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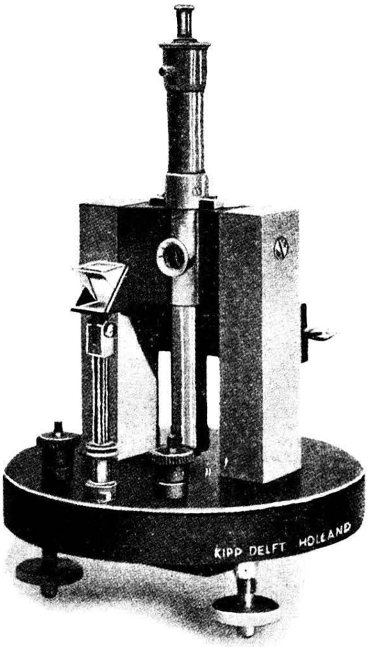

KIPP DELFT HOLLAND

Journal of Scientific & Industrial Research

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The Editorial Board in its work of examining papers received for publication is assisted, in a honorary capacity, by a large number of distinguished scientists working in various parts of India.

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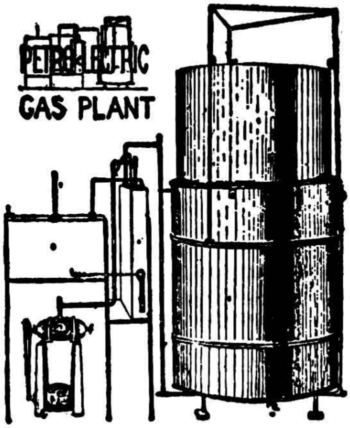
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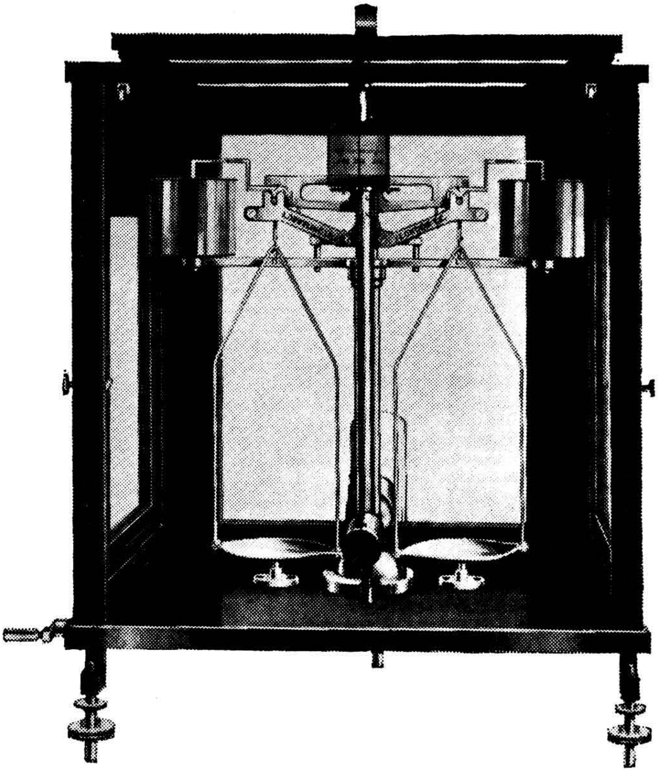

Science & Practice of Food Production

MUCH has been written on India's food problem and the necessity and the urgency for attaining self-sufficiency. The problem has been before the people in all its poignancy since the fateful Bengal Famine of 1943. Commissions and committees have examined the many aspects of the problem and recommendations have been made. Fertilizer factories and irrigation projects have been planned and are being speedily executed. The partition of the country has emphasized the need for more intensive effort to step up food production. The crippling effect of importing large quantities of foods on India's national economy has been repeatedly emphasized, and the decision of Government to stop imports from 1951, and the more recent suggestion following the devaluation of the rupee, to stop imports even from 1950, has accelerated Government and private effort to raise production and attain the target of self-sufficiency at the earliest possible date.

Farming and food production are of vital importance ranking with the supply of drinking water and fresh air as an essential for the well-being and efficiency of the nation. The problem of food production concerns all,— the primary producer, the factory worker and the scientist included. In this drive for more and better food, what is the part that science and the scientist have to play? Dispassionate inquiry leads to the conclusion that our resources are adequate to provide sustenance to all, if and only if, the knowledge revealed by scientific research and inquiry is adequately utilized for their planned development.

Science alone cannot bring about increased food supplies, but it can, and must, play a prominent rôle in the achievement of the result. The scientist has the responsibility— nay, the obligation, to develop methods and techniques necessary for the attainment of the objective, viz. production of food of the quality and in the quantity required to meet the needs of a continuously growing population on a sustained basis and at a price level which can be reached by all. The methods and techniques should be such that they can be adopted and applied effectively within the existing social and economic structure of the primary producer.

This obligation appears to be a formidable one, but the knowledge which inspires the scientist and the faith that sustains him in his endeavours, encourages him to undertake it with confidence. Recent achievements in the United States of America and elsewhere provide the assurance that similar results are attainable in India by harnessing science to farm production.

In any objective examination of the agricultural situation, one thing stands out — agricultural practice lags far behind agricultural science. The most urgent and compelling problem before the scientist is to shorten this lag, to bridge the gap between science and practice, knowledge and application. In an earlier number of the *Journal* we drew attention to the following excerpt from the address by Prof. N. V. Joshi to the Indian Science Congress (1946):

> " While the crop yields from improved seeds and milk yields of improved cattle breeds on experimental farms are high, the general level of agricultural and milk yields does not appear to have increased. In what way, then, have we benefited the agriculturist or the agricultural business during all this period of fifty years, from 1890-1940, a period during which research workers in agricultural sciences have accomplished remarkable results which are held by scientists to be of great practical utility? Since we have not benefited the cultivators, it may be concluded that somehow we have failed to convey the information about the results of agricultural research and improvements to the proper quarters or to

> get them into common practice by the cultivators."*

Here is the crux of the problem,— to devise the means to overcome our past failure to convey to the primary producer the methods and techniques of agricultural science in a manner that he can adopt on the field within the means available to him. To say that the farmer is conservative, that he is illiterate and ignorant, is to evade the issue. The fact that he has enthusiastically adopted practices which have produced more and better sugarcane gives the lie to the charge. The failure of scientists to transform the results of soil and crop science to workable farm schedules must be accepted, and the experimental station, which is the scientist's answer for proving and demonstrating the results of research, has obviously proved ineffective.

Discussing the causes that have led to the lag in the use on farms of the best practices of soil and crop management, Dr. McCall of the Bureau of Plant Industry, U.S. Dept. of Agriculture, states :

> " When a new product or process is developed in commercial industry, it is axiomatic that an oftentimes extended and expensive period of pilot plant study is necessary before the final economically workable process or equipment is developed from original principles and procedures. This is true no matter how sound and workable the new development may be.
>
> " In the field of agriculture the research scientist has thrown the entire responsibility and expense for the pilot plant stage upon the individual farmer. The farmer is given only the original patent, as it were, and he must adjust the new principles to his own farm and operations. It is true that he may see a preliminary working model which demonstrates the principles on experiment station plots, but this may have little relation to his present farm plan and economic set up. It is up to him to work out the new operation schedules and how he will convert to them. This may require a more or less complete revision of his farm set up. It may mean a change in constituent enterprises in the farm plan and the necessity for him to learn all the basic requirements of the new enterprises with an attendant probability of loss during the learning period. . . . A farmer faced with such a situation is naturally conservative about change except as it fits rather easily into his present set up. In most cases he cannot be otherwise."*

The old demonstration farm idea has failed to bring forth the desired results even in a progressive country like the United States. Certain modifications of the idea, however, have been tried and offer possibilities for implementing research. Dr. McCall mentions the following :

> Test Demonstration Farms of the Tennessee Valley Authority — A farmer in each community chosen by his neighbours enters into an agreement to carry out certain cropping and fertilizer practices, keeping records which are available to the co-operating State College and Agricultural Experiment Station, the T.V.A., and his neighbours, and permitting his farm to serve as a local demonstration.
>
> State Pilot Farms on which soil management systems indicated by soil research as probably feasible, could be evaluated in economic terms on different soil types and for type of farming factors. As a next step in this plan, community farms similar to the Test Demonstration Farms of the T.V.A. plan would carry the proven findings of the Pilot Farms direct to individual communities.
>
> Individual Farm Advisory Service which would provide in a county or district for well-trained technicians to work with each farmer desiring the service in developing a well-rounded farm plan for his farm. Such a plan to be adequate should cover field layout and farm fences, crops (including varieties), crop rotations, land drainage, liming, fertilizers, tillage schedules for preparing soil in cropping and for weed control and water conservation, erosion control practices, manure conservation and use, timing of all operations, choice and effective use of equipment, numbers and kinds of livestock, animal breeding, feeding and health, preparing and storing feed and other crop materials, housing livestock, facilities for farm processing, crop insect and disease control, and any other pertinent factor.

No one plan, however well conceived, meets all situations. Likewise, no plan however effective in one country proves equally effective when transplanted into another country. It is necessary first to analyse the current practices and examine in what respects they

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* *J. Sci. & Ind. Res.*, 1946, **4**, 451.

* *Freedom from Want*, a symposium, edited by E. E. DeTurk, *Chronica Botanica*, 1948, **11**, 276.

have proved defective as instruments of education and research development. It is also necessary to accept the truth in the farmers' complaint that research workers, generally, are out of touch with farming needs and conditions, that the results of research lie published in learned periodicals and bulky tomes and are described in a jargon unintelligible to him, that such publications deal with fragments of the problem and offer no overall solution to his difficulties in practical farming. It should then be possible to work out a plan which would prove operative. In the selection of problems for investigation, or for demonstration on experimental farms, the farmer has so far stood apart from the investigator. It is not always realized that interest which the farmer takes in the development plan is the desideratum for the practical utilization of research. From *prima facie* considerations, a practical approach appears to be one in which the farmer is one of the investigating team, and the farm the experiment station. Research problems should flow from the field to the laboratory and not vice versa.

The raising of abundant crops — either by intensive farming, or by the use of improved seeds, or by any other means — involves the taking away from the soil of large quantities of nutrients which have to be put back into the soil by a proper system of manuring. This and other aspects of farm management for securing food, and the utilization of our basic capital, namely the soil, so that its productivity is sustained to perpetuity, assumes a certain level of education which the practical farmer should find no difficulty in acquiring if his interest, importance and responsibility as the producer of the nation's vital requirement is adequately recognized. The schemes of craft-centred basic and adult education, which are now being rapidly implemented on a nationwide basis, would be of immense assistance in educating the farmer to adapt himself to the new and the better farming practices.

Sufficient knowledge has accumulated for producing more and better food; what is necessary *now* is to apply it effectively and produce results. In the words of the Hon'ble Minister for Food and Agriculture, if the thousands of public servants manning the agricultural services of our provinces and states function as true guides, philosophers and friends of those who produce the nation's food, the task of making India self-sufficient with respect to the food needs of her rapidly growing population can be satisfactorily accomplished.

United Nations Scientific Conference on the Conservation & Utilization of Resources (UNSCCUR)

LAKE Success, the venue of discussions on international problems, attracted the attention of the scientific world when, on August 17 this year, for the first time in history, 640 scientists, engineers, economists and other experts from 50 countries assembled for a 3-week Conference on the Conservation and Utilization of Natural Resources.

