technology of Oils, Fals & Waxes, by Lewkowitsch, 1938, 6th Ed., Vol. 1, p. 574, 585. 8. JAMIESON, G. S. & BOUGHMANN, W. F.: J. Am.
- Chem. Soc., 1920, 42, 152.

### Destructive Distillation of Some Central Provinces Woods

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The products of destructive distillation of 19 woods from Central Provinces and Berar have been studied. The yields are tabulated.

EARLY half the area in Central Provinces and Berar is under forest and considerable quantities of charcoal, estimated at 1,80,000 tons, worth Rs. 7,20,000, are being produced by wasteful indigenous methods. If a judicious selection of woods available in the State is made, and if modern techniques of wood distillation are adopted and the by-products recovered, there is scope for establishing a flourishing wood distillation industry in this State.

Nineteen varieties of firewood have been subjected to dry distillation on a laboratory scale and the yields of charcoal and other byproducts have been recorded. The results of this exploratory investigation give a fair idea of the suitability of different woods for the wood distillation industry.

#### Experimental

The apparatus consists of an electrically heated cylindrical mild steel retort (capacity, 1 litre), a Liebig's condenser made of

### WARHADPANDE et al.: DISTILLATION OF SOME CENTRAL PROVINCES WOODS

copper, G.I. gooseneck, a water manometer, a 500 c.c. measuring jar which serves as a receiver for the distillate and a wet-gas meter to measure the quantity and the rate at which gases are evolved. The retort was provided with two thermometer pockets for recording temperatures at the periphery and centre of the charge.

### **Distillation Procedure**

The wood received in logs (3' long and 12" girth) was debarked and chipped to approximately  $1" \times 1" \times 2"$  pieces. About 2 kg. of the chips were dried in an air oven at 105°C. to constant weight. The dried chips were used for charging the retort, which was then heated. No attempt was made to control the distillation at the exothermic stage. The heating was stopped when the temperature at the centre of the charge reached 360°C. The distillation schedule for one variety of wood is given in Table I.

Analysis of Products - The distillate was allowed to settle overnight in a measuring jar and the volume of tar and pyroligneous liquor read off the next day. The acetic acid in the pyroligneous liquor was estimated according to the new method by Warhadpande and Mene<sup>1</sup>. Formic acid in the pyroligneous liquor was estimated by noting the difference in total acidity and acetic acidity in 10 c.c. of the liquor. The total acidity was determined by distilling 10 c.c. of the liquor with xylene (Grotlisch<sup>2</sup>) and titrating the distillate with N/5 NaOH. Methyl alcohol was estimated according to the method of Griffin<sup>3</sup>. Soluble tar was estimated by weighing the residue after eva-porating 10 c.c. of the liquor in a dish and ascertaining the density of the liquor in a Westphal balance. The yield of gas was calculated from the gasometer readings. The results obtained for 19 different species of woods are given in Table II.

		TA	BLE I		
Wood used : Hiwa	r (A. leucophloea);	weight of wood, 678 total gas evol	gm.; weight of cha ved, 3.97 cu. ft.	arcoal obtained, 241	gm.; settled tar, 45 c.c.;
Тіме	PERIPHERAL TEMP., °C.	TEMP. AT CENTRE,	TOTAL LIQUOR,	MANOMETER READING, WATER	Remarks
11.45 a.m.			<b>U</b> ICI		
1.25 p.m.	170	115	nil	0.2	Distillation starts
1.35	194	140	10	0.2	
1.45	214	155	25	0.2	
1.50	228	165	35	0.3	
1.55	238	172	46	0.4	Tar distils over
2.00	258	190	110	0.4	Distillate turbid
2.05	274	917	175	0.4	
2 10	284	245	205	0.3	
2.15	290	264	220	0.2	Tar begins to settle and more tar distils over
2.30	302	266	240	0.2	
2.45	314	270	245	0.2	
3.00	324	278	247	0.2	
3.30	342	294	250	0.2	
4.00	360	307	255	0.2	
4.30	378	330	260	0.2	Distillation slows down
5.00	395	352	260	0.2	