The story of the Conference dates back to the early years of the present century, when the then U.S. President, Theodore Roosevelt, called to conference the Governors of 48 States of America to consider the problems relating to the preservation, protection and wise use of natural resources of the nation. Several efforts have since been made to convene a world conference of scientists to discuss ways and means of solving problems facing the peoples of our " plundered planet ", where resources have been especially drained through wars among nations. Early last year the President of United States put up a suggestion to the U.N. Economic and Social Council to call a United Nations Scientific Conference on Resource Conservation and Utilization. Accepting the suggestion, the Economic and Social Council requested the U.N. Secretary-General on Feb. 11, 1948, to proceed with the plan of the Conference,

" keeping in mind that the task of the Conference is to be limited to an exchange of experience in the technique of conservation and utilization of resources ".

During the Conference, 18 plenary meetings and 54 sectional meetings were held. Over 500 technical papers, based on practical experience in the planned use of resources, covering a wide range of fields, — from agriculture to wild-life protection — were presented and discussed.

The following brief résumé of the more important papers presented and discussions held under each section has a special reference to the contribution made by the Indian delegation to the Conference :

Dr. S. S. Bhatnagar, leader of the Indian delegation, who presided over the first meeting of the session, pointed out that " the convening of the Conference was perhaps the greatest work the U.N.O. had done from the scientific point of view ". Mr. H. L. Keenleyside, the Canadian Deputy Minister of Mines and Resources, advocated vigorous scientific research on a worldwide basis to tackle problems arising from the possibility of critical mineral shortages in the world in the not too distant future.

The problems facing food production received serious attention. It was pointed out by Sir Herbert Broadley, Deputy Director-General, F.A.O., that to meet the food requirements of the world population in 1960, milk production should be increased by 100 per cent, fruits and vegetables by 163 per cent, meat by 46 per cent, and fats by 34 per cent. He called upon the nations of the world to establish a World Food Fund to " provide resources for accurately measuring the possibilities, organizing the necessary research, planning the strategy of international food campaign and training those upon whom will fall the responsibility of directing the technical operations of the campaign ". He further emphasized that the thin layer of top-soil enveloping our planet, which he likened to a tissue paper, " on which depends the fertility of the land and which took from 300 to 1000 years to form ", should be carefully preserved and husbanded.

In the subsequent plenary meetings, the delegates examined the application of conservation techniques to mineral, water and wild-life resources ; fuel and energy ; food and forest products and other resources, with special reference to techniques applicable to under-developed countries of the world. The sectional meetings devoted particular attention to techniques which would ensure sustained production and enhance supplies by bringing into use resources which were hitherto economically unusable.

Soil & Forest Resources — Mr. Hugh H. Bennett, Chief of U.S. Conservation Service, pointed out that the productive capacity of good land, which was limited to 4,000,000,000 acres, was being reduced by abusive use in many countries. He emphasized the urgency of classifying land according to its best use.

Dr. J. N. Mukherji, Director, Indian Agricultural Research Institute, New Delhi, speaking on soil productivity in tropical climates, stated that a rotation crop system suited to the individual soil type was no less important than the use of fertilizers. In the absence of adequate supplies of inorganic fertilizers, green manures are specially important for ensuring soil productivity.

Reclamation and effective use of new agricultural lands present several problems for solution. Dr. R. Sethi, Agricultural Development Commissioner to the Government of India, emphasized that attention to malaria control is essential for the development of new lands. He stressed also the need for securing suitable machinery for the reclamation of land in India.

The provision of improved tools and implements for small-scale farming would prove effective in augmenting food supplies. A paper by Mr. Mason Vaugh, presented before the Conference, described a soil-inverting plough and a small cultivator with suitable attachments for both, which would meet practically all the needs of the small farmer.

The part played by forests in the protection of resources was discussed at one of the sessions of the Conference. Mr. C. R. Ranganathan of the Indian Forest Service pointed out that more attention should be paid to hydrological and protective aspects of forests than hitherto and expressed the view that State ownership was essential for securing the best results.

Preservation and chemical utilization of wood resources formed the subject of a paper by Mr. J. F. Harkam of the Department of Mines and Resources of Canada. Preservative treatment increased the life of timber in service 3 to 4 times, reduced drain on forests, increased the utility of wood, and opened up a market for timber products derived from species of low natural durability.

Fisheries — The delegates exchanged views on pond culture, i.e. the systematic raising of fish farms, which can, under favourable conditions, be an important source of food and a lucrative industry.

Dr. S. L. Hora, Director of the Zoological Survey of India, presented a general review of the problem. Describing the multiple uses of natural ponds, Dr. Hora stated that in Philippines alone there were 600,000 hectares of undeveloped fresh and salt water swamps capable of producing 200 million fish annually. He estimated that even if only 10 per cent of the money, material and men now employed in considering the ways and means of developing marine fisheries in the tropics were diverted to improve ponds for fish culture, the result would go a long way in solving the food problem of the world.

In this connection, Dr. Hora drew attention to the cultivation of fish in rice-fields as practised in Japan, which has benefited paddy crops by as much as 10 per cent. By proper embankments, vast low-lying areas in the vicinity of sea coasts could be made productive. Besides their use for raising fish crops, the areas could be converted into salt ponds which, when silted up, would become fresh-water ponds and finally rice-fields.

Coming to the conservation of marine resources, Dr. H. Srinivasa Rao of the Central Marine Fisheries Research Station, Madras, pointed out that the integration of hydrological, biological and related studies is a feature of the fisheries research programme in India. Exploitation, rather than conservation, is the immediate problem of India's marine fisheries. Nevertheless, the Government is taking a long-range view of the problem and taking steps to obtain scientific data necessary for development programmes which would lead to sustained yields from this resource.

Minerals & Metals — Geologists were unanimous in the opinion that the world's store of metals would continue to contribute to better living conditions only if the " shocking " waste caused by war was done away with, and if national and political boundaries, which stand in the way of international co-operation, were removed.

In a paper entitled *Metals in Relation to Living Standards* Dr. D. N. Wadia revealed that more basic metals have been used up since 1914 than during the whole of human history.

During a discussion on metal reserves of the United States, it was estimated that the commercial reserves of iron in the United States would be consumed within 75 years and of other important minerals and metals possibly in 1 to 19 years.

The undiscovered reserves of petroleum have been estimated to be 150,000 million barrels, i.e. about 500 times the current annual consumption, but this estimate has been considered to be too high. Mr. Aguilar, the Mexican oil expert, urged the need for setting up of an international committee under the United Nations or any other agency to undertake a world survey of oil reserves.

The topics discussed at the sectional meetings on *Fuels and Energy* included the use of giant turbines for harnessing wind power to generate electricity ; methods of overcoming critical shortages of coal ; and techniques used in the United States, France and England to stretch their coking coal reserves.

Problems connected with the development of river basins ; importance of forests and plant cover in controlling floods ; river navigation as a means of transport in underdeveloped countries ; and irrigation and drainage in relation to food production were among the other problems that engaged the attention of the delegates in the various sections of the Conference.

The example of the Tennessee Valley Authority in the development of a river basin formed the subject-matter of a pamphlet presented at the Conference.

New Resources — The possibilities of tapping new resources to meet the needs of industry were examined in some detail during the Conference. Dr. Neville Woodward, Director of the Scottish Seaweed Research Institute, drew attention to the fast-developing fields of *Chemurgy* which deals with the utilization of agricultural products and wastes as raw materials for secondary industries. Recent developments in seaweed utilization were described.

Appraising Resources — Resource appraisal requires the application of systematic inventory procedures for which surveyors had to collaborate more closely with official agencies responsible for development projects. Dr. P. C. Mahalanobis of the Indian Statistical Institute, dealt with the application of statistics to resource appraisal and utilization.

Resources Conservation in Under-developed Countries — Dr. Hernan Santa Cruz of Chile led the discussion on the problem of techniques for conserving resources in the less developed countries. Measures adopted for conservation of resources in these regions were inadequate, and foreign capital has possibly accelerated the " intensity " of exploitation of resources. The world prices of commodities were fixed without consulting the producing countries with the result that the prices realized were not such as to raise the standard of living of the producers.

Dr. S. S. Bhatnagar sounded a note of caution against transplantation, without precaution of techniques devised for, and specially suited to, a highly industrialized country to another where manpower was cheap and plentiful. He stressed that the less developed regions would be benefited if attention is paid to the following :

(1) Training and acquisition of technical personnel.
(2) Procurement of literature, equipment and machinery.
(3) Means to make foreign and indigenous capital flow into less developed countries without the imposition of conditions which might be considered humiliating by nations requesting help.
(4) Application of technical knowledge already in existence in order to improve the process techniques which are indigenous ; and
(5) progressive measures in the legal and political fields, without which it was possible to " stifle " any technical or scientific activity.

Of special interest among other papers discussed at the Conference were those dealing with the production of food yeast and artificial foodstuffs. Another paper described recent developments in air surveys and aerial photography which made it possible to carry out rapid surveys of forest resources.

The success of the Conference, as Dr. Bhatnagar stated, will be determined by " the rate at which the less developed regions ' catch up ' with the developed regions ". In the words of the Secretary-General, one of the most important results of the Conference had been " to demonstrate to the peoples of the world that the United Nations Organization was concerned with building the economic and technological foundations of international peace by enlisting the collaboration of the scientific and technical world ".

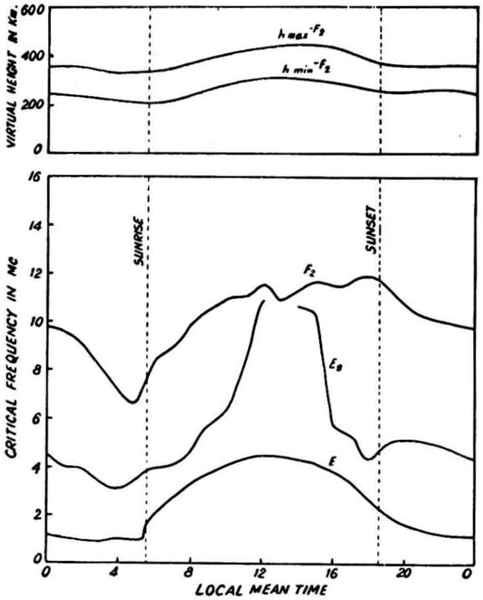
Characteristics of the Ionosphere over Calcutta (August 1949)

S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY, (MRS.) T. K. BHAR & A. P. MITRA
Wireless Laboratory, University College of Science, Calcutta

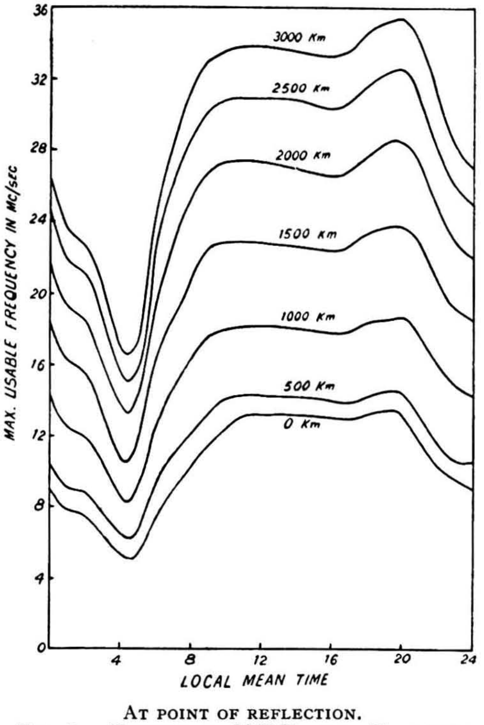
THE following are the ionospheric data observed at Calcutta for the month of August 1949.

Fig. 1 represents the mean hourly values of the penetration frequencies of the E and E_s regions and the penetration frequencies and virtual heights of region F_2. The figures are obtained from average values of the data taken for each hour of the day for 5 days a week. Fig. 2 gives the prediction of the maximum usable frequencies which can be used for different distances of transmission by reflection at the F region over Calcutta for the month of November 1949. Table I gives the different occasions during routine observation when sporadic E ionization was observed and the values of the corresponding penetration frequencies and heights.

Sporadic E ionization was found to occur frequently during early morning and also in the afternoon hours. Absorption of normal E echoes at mid-day was less than in the previous month. The behaviour of region F_2 was found to be normal in this month except for the last few days.



(5 HOURS 54 MINUTES AHEAD OF G.M.T.)
FIG. 1 — AUGUST 1949.



AT POINT OF REFLECTION.
FIG. 2 — PREDICTED M.U.F. VIA F_2 LAYER, NOVEMBER 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$^{h}E_s$ Km.
August 1949	1	08.00	4·75	120
		09.00	6·10	120
		10.00	6·80	135
		11.00	7·10	135
		18.00	5·50	135
		20.00	6·00	120
	2	06.00	4·50	105
		08.00	4·50	120
		09.00	6·75	120
		10.00	7·00	120
		16.00	6·60	120
		17.00	4·75	120
		18.00	3·60	120
		19.00	4·25	105
		20.00	4·00	105
	3	08.00	4·55	120
		09.00	6·00	120
		10.00	4·50	120
		11.00	6·25	135
	4	11.00	6·80	135
		16.00	6·80	135
		18.00	4·50	120
		23.00	2·85	105
	5	00.00	3·25	90
		04.00	3·00	90
		05.00	3·65	90
		19.00	3·00	105
	6	07.00	4·50	120
		09.00	6·10	120
	8	09.00	4·20	120
		12.00	11·00	150
		16.00	6·00	135
		17.00	7·10	135
		18.00	6·00	120
		19.00	6·25	120
		21.00	6·00	120
		22.00	5·25	105
		23.00	5·35	105
	9	00.00	8·15	105
		01.00	5·20	105
		02.00	4·50	105
		03.00	3·25	90
		07.00	4·25	105
		08.00	5·25	120
		09.00	6·00	120
		10.00	8·00	120
	9	11.00	9·75	135
		13.00	10·70	150
		14.00	10·70	150
		15.00	10·50	150
		19.00	10·00	150
		20.00	10·00	150
		21.00	5·25	135
		22.00	8·00	135
		23.00	6·25	135
	10	00.00	4·00	120
		07.00	3·00	120
		08.00	5·00	120
		10.00	6·00	135
		13.00	12·40	150
		16.00	5·75	150
		17.00	5·20	135
		18.00	4·70	135
		19.00	3·50	120
		22.00	2·70	105
		23.00	6·85	105
	11	08.00	4·20	90
		09.00	5·00	120
		10.00	5·40	120
	18	13.00	10·95	150
		16.00	3·35	135
		17.00	3·20	120
		18.00	3·20	105
	19	09.00	4·70	120
		17.00	5·85	120
		21.00	6·20	120
		22.00	5·70	105
		23.00	4·20	105

TABLE I — *contd.*

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$^{h}E_s$ Km.
August 1949	20	09.00	6·70	120
	22	11.00	10·40	150
		20.00	6·25	135
		21.00	5·00	120
	23	18.00	3·40	120
		23.00	3·30	90
	24	00.00	3·40	90
		01.00	3·50	90
		02.00	3·30	90
		05.00	3·80	90
		06.00	4·70	105
	25	17.00	6·55	120
		18.00	3·40	120

TABLE I — *contd.*

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$^{h}E_s$ Km.
August 1949	25	19.00	3·50	120
		20.00	4·25	120
		21.00	4·50	105
		23.00	4·75	105
	26	00.00	4·00	105
		01.00	3·20	90
		20.00	3·25	105
		21.00	3·20	105
	29	20.00	4·00	105
		22.00	4·25	105
	30	07.00	3·75	120
		18.00	4·80	120
		23.00	4·00	90

Design of All-India Community Radio Receivers for Villages

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THE Provincial Broadcasting Department of the Government of Madras started installation and maintenance of community radio receivers in villages in 1938. About 100 sets were installed in 1938-39, as an experimental measure, after the installation of 5 kW. medium-wave transmitter at Tiruchi and 10 kW. short-wave transmitter at Madras.

Subsequently, Government of Madras sanctioned a scheme for installing 300 sets in the villages. The maintenance of these sets is the responsibility of the Provincial Broadcasting Department. The community sets installed in the province increased year by year and, by 1947, there were about 700 sets installed.

Early 1946, the Government of India announced a basic plan for providing a large number of additional transmitters throughout India. The Government of Madras had also contemplated installation of the community receivers on a large scale in various villages in the province to enable the villages to avail of these increased broadcasting facilities. Early in 1947, a comprehensive scheme was, therefore, drawn up by this Department for installing 8,000 sets in as many villages.

Problems of Large-scale Installation of Community Radio Receivers

The important aspects of rural broadcasting are: (i) keeping down the initial cost of installation of a community set; (ii) lowering the recurring cost of maintenance of the set; (iii) policy as regards: (a) the replacement of the sets at the end of their useful life, (b) effecting a change from a battery-operated set to a mains-operated receiver whenever a village is electrified, at low cost; and (iv) the minimum requirements of reception with respect to wavelengths and output which a community receiver should fulfil.

(i) *Initial Cost of Community Set* — The Provincial Broadcasting Department has been purchasing, as and when necessary, various types of commercial, domestic, 5 or 6 valves, battery or mains-operated radio receivers of American or British manufacture for installation in the villages. Prior to 1941, the cost of such a receiver was ranging between Rs. 150 to Rs. 250 and from 1941 to 1944 from Rs. 300 to Rs. 350. In 1944 about 600 lease-lend receivers (for which only "out of pocket expenses" were paid) were obtained by the Madras Government from the Government of India at cost varying from Rs. 88 to Rs. 142, and they were installed in various villages.

Extra cost is incurred by the Department before the installation of the sets in the villages for carrying out certain modifications. They are mainly as follows :

1. An insect-proof frame with a lock, for the back of the set, to protect it from insects and to avoid tampering of the internal mechanism.

2. The community sets are designed to receive programmes of either Madras or Tiruchi stations depending upon the language spoken by the local people. The tuning of the sets, therefore, was adjusted to either of these stations, so that their operation is simplified.

3. An additional cabinet or horn-type baffle for the loud speaker of the set, with an extension cable 15′ in length, is provided, so that even though the set is installed in a room, the loud speaker can be brought out to the open to serve the listeners in the open. These modifications limit the working of the sets to the essential purposes of community listening.

(ii) *Cost of Maintenance* — As the number of sets installed in the province rose from about 100 in 1940 to about 700 in 1947, the cost of maintenance per set per year assumed importance. Apart from the expenditure on common items like transport, establishment, etc., items such as (1) cost of spares used for servicing ; (2) types and design of sets (whether simple, or complicated, using exclusive types of components, etc.) with respect to their simplicity for servicing and maintenance assumed greater importance. Table I gives the types installed and maintained by this Department in 1948.

The large variety of sets to be maintained compelled the Department to keep stock of a variety of spare parts, e.g. valves, band switches, condensers, etc., some of these being special to a given model of the receiver.

Another important factor contributing to the cost of maintenance is the non-essential electrical and mechanical parts in the commercial types of sets, which are superfluous in a community set. The commercial sets have highly finished and costly cabinets, multi-band tuning and dial-drive mechanisms, band spread circuits, tuning ranges covering wide frequency bands, and other gadgets like push-button tuning, etc., which, even though not necessary for a community set, have to be maintained to keep the set as a whole in good condition. As the designs of commercial sets are complicated, and a large number of different types of sets had to be maintained, skilled technical staff became necessary, and the number of sets under the charge of any one person had to be restricted to about 20. The commercial sets are not designed for frequent transport over long distances by road or rail, whereas the sets installed in the interior villages have had to be moved from place to place and they frequently developed faults due to exposure or jolting.

TABLE I — RADIO RECEIVER MODELS

Battery models			Mains models		
No.	**Manufacturers**	**Model**	**No.**	**Manufacturers**	**Model**
1	R.C.A.	8QB	1	R.C.A.	8Q2
2	R.C.A.	QB1	2	R.C.A.	8Q1
3	R.C.A.	7QB	3	R.C.A.	85T8
4	R.C.A.	85BT	4	R.C.A.	9Q4
5	R.C.A.	QB9	5	R.C.A.	Q11
6	Philco	38-624	6	Philco	38-620
7	Philco	40-748T	7	Philco	37-2670
8	Philco	39-744	8	Philco	40-780T
9	G.E.	JB-72	9	G.E.	HE 74
10	G.E.	GBE 63	10	G.E.	40-HE 740
11	H.M.V.	699	11	G.E.	X 115 C.A.
12	Marconi	822	12	Cossor	EX 396
13	Andrea	E6B	13	H.M.V.	673
14	Pye	V41RF	14	H.M.V.	675
15	Lincoln	Mains and battery	15	H.M.V.	5211
			16	Marconi	829
16	Crystaltone	7906	17	Roburn	
17	Philips	739 VN	18	Philips	HN 436
18	Philips	663 VN	19	G.E.C.	BC 4172
			20	Westing house	M116A

(iii) *Cost of Replacements* — Another important aspect of rural broadcasting is that some of the sets installed in 1938 have completed nearly 10 years of service. Some of the sets had to be condemned as unfit for use for the following reasons :

1. Important original components like tuning coils, band switches and other circuit components were no longer available for servicing the sets nor were standard components available in the market.

2. With the progress of rural electrification, a certain number of battery sets will have to be replaced by mains-operated receivers every year.

(iv) *Basic Requirements of a Community Receiver* — Taking into consideration the various aspects of rural broadcasting, it was considered imperative to evolve a standard type of community set, if installation and maintenance of community receivers in thousands of villages have to be successfully and economically carried out. The requirements are :

1. The initial cost of installation should be low.
2. The maintenance cost of the sets should be low.
3. The set should have a long life. It should not be out-moded with time for want for any particular spare part.
4. The cost of conversion from battery to mains-operation should be low.

To meet these requirements, the design of a suitable receiver was taken up early in 1947 in the Research Section of the Provincial Broadcasting Department.

Standard Type of Community Radio Receivers

A superheterodyne type was decided upon in preference to a straight receiver owing to the greater sensitivity, selectivity and stability of the former. The provision of reaction control for increasing the sensitivity of a straight receiver may result in instability in the set, thus causing disturbance through radiation.