#### TABLE II - YIELD OF DISTILLATION PRODUCTS FROM 100 GM. OF DRY WOOD

NAMES OF THE WOOD		CHARCOAL,	ACETIC	FORMIC	METHYL	SOLUBLE	SETTLED	Pyro-	GAS,
Botanical name	Vernacular name	gm.	gm.	gm.	gm.	gm.	C.C.	LIQUOR, C.C.	ittes
Schrebera swieteniodes	Mokha	34.66	2.22	0.21	1.57	4.30	8.53	28.49	13.61
Ziziphus xylophyra	Ghont	34.65	2.08	0.29	1.72	5.81	6.04	31.42	13.91
Cleistanthus collinus	Garari	37.86	2.56	0.29	1.41	5.26	4.31	35.37	14.49
Dalbergia paniculata	Dhobin	36-29	2.01	0.08	1.24	3.73	5.64	32.08	15.35
Madhuca latifolia	Mahua	37.46	2.06	0.18	1.14	3.50	5.50	30.28	15.47
Aegle marmelos	Bel	34.37	2.42	0.14	0.88	5.25	9.69	35.22	15.37
Emblica officinalis	Aonla	89.16	2.76	0.43	0.80	4.64	5.03	30.00	15.76
Butea frondosa	Palas	39.21	0.86	0.14	1.33	2.30	5.03	26.88	16.91
Diospyrous melanoxylon	Temur	36.58	2.12	0.35	1.24	3.49	6.33	31.65	17.28
Adina cordifolia	Haldu	36.61	1.80	0.30	0.38	4.90	9.30	31.40	15.84
Albizzia odoratissima	Chichwa	36.64	2.01	0.25	0.60	8.49	5.15	25.12	9.96
Schleichera oleosa	Kusum	40.65	1.95	0.20	0.56	8.11	6.89	28.78	14.86
Nyctanthes arbortristis	Kharasi	83.04	8.65	0.10	0.73	6.81	3.87	37.59	15.58
Acacia leucophloea	Hiwar	34.23	2.83	0.06	0.92	4.96	6.31	35.75	16.89
Acacia arabica	Babul	34.93	2.91	0.66	0.44	4.34	5.17	30.28	12.29
Hardiwickia binata	Anjan	37.24	2.70	0.28	0.31	5.75	3.91	26.58	18.21
Ziziphus jujuba	Ber	35.81	2.27	0.06	0.49	4.56	5.49	31.21	13.03
Shorea robusta	Sal	37.20	2.50	0.27	0.76	4.91	4.31	28.55	12:55
Xylia xylocarpa	Suria	41.31	1.68	0.25	1.35	3.47	10.05	27.34	18.76

### Results

Among the woods investigated Aonla, Palas, Suria and Kusum give the best yields of charcoal (c. 40 per cent), while others yield 35 to 38 per cent. Kharasi gives the lowest yield (33 per cent).

Kharasi gives the highest yield of acetic acid (3.6 per cent); the yields from other varieties generally vary from 2 to 2.5 per cent.

The yield of formic acid varies from 0.08 per cent in the case of D. paniculata to 0.66per cent in the case of A. arabica.

The highest yield of wood alcohol was obtained with Z. xylopyra. The highest vield of settled tar (10.05 per cent) was given by Xylia xylocarpa.

The amount of gas generated varies from about 10 to 17 litres per 100 gm. of wood distilled; D. melanoxylon (Temur) gives the highest yield (16.91 litres) while A. odoratassima (Chichwa) gives the lowest yield (9.96 litres).