Stages in the Set — Keeping in view the economy and simplicity in construction, the following essential stages are provided without sacrificing the sensitivity and selectivity : (i) first detector and oscillator ; (ii) intermediate frequency amplifier ; (iii) second detector and audio voltage amplifier ; (iv) audio driver amplifier ; and (v) power output.

Power pack is built as a separate unit. A complete circuit diagram and the values of components are given in Fig. 1 and Table II. The components used are standard types readily available in the market.

Controls — The number of controls necessary to operate the various stages has been reduced to a minimum in order to simplify the handling of the set by a village operator. A heavy-duty main switch, separate from the volume control, is provided so that it may not be easily spoilt by frequent use and to handle the heavy current that flows through when the set is working off a 6-volt battery. The usual type of continuously variable volume control is provided. No tone control is required as the set is designed for balanced tone output which meets the requirements of community listening in public parks, etc. An air dielectric low-capacity (0-50 p.f.) tuning condenser (incorporated in the oscillator grid circuit) is also provided. These controls, viz. main switch, volume control and tuning control are fitted to a panel in the front of the chassis for easy manipulation.

Power Pack & Audio Power Output Rating — In view of the necessity for providing an easy change-over from battery to mains operation when required, the power pack is built separately and fitted to the chassis frame so that it can be taken out without disturbing the wiring or the layout of the main receiver circuits. The power pack is connected to the receiver circuit by means of a non-reversible plug and socket and a short length of screened multi-core cable. The socket in the receiver chassis is common and the plug

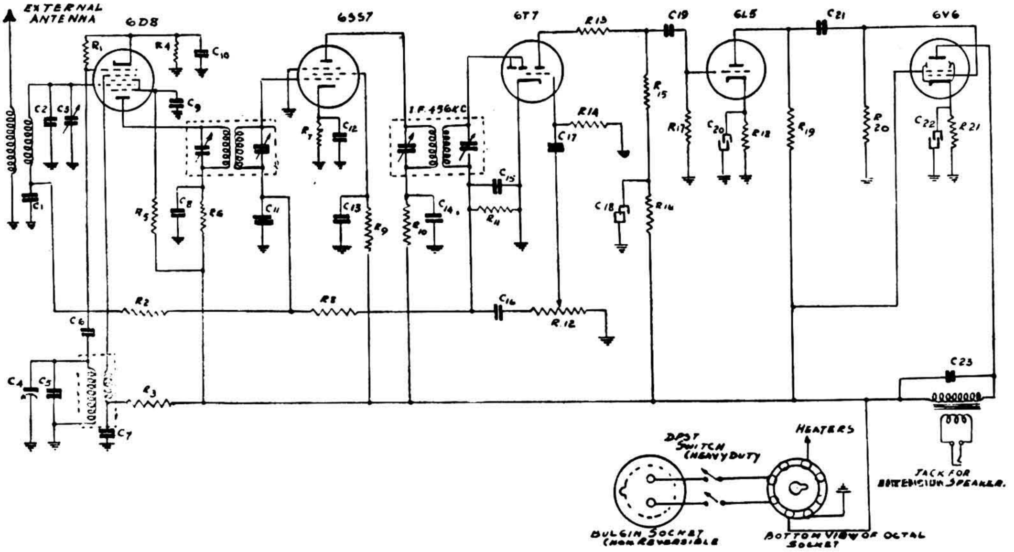

FIG. 1 — CIRCUIT DIAGRAM OF COMMUNITY RECEIVER.

TABLE II — COMPONENTS & THEIR VALUES

Receiver Section

R. 1	35 k. ohms	C. 1	0·04 μfF paper
R. 2	2 megohms	C. 2	150 p.f. mica
R. 3	20 k. ohms	C. 3	30 p.f. mica trimmer
R. 4	350 ohms	C. 4	30 p.f. air trimmer
R. 5	25 k. ohms	C. 5	150 p.f. mica
R. 6	5 k. ohms	C. 6	100 p.f. mica
R. 7	350 ohms	C. 7	0·01 μF paper
R. 8	1 megohm	C. 8	0·01 μF paper
R. 9	200 k. ohms	C. 9	0·05 μF paper
R.10	20 k. ohms	C.10	0·02 μF paper
R.11	500 k. ohms	C.11	0·02 μF paper
R.12	1 megohm volume control	C.12	0·03 μF paper
		C.13	0·01 μF paper
R.13	20 k. ohms	C.14	0·01 μF paper
R.14	5 megohms	C.15	250 p.f. mica
R.15	50 k. ohms	C.16	0·01 μF paper
R.16	100 k. ohms	C.17	0·04 μF paper
R.17	200 k. ohms	C.18	8μF, 50 W.V. dry electrolytic
R.18	2 k. ohms		
R.19	50 k. ohms	C.19	0·01 μF paper
R.20	100 k. ohms	C.20	8μF, 50 W.V. dry electrolytic
R.21	500 ohms		
		C.21	0·01 μF paper
		C.22	8μF, 50 W.V. dry electrolytic
		C.23	0·01 μF paper

Battery Power Pack Section

T. 1	Triller, Synchronous, 6-volt operation, 6-pin base	
C.24	0·2 μF paper	
C.25	0·01 μF mica moulded	
C.26	0·05 μF paper	
C.27	0·01 μF paper	
C.28	40 μF, 450, W.V. dry electrolytic	
C.29	40 μF, 450, W.V. dry electrolytic	
R.22	2500 ohms	No load primary current 0·35 amp. for 28 ma. secondary current at 160 volts primary drawn 1·15 amp.
R.23	200 ohms	
RFC	R.F. choke 20 m.H.	
T. 2	Vibrator transformer 1 : 35 ratio	

A.C. Power Pack Section

T. 3	Power transformer	230-0-230, 50 ma.
R.24	1000 ohms, 5 watts	
C.30	16 μF 450 W.V. dry electrolytic	
C.31	16 μF 450 W.V. dry electrolytic	

from either mains or battery power pack can be directly plugged into it. As the set has to be worked either off 220-volt main or a 6-volt battery, the valves are so chosen that their heater currents as well as the H.T. current drain is low to keep down the battery current within limit. The working H.T. voltage in the set is stabilized at 160 volts for both mains and battery operation. At the same time, the maximum undistorted power output of the set, while working on battery, does not fall below 1·25 watts. The sensitivity of the set is also maintained in the order of 20 μv. for 500 m. watts output.

Design of the Circuits

First Detector Stage — As the set is mainly required for spot frequency reception, that is to tune to one station only at a time, it is provided only with a tuning arrangement to cover a range of about 100 kc./s. on either side of the chosen spot frequency.

The aerial input is fed to the tuned grid circuit of the frequency changer valve 6D8. As already explained, this particular valve is selected for its low heater drain of 0·15 amp. The tuned grid circuit is conventional rejector type circuit, the parallel capacity being in this case a mica trimmer condenser (0-30 p.f.) and with another low-loss fixed mica condenser (about 150 p.f.) in parallel. The aerial coil is of high impedance section wound type (1·25 mh). The coil former is made of 1″ outside diam., low-loss ebonite tube with $\frac{1}{8}''$ wall provided with suitable brackets for fixing it to the chassis. The grid circuit is pre-tuned by means of the trimmer condenser for the required signal frequency. The Q factor of this air core coil is of the order of 100 ensuring fair stage gain and sufficient selectivity.

The coil tried was at first of the plug-in type with pin connections at the base and there were 2 separate coils, one for medium and the other for short-wave bands. Later it was found that the plug-in coils develop high resistance contacts at the base pins and can be advantageously replaced by coils mounted on the chassis permanently and connections made through a simple band switch.

Oscillator Circuit — The oscillator is of the conventional type with tuned grid circuit and a feed-back coil in the anode. This is an air core coil, wound on a 1″ ebonite former as in the case of the aerial coil and it is shielded by a cylindrical copper shield of $2\frac{1}{4}''$ diam. made of 12 SWG sheet. The parallel capacity in the circuit is provided by means of a fixed mica dielectric condenser in parallel with an air dielectric variable condenser of 5-50 p.f. range. This variable condenser is robust, with an extension shaft for manual tuning. This condenser is specially provided to give a range of 100 kc./s. variation in the oscillator frequency. This enables tuning of the set within a range of 100 kc./s., about the spot frequency for which the aerial coil is tuned. It is seen, therefore, that this control of the oscillator frequency which can be manipulated by the operator enables him to retune the receiver properly, compensating for any tuning drift in the set. At the same time, this device has simplified the tracking problem in the set as the band width is limited to 100 kc./s.

unlike in the case of a broadcast receiver where the band width is about 1,000 kc./s. on broadcast band and 6 to 7 mc./s. on medium short band. On account of this, the gang condenser in this community set design could be eliminated. This simplification in the circuit enables the alignment of the aerial trimmer at any time to be done directly while receiving the signal from the station for which the set is pre-tuned. The oscillator grid condenser, on being tuned for maximum output, automatically tracks the oscillator frequency in relation to the signal frequency. Therefore, the usual multi-band signal generator is not necessary for the alignment of the set while servicing it. A compact oscillator generating the intermediate frequency will only be necessary for the occasional alignment of the I.F. circuits of the set. This simplification in the design has the added advantage of covering two spot frequencies in broadcast band and a short band width of 100 kc./s. in each of 31, 41, 49 and 60 metre bands with the help of only two coils with fixed capacitors connected through a band switch. This enables easier servicing and checking of the circuits when necessary, than in the case of the usual commercial receivers with 5 to 8 bands including spread band circuits, etc. Most of these extra circuits in commercial sets are superfluous for this purpose, and involve complicated servicing work.

Intermediate Frequency Stages — The intermediate frequency stage in the set is of the conventional design using 6SS7 (0·15 amp. heater) and the frequency is 456 kc./s. The transformers used are Meissner standard type Nos. 16-5712 and 16-5714. They are air-cored types with ceramic base and mica dielectric trimmers. The Q factor of the coils in these transformers is between 85 and 110. The circuit impedance of the first I.F. transformer is 0·4 megohm corresponding to an r/L ratio of 30,000. The primary impedance of second I.F. transformer is also 0·4 megohm corresponding to an r/L ratio of 26,000. These figures compare favourably with those that are met with in the usual commercial broadcast receivers (TABLE III).

Second Detector Stage — The second detector is 6T7 valve (0·15 amp. heater) in which the two diodes are connected together to one side of the secondary of the second I.F. transformer and circuit being complete to cathode and earth through a load resistance of 500,000 ohms. The audio voltage is fed through a volume control to the grid of the triode section of 6T7. Simple AVC voltage is fed to the grids of the previous I.F. amplifier and frequency changer valves.

The triode audio amplifier of 6T7 is designed with a high grid leak resistance (5 megohms) to reduce the shunting effect on the detector load. The anode of the audio amplifier is sufficiently decoupled to increase the stability of the stages. The voltage gain in the stage is of the order of 35.

Audio Driver Stage — A simple triode voltage amplifier stage using 6L5 valve (0·15 amp. heater) is incorporated to give sufficient drive to the output stage. This is considered advantageous in keeping down voltage gain to be obtained in the previous stage, thus reducing the tendency for instability. This stage also enabled the overall sensitivity of the set to be increased without sacrificing stability. The increase in the battery current owing to this stage is comparatively small (0·15 amp. heater current ; about 5 ma. plate current) and the total battery current does not exceed the specified amount of 2·9 amp. for the set. The voltage gain of this stage is 12.

Output Power Stage — In the output power stage, a beam power output valve 6V6 (0·45 amp. heater) is used for the following consideration. It is worked as a class A power amplifier with cathode resistance for generating the grid bias. This valve is rated at 2 watts with 180 volts anode voltage with a maximum grid voltage swing of 8·5. In order to economize in the H.T. anode current

TABLE III — COMPARATIVE DATA OF MEISSNER & QB9 I.F. TRANSFORMERS

TRANSFORMER	FREQUENCY kc./s.	PRIMARY					SECONDARY			GRID COUPLING FACTOR	EXISTING COUPLING FACTOR	MUTUAL INDUCTANCE m.H.
		Inductance (L) m.H.	Tuning capacity p.f.	Q	r/L	L/cr ohms	Inductance (L) m.H.	Tuning capacity p.f.	Q			
QB9 1st I.F.	455	1·05	117	86	33,240	258,000	1·09	111·4	82	0·012	0·019	0·021
Meissner 1st I.F.	456	1·42	84·3	96	30,100	394,000	1·392	86	85	0·011	0·012	0·017
QB9 2nd I.F.	455	1·85	66	85	33,620	449,400	1·95	62·2	82	0·012	0·02	0·0392
Meissner 2nd I.F.	456	1·247	96	112	25,800	403,500	1·174	94	95	0·0026	0·002	0·273

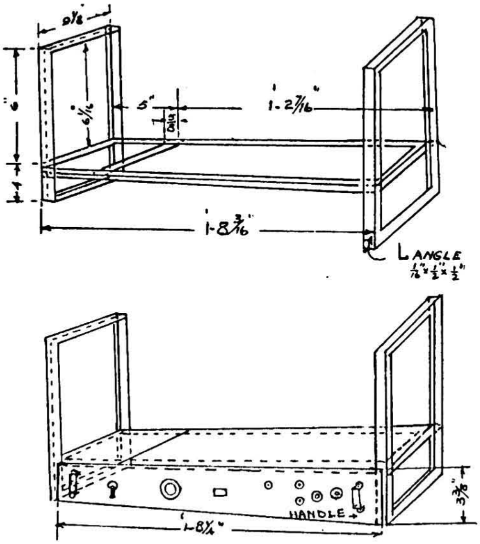

FIG. 2 — CHASSIS FRAME.

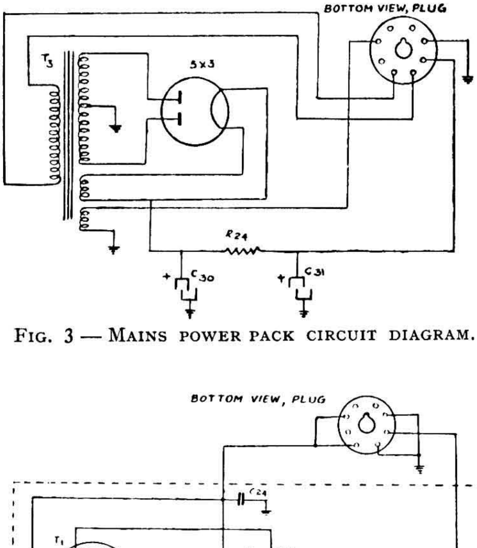

FIG. 3 — MAINS POWER PACK CIRCUIT DIAGRAM.

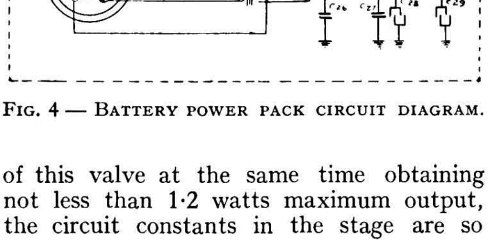

FIG. 4 — BATTERY POWER PACK CIRCUIT DIAGRAM.

of this valve at the same time obtaining not less than 1·2 watts maximum output, the circuit constants in the stage are so adjusted as to give the following operating conditions :

(i) Anode voltage, 150
(ii) Anode current, 25 ma.
(iii) Cathode voltage, 9
(iv) AC grid volts (for 1·2 watts output), 12 volts.

The question of economy in the cost of the set has also been taken into consideration in deciding upon this beam power class A output stage in preference to a push-pull output stage. A triode or pentode push-pull output stage will require additional components and circuit in the shape of either a driver transformer or a phase inverter circuit. A suitable valve for the push-pull output stage will be 6Y7 driven by a 6W7 with the help of a transformer. It may have an advantage of giving maximum output with less percentage distortion than a class A beam power tetrode used in this design. But on practical testing of the performance of this set, it is seen that the 6V6 class A output stage has given an equal performance as in the case of 6Y7 output stage, at the same time economizing in the cost of production. The test data of the output given by this set and the current drain compared to those of a similar commercial set using 6SA7, 6SS7, 6T7, 6W7 and 6Y7 are given in Table IV.

Power Pack Assembly — The power pack is assembled separately so that it can be fitted in the chassis frame of the set adjacent to the main receiver portion as shown in Fig. 2. The overall dimensions of the base plate of the battery and mains power pack are kept the same so that they are interchangeable. In Fig. 3 the circuit of the mains power pack is shown. Each power pack is self-contained with its filter circuits. The main switch fitted in the control panel is

TABLE IV — TEST DATA FOR R.C.A. QB9 SET ; BATTERY OPERATED
(*with permanent magnet speaker*)

Test frequency — 760 kc./s. Modulated 30% with 400 cycles A.F.

Sensitivity—

Input for 500 m.w. output	55 μv.
High tension voltage supply to the bands	105 volts
Maximum output at 760 kc./s.	1·2 watts
L.T. current drain off 6-volt battery	2·2 amp.

Test Data for Community Receiver —

Sensitivity ; Test Frequency	1290 kc./s. modulated 30% with 400 cycles A.F.
Input for 500 milliwatts output	20 μv.
High tension voltage supply to the anodes	160 volts
Maximum output at 1290 kc./s.	1·25 watts
L.T. current drain off 6-volt battery	2·1 amp.

wired to the input socket fitted in the receiver chassis. From the circuits of these power packs given in Fig. 1, it may be seen that the mains or battery lead, H.T. lead and valve heater supply lead are brought out to the various pins of an 8-pin plug. The connections to these plugs are made through a short multi-core shielded cable. The connections to the 8-pin socket fitted to the receiver chassis are so wired that the plug of either the mains or battery power packs can be plugged-in direct so that the set is ready for instantaneous operation.

Chassis & Other Mechanical Parts — The simplification in the design resulting in the elimination of dial, dial drive gang condenser, etc., enables the use of a frame type of chassis for the whole set. The power pack is mounted in a separate sheet from the main receiver and can be screwed down to the chassis frame, a sketch of which is given in Fig. 2. The battery power pack (FIG. 4) and the mains power pack are thus interchangeable. The shape of the frame-chassis is such that the set can be rested in any position for quick servicing without damaging the valves or other components. It is robust and at the same time light, being built of aluminium L angle. It has been made slightly larger than a conventional chassis of usual 5-valve commercial receiver to provide room for clear and neat wiring and for facilitating quick and easy servicing. The loud speaker is not mounted in the receiver frame but is housed in a separate cabinet for installation away from the set. The loud speaker is provided with a 15′ twin core extension cable ending in a 2-pin non-reversible plug, and is plugged into a corresponding socket provided in the control panel of the receiver to connect it to the output of the set. The controls for the set and the aerial-earth terminals are also fitted in the front panel for easy accessibility. The receiver chassis frame goes into a simple and strong wooden cabinet which is made insect and dust-proof. The receiver cabinet has been constructed with the front door in two halves, so that by raising the lower half, the controls alone are accessible to the operator. The top half is closed and sealed until required to be opened for taking out the set for servicing, only when necessary.

This design of the simple community set meets the following requirements :

1. The general design of the circuits, layout of the components and the mechanical parts are simple such that they can be built in any moderately equipped workshop and laboratory.
2. No special skilled labour or special plant and machinery will be required to produce these sets even in bulk.
3. The design of the set is directly suited to community listening purposes such that no further modifications are necessary.
4. The cost of maintenance will be considerably lower due to simplification in the stocking of spare equipment required for servicing and ease of servicing.

Acknowledgements

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Manufacture of Sponge Iron — Literature Report No. 2

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SPONGE iron is the product of direct reduction of iron ore below the fusion point of iron. Considerable work has been carried out in the United States (mainly by the *U.S. Bureau of Mines*), Sweden, Germany and France, stress being laid mainly on the following important aspects :

(1) Sponge iron as a substitute for scrap in the production of steel, especially in electric smelting furnace for the manufacture of high-quality steel[1-4].

(2) Utilization of non-coking type of coal and other gaseous fuels for the reduction of iron ores. This problem is of special interest to India ; she has ample high-grade iron-ore deposits but lacks coking coal.

(3) Use of sponge iron in chemical and metallurgical operations requiring finely divided metallic iron of high porosity. Sponge iron has many advantages as a precipitant. Precipitation of metals from solutions as used in copper metallurgy has been advocated for many years.

Methods of Manufacture

1. *Methods Developed by the United States Bureau of Mines* — The experimental work of the *Bureau* deals mainly with the production of sponge iron by heating iron ores at low temperature using solid carbon as the reducing agent.

The small-scale experiments are carried out in muffle furnaces. The reduction temperature for iron ore without fusion and using solid carbon as reducing agent is about 875° to 1,025°C., the most desirable temperature being about 950°C. The heat conductivity of finely divided iron-coal mixture is low. Also the reduction reaction in such changes are endothermic. These conditions make rapid heating of the charge almost the first essential factor in the design of a high output furnace. The muffle furnace is less practical than the direct-heated type for the reduction of iron ore on a large scale.

The large-scale experiments are carried out in continuous rotary gas-fired kilns. The kilns are built with sections of different diameters. The charge of ground iron ore and coal enters the upper end of the kiln and passes rapidly in a thin layer through the preheating section. During the preheating period of 15 to 20 min., the volatile matter of coal and the moisture in the charge are driven off and the charge enters the enlarged section at approximately 900°C. with 3 to 5 per cent iron reduced. In this section the charge is in a layer of 13″ to 15″ deep. Maintained at a nearly constant temperature of 900° to 1,000°C., the charge remains in this section for about 1 hr. during which period the reduction of the iron oxide is complete. The reduced product is discharged continuously through a pipe into a cooling chamber below the furnace. The cooled product contains a large quantity of excess carbon and coal ash which is removed by means of a magnetic separator. The concentrate usually contains less than 1 per cent carbon and 65 to 90 per cent iron according to the grade of ore treated. The experiments indicate that rotary kilns give consistently good results[5-13].