#### REFERENCES

- 1. WARHADPANDE & MENE: Curr. Sci., 1949, 18. 296.
- GROTLISCH: Ind. Eng. Chem., 1920, 12, 1183.
   GRIFFINS: Technical Methods of Analysis, Mc-Graw-Hill (1927), p. 497.

# Letters to the Editor

### ACTIVATED CHARCOAL FROM **GROUNDNUT HULLS**

ANNUALLY ABOUT 1.00,000 TONS OF GROUNDnut hulls are available in Hyderabad, which at present are burnt as fuel. Nearly 40,000 tons of charcoal are obtainable from this quantity, which may be used as domestic fuel after briquetting. The hulls can be employed also as basic material for the manufacture of activated charcoal.

Mukerji and Bhattacharya<sup>1</sup> prepared activated charcoal by heating groundnut hulls in a closed crucible either alone or in presence of zinc chloride. The yield of charcoal was poor and the results were not reproducible.

A process for the preparation of active charcoal for groundnut hulls, without employing an activating agent, has been worked out in our laboratories.

The second

#### Experimental

The experiments were carried out in a tube furnace (30 cm.  $\times 2.8$  cm.). 10 gm. of groundnut hull powder (20 mesh) was put into a quartz tube (1.6 cm. dia.), open at one end, and the tube was placed in the middle part of furnace. The material was



gradually heated to the desired temperature, the total period of heating being 4 hr. The quartz tube was sealed off after the completion of heating and the contents allowed to cool in the furnace.

The charcoal was powdered and the fraction passing through 60-mesh screen used for adsorption measurements<sup>2</sup>. Weighed quantities of charcoal were shaken up with iodine, methylene blue and potassium permanganate solutions of known strength for 3 hr. and allowed to stand overnight. The charcoal was separated by centrifuging. The residual methylene blue was estimated by the colorimetric method. The loss of iodine during centrifuging was corrected by running a control. Iodine and permanganate were estimated titrimetrically. The results are recorded in Table I.

The yield of charcoal obtained by us is nearly three times that obtained by Mukerji and Bhattacharya<sup>1</sup> who prepared active charcoal having an adsorption value of 760-850 mg. of iodine per gram of charcoal with an overall yield of only 10-13 per cent on the weight of the hulls. We have observed that when carbonization is effected in crucibles where air has partial access to the material inside, there is considerable loss of

S. No.	TEMPERATURE OF	YIELD OF	ADSORPTION PER GM. OF CHARCOAL, MG.				
	CARBONIZATION, °C.	CHARCOAL, ~	Iodine	Methylene blue	Potassium permanganate	100-X	
1	820	44	616	30	125	0.049	
2	370	41	649	29	126	0.048	
3	420		645				
4	500		985		267		
5	640	34	837	30	220	0.042	
6	820	31	555		127	0.044	

carbon. We have further noticed that the activity of charcoal is affected if the charcoal remains in contact with air during cooling.

It will be noted that the yield of activated charcoal is related to the temperature of carbonization (FIG. 1). The values in the last column of Table I for  $\log T/100$ -X further show that the volatile matter liberated on heating the husk out of contact with air is proportional to the logarithm of temperature.



Fig. 1 — Yield of activated charcoal in relation to temperature of carbonization.

The yield of charcoal diminishes with rise in temperature of carbonization while the activity of charcoal does not vary very much with the temperature of carbonization between  $320^{\circ}-420^{\circ}$ C. The samples obtained at  $500^{\circ}$ C. show maximum activity. The iodine and potassium permanganate adsorption values for samples obtained at higher temperature ( $820^{\circ}$ C.) are lower than those at  $500^{\circ}$ C. The results indicate that the optimum temperature for activation is about  $500^{\circ}$ C.

The work is being continued.

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Central Laboratories for Scientific & Industrial Research Hyderabad (Deccan) REFERENCES

- 1, MUKERJI & BHATTACHARYA: J. sci. industr. Res., 1945, 5, 235.
- Cf. CHOWDHURY, CHAKRABORTY & GHOSH: J. Ind. Chem. Soc., Ind. & News Ed., 1947, 10, 40.