The *Bureau of Mines* have also carried out experiments, using natural gas as a reducing agent[14,15], with ore containing about 50 per cent iron, 10 per cent silica and 3 per cent alumina. A large part of the investigation is devoted to the development of a suitable reducing gas from natural gas. The following conclusions have been reached : (i) In a 25,000 lb. charge, 80 per cent of the iron ore is reduced to metal in 13 hr. ; (ii) a higher degree of reduction is not obtained because the amounts of carbon dioxide and water vapour present retard the rate of reaction, and because the endothermic nature of the first reaction reduces the temperature below the optimum ; (iii) sintered ore is not reduced as rapidly as unsintered ore ; (iv) discharge of the sponge iron is difficult because of sticking ; and

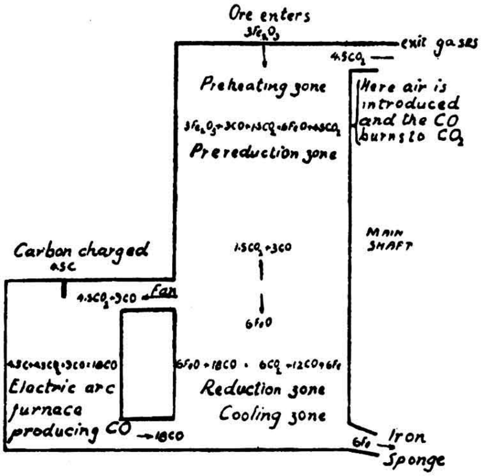

FIG. 1 — DIAGRAMMATIC REPRESENTATION OF WIBERG PROCESS.

(v) the sponge, containing 80 per cent iron in the reduced state, is easily melted in an electric furnace.

Producer gas was successfully used in a multiple-hearth furnace[16]. *Republic Steel Corporation* has built a multiple-hearth furnace using coke-oven gas[17]. Hydrogen gas was tried in what is called bubble-hearth process[18] but the process was found unworkable.

2. *Swedish Processes : Wiberg Process* — The theoretical basis of a suitable process for the manufacture of sponge iron was laid down in 1918 by Prof. Wiberg. In principle (FIG. 1) the method is as follows : The ore, either in sintered form, or in the form of uncrushed ore of high iron content, is charged into the furnace, which works on the counter-current principle.

In the lower part of the shaft, carbon monoxide is passed in at a temperature of 900° to 1,000°C. The gases passing up the shaft reduce the ore to iron, and the carbon dioxide content of the gas rises to 25 to 30 per cent with respect to iron, an equilibrium between ferrous oxide and carbon dioxide being set up. Three-fourths of the gas is sucked out by fans into an electric arc gas producer, in which it is reduced to carbon monoxide. The reaction $C+CO_2 \rightleftarrows 2CO$ being endothermic, heat must be supplied, and this is done by electrical energy. The remaining gas (one quarter) passes further up the shaft where an air intake causes it to be burnt, thus heating up the ore to 900° to 1,000°C.

A furnace capable of producing 10,000 metric tons per year was erected in Soderfors by the *Stora Kopparberz Berzslag Company* in 1932. Fig. 2 shows a sketch of the furnace. The ore or sinter is laid in at (1). The shaft is lined with fire-bricks and has a wall thickness of about 900 mm. This is surrounded by a sheet-iron cover. The gas is laid in at (2) through a ring conduit, and then through a number of narrow twyers in the shaft (3). The gases are led out through 4 exits (4). The air for the combustion in the upper part of the shaft enters through 12 twyers and the used gases finally leave by the chimney (6). The chimneys have valves to regulate the pressure in the shaft. The lowest part of the shaft is surrounded by circulating water (7). The sponge leaves the outlet (8) into the container (9). The gases from (4) pass through the circulating fan (10) which, revolving at 2,950 r.p.m., could deal with 110 cu. m. per min. at 360 mm. pressure and 800°C. The temperature and pressure conditions in the furnace installation is illustrated in Fig. 3.

The gas producer (FIG. 4) is lined in the same way as the shaft, the outer cover being of 18-8 Cr-Ni steel. The producer has three pairs of Soderberg electrode, 300 mm. diam., passing down through a collar, each electrode being coupled to its own single-phase transformer. The transformers are of 400 kW. at 55 to 90 volts. The carbon is fed in through (12) from the carbon container (13) of 14 cu. m. capacity. The dust and ash have to be shovelled out at the bottom.

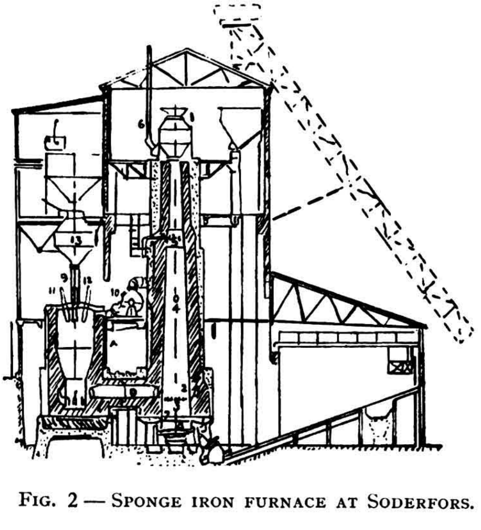

FIG. 2 — SPONGE IRON FURNACE AT SODERFORS.

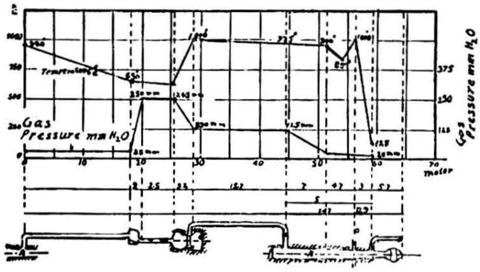

FIG. 3 — TEMPERATURE AND PRESSURE CONDITIONS IN A SPONGE IRON INSTALLATION.

With a given size of the furnace, the output is dependent on (a) temperature and composition of in-going gases ; (b) speed of gas circulation ; and (c) quality of the ore. The correct composition of the gas is easily achieved and its temperature can be closely defined within practical limits. Increase in speed of circulation increases output. Above a certain limit, however, the amount of carbon dioxide in the gases entering the producer is reduced, since the ore is not sufficiently rapidly reduced for the carbon dioxide to be increased to the required theoretical value of 30 per cent and the electricity and charcoal consumption per ton of reduced iron ore automatically increases. The suitability of the ore can be increased by pretreatment[19-23].

According to Ramoeyer[24], the Wiberg process is the only one in successful operation at present, but it has three disadvantages. It cannot use the fine concentrates just as they come from the magnetic separator ; secondly, the operation has to be carried out at such a high temperature that sticking difficulties are encountered ; and thirdly, regeneration of carbon dioxide to carbon monoxide is not very satisfactory. The use of hydrogen under pressure is advocated as a reducing agent. The reduction of magnetite to iron with hydrogen should not be carried out above 1,100°F., otherwise ferrous oxide will be formed which is difficult to reduce.

These considerations have led to the development of the Brassert process, in which coke-oven gas is used as the source of hydrogen. The coke-oven gas is desulphurized, preheated to 1,200° to 1,300°F. and passed into the bottom of a vertical Herreshof furnace into which ore is fed at the top. The finely divided ore passes down over a series of disc plates to the slotted grate at the bottom through which the hydrogen passes upwards. By bubbling the gas through the fine ore, the latter is made to flow along the channels and from one plate to another as if it were a liquid. To prevent reoxidation, the hot reduced fines are passed directly to a briquetting press designed to make corrugated slabs of compressed sponge iron.

3. *German Processes : The Krupp & Krupp-Renn Process* — The direct reduction of ore is carried out in a rotary furnace ; the iron is obtained in the form of compact lumps containing but little carbon. On leaving the furnace the lumps are embedded in a pasty slag, from which, after cooling, they are separated by a crushing process. All sorts of low-grade fuels, such as coke screenings, fine coal and semi-coke residues, can be used in the furnace ; the recovery of iron in the lumps amounts to 90 to 96 per cent, depending on the ore and the mode of operation.

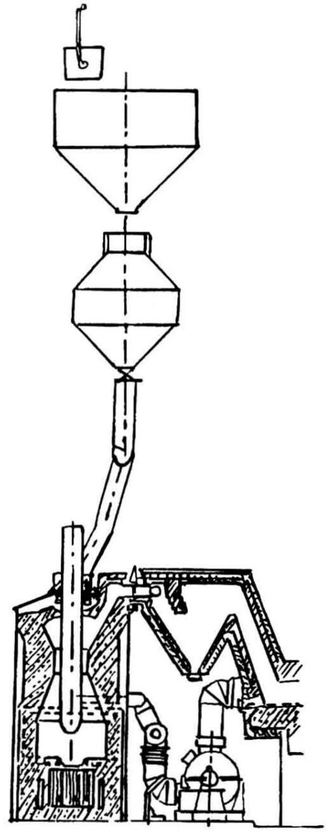

FIG. 4 — SINGLE-PHASE GAS PRODUCER.

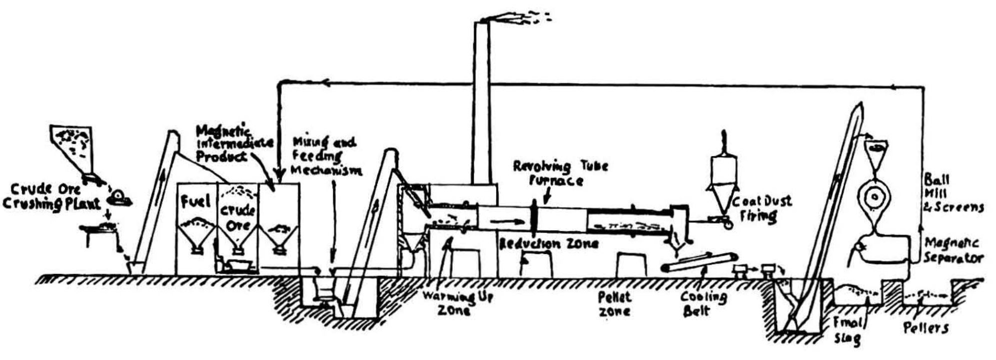

FIG. 5 — OPERATIONAL SEQUENCE IN THE IRON PROCESS.

Most of the phosphorus from the ore passes into iron, but manganese goes largely into the slag. The sulphur content of the lumps depends on the basicity of the slags. When treating rich ores, it would appear economical to aim at the production of low-sulphur lumps by making suitable additions to the charge, and then to use lumps directly for steel-making. With poor ores, and particularly those high in silica, it is better to make no additions but to use the process simply as a preparatory treatment and then charge the lumps into a blast furnace[25-27].

The operational sequence of the Renn process is set out schematically in Fig. 5. As opposed to the well-known processes for the production of iron by means of the revolving-furnace process in which, as a rule, the iron is produced as a sponge or liquid pig iron, the essential future of the Krupp-Renn process lies in the special function of the " pallet-zone ".

Fig. 6 shows the bottom ends of a Walz furnace (used for the distillation of zinc and lead) and a Renn furnace in sectional elevation.

The Krupp-Renn process definitely makes smaller demands as regards the composition of the slag than does the normal smelting process. The process only requires a slag which is semi-soft at 1,200° to 1,300°C.

The physical properties of the fuel, such as screen size and resistance under pressure, play no part as the fuel is finely ground and mixed with the ore. Fuel with a high ash content causes no difficulties and is preferable to low ash material if it can be obtained at lower price.

The pellets obtained by the Krupp-Renn process are practically slag free and vary in size from 1·5 to 50 mm.

There are three principal applications of the Renn process (FIG. 7):

(i) The direct production of steel ;
(ii) the enrichment of a poor quality ore, especially one with a high silica content ; and
(iii) the production of non-ferrous metals, for example, nickel, cobalt, copper, silver, gold and platinum metals.

The direct production of steel without the use of the blast furnace is particularly suitable in cases where a supply of ore is available, but suitable coke for blast-furnace smelting is not too plentiful.

Briquetted iron ore is used in another German process[28] in which carbon monoxide, hydrogen or other suitable gas brings about the reduction in a shaft furnace. The ore reaches its highest temperature about half way down to the furnace, and is cooled down almost to the atmospheric temperature by

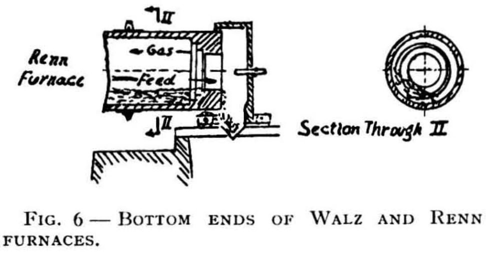

FIG. 6 — BOTTOM ENDS OF WALZ AND RENN FURNACES.

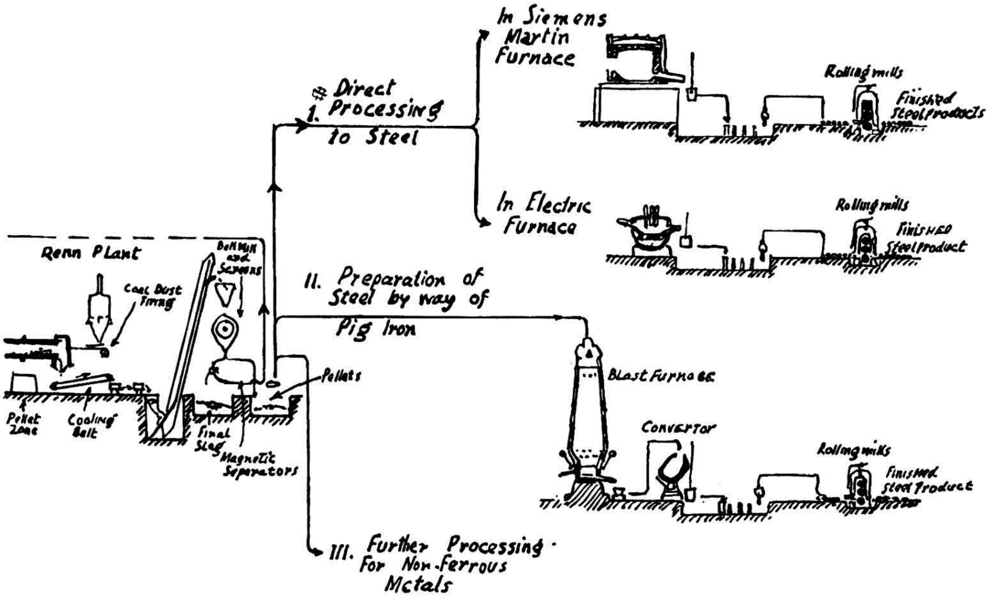

FIG. 7 — PRINCIPAL APPLICATIONS OF RENN PROCESS.

a cold blast of reducing gas admitted at the base of the furnace, wherefrom the sponge iron is extracted.

Norsk-Stahl process[29] uses a mixture of carbon monoxide and hydrogen as a reducing agent. The equipment consists of three vertical ovens for preheating, reducing and cooling the charge and apparatus for regenerating the spent gases.

Other important processes are : Hornsey process worked at Chesterfield[30] and Lorain and Ohi[15] in which rotary kilns or retorts are employed. The Smith process is carried out in vertical ovens or retorts, similar to coke ovens in design. The crushed ore is mixed with carbonaceous material and charged into the oven where it is heated and cooled by means of horizontal flues. It is preheated in the upper part of the oven by waste gases which leave the stack at about 400°F. The charge then enters the reduction zone where temperature ranges from about 1,600° to 2,000°F. The charge is subsequently cooled by the in-coming air for combustion in the heating flues, and discharged below 250°F.[31].

Economic Aspects[32]

The economic success of a direct process depends on : (i) whether it can produce a finished product which requires no remelting before marketing at a fair price in competition with other metals ; (ii) whether it can produce a material at as low a price as steel scrap ; and (iii) whether the manufacturers can obtain for sponge iron a price better than that of steel scrap. The direct process has a few disadvantages compared with blast furnace. It cannot easily flux off sulphur (Bushi and Schout have investigated the production of low-sulphur sponge iron[11,33]). Another disadvantage is that the material is readily oxidized and is difficult to handle during melting operation.

Summary

This report deals with the survey of the existing literature on the manufacture of sponge iron.

Methods of manufacture developed by the *United States Bureau of Mines* are enumerated, their experiments on continuous rotary gas-fired kilns are dealt with in detail.

The Swedish Wiberg process is described at great length. Given cheap electric power supply, this process is the most successful one.

The German Krupp-Renn process has been worked very successfully in Germany. In this process fuel with high ash content causes no difficulties and is preferable to low ash material. Work carried out in other countries is also referred.

The economic aspects of the direct process are discussed.

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Obituaries

SIR ARDESHIR RUSTOMJI DALAL

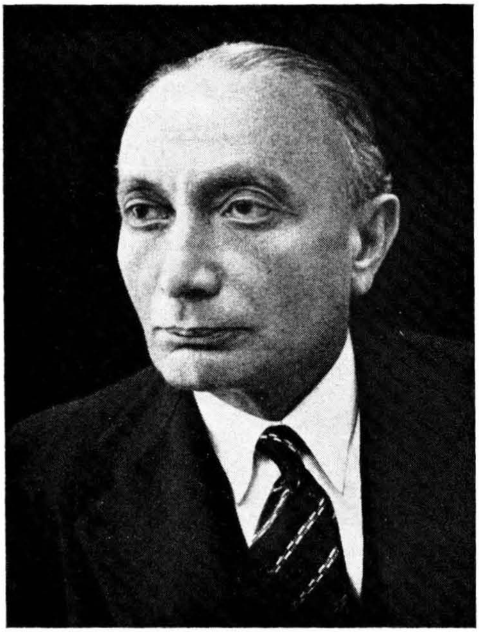
(April 1884–October 1949)

WE REGRET TO RECORD THE DEATH, ON *October* 8, 1949, of Sir Ardeshir Rustomji Dalal at Bombay.

Sir Ardeshir was educated at the Elphinstone College, Bombay, and later at the Cambridge University. He qualified himself for the Indian Civil Service and served the Government of Bombay in various capacities and was for sometime Secretary, Department of Education, Health and Lands, Government of India. In 1943 he was appointed a Member of the Viceroy's Executive Council in charge of Planning and Development, in which capacity he served till the end of 1945. During the latter period he was President of the Governing Body of the *Council of Scientific & Industrial Research.* More recently, as the Chairman of the Reviewing Committee set up to review the work of the Council, he was responsible for formulating far-reaching recommendations on the development and utilization of scientific research in India.

Sir Ardeshir was one of the principal signatories to the well-known 15-year Bombay Plan for the industrial development of India, a plan which is regarded as a landmark in the history of post-war economic planning.

After retiring from the Indian Civil Service, he joined the *Tatas* as Director and partner, and was one of the leading industrial magnates of the country. Dignified and broadminded, he represented in the larger national life of the country the great traditions associated with India's leading Parsis.



SIR A. R. DALAL

MR. C. C. GHOSH

WE REGRET TO RECORD THE DEATH, ON *August* 20, of Mr. C. C. Ghosh, retired Deputy Director of Sericulture, Bengal, at the age of sixty-five.

Born of a middle class family, Mr. Ghosh developed a keen interest in entomology and from a technical assistant in the Pusa Agricultural Research Institute, he rose to the post of the Entomologist to the Government of Burma, in which capacity he made valuable contributions to the organization of sericulture industry in Burma. His contributions to the development of sericulture in Bengal, during nearly a decade prior to his retirement from service, are well known.

Among his many publications, mention may be made of *Insect Pests of Burma*, which is accepted as a standard work in economic entomology, the *Silk Industry of Japan* and *Silk Production and Weaving in India.* He was Chairman of the Silk Research Committee of the *Council of Scientific & Industrial Research.*

REVIEWS

Forest Pathology, by John Shaw Boyce (McGraw-Hill Book Company Inc., New York), 1948, Second Edition. Price $6.

THE BOOK IS BASED ON THE RESEARCHES on forest and plant pathology contributed by about 300 workers all over the world covering a period of three quarters of a century. The first edition was published in 1938 which underwent 4 impressions in a comparatively short period. The first book on this subject was published in 1931 under the title *An Outline, of Forest Pathology* by Dr. Earnest E. Hubert. Dealing with the same subject is a third book, *Pathology in Forest Practice*, by Professor Dow Vawter Baxter published in 1943.

Forest pathology, which principally deals with the diseases of woody plants of forests and also takes into account the decay of timber and deterioration of forest products, is a daughter science of plant pathology and applied mycology. But the extension of its scope to forest products has made it more attractive and important than the nucleus from which it developed. Like every applied science, the field of application of forest pathology is ever expanding. This is shown by the growing demands of a treatise like this and the publication in the last 12 to 15 years of 3 well-written books and a large number of subsidiary texts on decay of timber and forest products, monographs on tree rusts and other fungi connected with tree diseases.

The fact that the first edition of Professor Boyce's book underwent 4 impressions during the war time is significant of its popularity and of the demand by increasing numbers of workers in this field for information on more recent developments. A large amount of new information has accumulated during the past 10 years, mainly on the exploitation of timber resources and intensive utilization of forest products for purposes of war and this had to be incorporated.

The book is compiled in 21 chapters, 2 appendices (containing a description of fungicides and a list of common plant names with their scientific equivalents), and an index. The book can be broadly considered under 7 parts, namely general mycological text, non-infectious diseases, seedling diseases, pathology and tree diseases, decay of wood and wood products, principles of disease control, and appendices and index.

The first 3 chapters consist of introduction, discussion on the causation of fungal diseases and a simple taxonomic description of different groups of fungi. Chapter IV on non-infectious diseases discusses pathological symptoms produced by high temperatures (sun scald), low temperature (frost injury, frost ring formation, frost canker, frost scars, etc.), water deficiency (drought, heat cracks, winter drying, etc.), water excess, nutritional defects, injury due to industrial process (smoke and smelter fumes injury), salt spray, mechanical injury (fire scars, snow storms, hailstones and lightening injury, etc.), and provides a complete bibliography to the recent works of Swingle (1944), Hepting (1945), Jackson (1945), Hansborough (1947), Hawboldt (1947) and Pamerleau (1944). The seedling diseases have been considered under 2 categories, namely non-infectious and fungus diseases. Under non-infectious diseases, the above factors have been considered from the point of view of protection. Under fungus diseases, damping off, root rot, snow blight, moulding, smoothening, spots, etc., due to the attack of fungi of lower orders (Phycomycetes and Fungi Imperfecti) and Ascomycetes and a few Basidiomycetes have been discussed. For controls of seedling and nursery diseases, selection of disease-free seeds, proper planning of nursery and selection of nursery sites have been given the utmost importance. Although the list of causal agencies is not complete and reference has been made to pathogens known in the United States of America, the items of control practically cover the whole range of such diseases. The root disease due to the shoe-string fungus (*Armillaria mellea* (Vahl.) Quel.) has received adequate recognition (chapter VI). Contributions on the pathology of root and buttrot fungus (*Fomes annosus* (Fr.) Cke.) from the United States of America and Europe are mostly of recent years (1930 to

1946) ; the readily accepted theory of root wounds and frost injury in the butt region has been discussed with a few remarks on control methods. A short description of root diseases due to other organisms of the family Basidiomycetes, Ascomycetes, Phycomycetes, Fungi Imperfecti and viruses (Phloem Necrosis of American elms — Swingle 1942, Tucker 1945) have been given. A short account of mycorrhiza has also been included.