### A NEW REAGENT FOR URANIUM

OXALO-HYDROXAMIC ACID IS FOUND TO GIVE deeply coloured complexes with a number of cations in slightly acid or ammoniacal solutions. Special attention has been given to uranium with which the reagent gives an orange complex. The formation of this complex has been found to provide a very effective method, both for detection and colorimetric estimation of uranium. In the absence of excess of electrolytes or in the presence of peptizing agents, the orange complex can be kept in solution even in moderate excess of ammonia and serves as a very suitable reagent for colorimetry. The maximum absorption of the coloured solution is at 420 mµ and obeys Beer's law. closely. The limit of identification determined on a spot plate is 0.2r and the limit of dilution is 1:2,40,000.

The applicability of this reagent in uranium ore analysis and the effect of ions like phosphates, oxalates, iron, vanadium, alkaline earths, etc., are being studied and a paper giving complete details will shortly follow.

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National Chemical Laboratory Poona August 1, 1950

### LOW-GRADE CLAY FOR THE MANU-FACTURE OF ALUMINA FERRIC

ALUMINA FERRIC IS USUALLY MANUFACtured from rich aluminous ores, e.g. bauxite

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and kaolin. When low grade clay is employed, only a part of the sulphuric acid is utilized and for obtaining a given quantity of alumina ferric, a lot more sulphuric acid is required than when bauxite is used. The use of low-grade clay is thus wasteful of sulphuric acid and the cost of production of alumina ferric is consequently high.

A modified process has been worked out in these laboratories for the treatment of low-grade clays such as those occurring in Asifabad district, Hyderabad State (alumina content, 20 per cent). In this process, the clay is digested with sulphuric acid (sp. gr. 1.25) at  $105^{\circ}-10^{\circ}$ C. for 12 hr. Only 70 per cent of the acid is used up. The acid remaining over is utilized for digesting the bauxite added to the digestion mixture at this stage. The mixture is then heated for a further period of 8 hr. A part of the bauxite can be replaced by kaolin. In this manner about 99 per cent of the acid is used up.

The settling of silica in the digested mixture is slow in the cold, but is hastened by warming. The rate of sedimentation at 60°C. is nearly twice that at 30°C.

Calcination of the clay prior to digestion does not offer any advantage; calcination at temperatures exceeding 800°C. renders the clay less soluble.

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Central Laboratories for Scientific & Industrial Research Hyderabad (Deccan)

### APPLICATIONS OF MUSCOVITE CHLORITE SCHISTS

THE MUSCOVITE CHLORITE SCHISTS, FOUND IN abundance in Belgaum and Ratnagiri districts of Bombay State, are usually considered to be of no economic value, although they contain a high proportion of mica. Unlike the ordinary mica the mineral is usually found in the highly crushed and stained condition, the size of the flakes being very small. The mineral can, however, be easily disintegrated in a hammer mill, and the powdered material has the following characteristics: moisture 1.22 per cent, water solubles 0.74 per cent, acid solubles 3.15 per cent, sp. gr. 2.68 and oil absorption 50.39 gm. per 100 gm. The finely ground powder, in conjunction with barium chromate is found to have great potentialities in the formulation of rust-resisting primers for structural steel, replacing the usual compositions containing red lead. Also addition of the powder to distempers to the extent of 5 to 10 per cent results in the prevention of settling and improved washability, the coatings having a smoother feel and greater impermeability to moisture. The powdered material has also been employed as a filler for thermosetting plastics.

Further work on the uses and applications of the powdered mineral as such or after "micronization" is in progress.

N. R. KAMATH

R. K. KULKARNI

Department of Chemical Technology University of Bombay June 22, 1950 THE LARGEST BRITISH ELECTRICAL MANUFACTURING ORGANISATION

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