The foliage diseases have been described in 2 parts : hardwoods (chapter VII) and conifers (chapter VIII). The former include leaf spots, tar spots, twig blights, powdery mildews, sooty moulds, rusts, blisters, flower and fruit rot, deformations, scab, and canker of tender shoots caused by various pathogenic fungi including rusts, Ascomycetes, Fungi Imperfecti, etc., a short but comprehensive account being given.

Under the latter are included distribution, pathology and biology of leaf blights and needle casts due to various ascomycetous fungi such as cedar-leaf blight due to *Keithia thujina* Durand, needle cast of Doughlas fir due to *Rhabdocline pseudotsuga* Syd., needle cast of balsam fir due to *Hypodermella nervata* Darkar, needle cast of pines due to *Lophodermium piniastri* (Schrad) Chev. *L. juniperinum* (Fr.) de Not., *Hypodermella concolor* (Dearn.) Darkar, of jack and lodgepole pines have been given, besides several others, under miscellaneous foliage diseases. Under needle rusts distribution, life-cycle, pathogenecity of cedar and juniper needle rust (15 species of *Gymnosporangium*) have been considered in detail because damage to the pomaceous hosts is often serious in the west coast, north, central and Lake Region States of U.S.A., and in the provinces in the north, central and western Canada and valuable orchard trees are attacked by " cedar apple " rusts. Control measures include spraying the orchards and isolation of alternate hosts. Besides *Gymnosporangium*, about 70 species of needle rusts, most of them being heteroecious species, have been described hostwise such as Pine Needle Rusts (*Melampsora*, *Melampsorrella*, *Uredinopsis* and *Milesia*), Hemlock Needle Rusts (*Pucciniastrum*), and Larch Needle Rusts (*Melampsora*). Also included in the list are unrelated species referred to by *form genera Peridermium.*

The stem rusts of conifers which cause " witches'-brooms ", swellings, blisters and galls of stem and cause die-back or kill young trees outright are of greater economic importance and have received detailed treatment in two sections. Distribution, biology and pathology of 10 heteroecious stem rusts, except in case of Woodgate Gall Rust, the identity of which is not known, and of Western Gall Rust (*Cronartium harkensii* (Moore) Menecke) which is suspected to be identical with Eastern Gall Rust (*Cronartium cerebrum* Hedge. and Long = *Cronartium quercuum*), have been described in detail. Nothing could be more welcome to students of forest pathology than the complete and up-to-date supplementary information at the end of each chapter. One chapter has been allotted to the White Pine Blister Rust in the U.S.A. which is introduced from Europe. The amount of damage it caused to the pines, endemic as well as introduced, made the problem of paramount importance to the States for a quarter of a century. References have been made to the works of about one hundred workers in the States and in Canada, and a direct method of control has been successfully worked out. The White Pine Blister Rust, the Chestnut Blight Disease and Dutch Elm Disease have strengthened the hands of American phytopathologists in enforcing quarantine laws.

This is followed by 2 chapters on the stem diseases of conifers and hardwoods. In the former, canker due to Ascomycetes such as canker of pines due to *Tympanis species*, canker of larch due to *Dasycypha willkommii* (Hart.) Rehm and other *species* of *Dasycypha*, and due to Fungi Imperfecti such as *Macrophoma species*, *Phomopsis lokoya* Hahn have been described and symptoms and etiology detailed. These diseases attack trees growing under unfavourable conditions in the virgin forests of the United States; the descriptions of these parasites, therefore, have been given from the point of view of the American workers. None of these diseases, however, has been recorded in India.

In dealing with the stem diseases and cankers of hardwoods, which are of more serious consequence than those of conifers, the author has recorded many new diseases which are difficult to diagnose, and still more to control unless their etiology is known. Protective measures, which were merely suggestions, have now become definite with the increasing knowledge of the etiology and biology of the organisms. This chapter has considerable additional information than the previous edition.

Amongst the Fungi Imperfecti, Dothichiza and Septoria cankers of oak and chestnut, Sphaeropsis cankers of oak; and amongst the Ascomycetes Black Knot of cherry (*Dibotryon morbosum* (Schw.) T. & S.), Didymosphaeria canker of alder, Cytospora canker of willow, Neofabraea canker and Hypoxylon canker of poplar and Eutypella canker of maple have been described. Nectria canker (*Nectria galligena* Bres.) of yellow poplar and magnolias and Beech Bark Disease (*Cryptococcus fagi* Baer. followed by *Nectria cocina* var. *faginata*) with symptoms and etiology have been described in detail. Lastly, the Chestnut Blight or Endothia canker of chestnut has been described. This section has been presented in a somewhat more condensed form than in the previous edition. This fungus was formerly considered a dangerous parasite of forest *cum* horticultural plants of American importance. The Chestnut Blight Disease of America is now a wider problem for the geneticists and tree pathologists as it affects vast regions of Europe and Asia and the information regarding all aspects of the problem will be useful for workers in this field.

Under stem diseases, non-infectious and infectious galls have been discussed in 2 chapters. Short accounts of dieback of hardwoods due to *Nectria cinnabarina* (Tode) Fr., of pines caused by *Cenangium abietis* (Pers.) Rehm., of butternut by *Melanconis jugulandis* (E. & E.) Graves and of other miscellaneous hardwoods due to various species of Fungi Imperfecti have been given. A complete description of Dutch Elm Disease has been given. For protection of elms, sanitation against the bark beetles that spread the fungus, spraying of technical DDT dissolved in fuel oil or xylene with emulsifying agents has been recommended.

A concise description of American mistletoes with a key to the American species of *Arceuthobium* together with the host list has been given in chapter XV. Protection methods have been discussed in detail from the pathological and sylvicultural points of view for all types of forest including nursery stocks and virgin stands. At the end of the chapter some aspects of biological control have been discussed and a few enemies of mistletoes have been referred to.

Further elaboration of stem diseases due to various hymenomycetous fungi which are regarded as facultative parasites and for brevity termed " decay " opens chapter XVI which introduces timber pathology. The descriptions are concise but a full treatment has been given under each section which follows.

The importance of the isolation of " decay " and the examination of cultural characters for the diagnosis of " decay " (Campbell 1938; Davidson, Campbell and Vaughn 1942; Robak 1942) have been emphasized. Special media which accelerate the formation of sporophores (Badcock 1943), oxidation tests with gallic and tannic acids in malt agar media and the guiacum tests are useful recent additions.

Under the section " Control of Decay ", methods of scaling and estimating " decay " in trees and logs have been discussed and the methods to calculate the volume of rot and sound timber in stands and timber have been given. This is a very important pre-requisite for those engaged in the timber trade. The author has considered decay in various American softwoods such as western conifers which include Doughlas fir due to *Fomes pini*, *Fomes laricis*, *Polyporus schweinitzii* and *Fomes roseus*; sitka spruce due to *Fomes pini*; western hemlock due to *Echinodontium tinctorum*, *Fomes emosus*, *F. applanatum* and *F. pini*; white pine and lowland white fir due to *E. tinctorum*; red fir also due to *E. tinctorum*; western white pine due to *F. pini*; ponderrosa pine due to *Polyporus anceps* and incense cedar due to *Polyporus amarus*. Data from various Canadian and American laboratories and references to well-known workers such as Bier, Foster, Salisbury, Weir, Lewis, Hubert, Englarth and the author himself have been included. Importance of such timber like sitka spruce which was used for aircraft during the second World War, red fir, ponderrosa pine, etc., not mentioned in the earlier edition has been stressed and decay tests included. Formation of the top rot of hardwoods and its relation to the incidence of wounds and rotten branches and the data from the works of Davidson and Campbell (1943) on Black Cherry Rot on the estimation of cull defects are recent additions.

The largest number of contributions on stem and timber decay in recent years is mainly from the forest products laboratories of Germany, Sweden, England, Canada and U.S.A. and also from the laboratories under many industrial concerns processing timber and wood products and are referred to in chapter XVII. The author has given a complete but concise description of some of of the important American timber-destroying fungi. Besides short descriptions of about 80 species of hardwood and softwood decay, estimates of losses due to decay, both in use and storage, have been included. The section on dry rot has, however, been disposed of with short remarks on *Poria incrassata* (B. & C.) Curt. with a mention of *Merulius lacrymans* (Wulf.) Fr. The bibliography on the rots is complete.

The last 3 chapters are important from the point of view of timber industries, lumbering, utilization of forest products, etc., and subjects dealt with in sections under different categories are: Deterioration of Dead Timber (chapter XVIII); Deterioration of Forest Products — Decay (chapter XIX); and Deterioration of Forest Products — Sap-stain (Chapter XX). The author discusses the methods of salvage of killed trees, decayed and stained wood and wood products. The sound principles of lumber industry are based on secondary operations and salvage of the so-called waste material. Insect-killed, fire-killed, wind-thrown timber and slash, etc., add to the losses.

Biotic factors are responsible for tremendous losses of timber resources in tropical forests and the information given in these 3 chapters should prove very useful to our foresters, lumbermen and industrialists. Fire, insect and wood-rotting organisms are considered as natural enemies of the tropical forests. We have no adequate means to stop them, but proper logging of insect-killed and fungus-decayed material would certainly save a fortune provided we are able to work up the proper schedule of deterioration that sets in at different stages so as to ascertain how long such timber could be left in the coupé at the time of the year when transport to the mills is not practicable. Fire is the greatest curse of tropical and subtropical forests. We have no means to calculate the actual amount of loss to forests caused by this agency, but the estimated loss runs into many crores of rupees.

From the utilization point of view, the natural resistance to decay due to the by-products of plant metabolism such as tannin, volatile chemicals, colouring matters, toxic oils, resinous exudations (non-toxic but prevent penetration of wood-destroying fungi), etc., have been considered. Under the section of decay of pulp wood and pulp, methods of hygienic storage, proper piling and chemical treatment to protect material for pulp have been briefly discussed. The loss to our pulp material (bamboo in particular) is very high due to the fact that sap-stain is active in the monsoon season for 4 to 6 months. Storage is a difficult problem for the paper industries. To save the tremendous wastage, chemical protection of pulp material is essential.

Much work has been done by American workers on sap-stain since the publication of Professor Boyce's book on forest pathology in 1938. Stains on wood products, which were regarded as minor defects in timber, especially the blue stains of softwoods, are now considered to be one of the major handicaps to the marketing of finished wood products. Information available on the control of sap-stain (Schaffer and Lindgreen 1940, Hartley 1945, Schaffer 1946) has been given.

Under the section " Decay of Structures ", fungi causing decay of building and other permanent structures, and methods to prevent decay have been summarized. The section on wood preservation has been unusually abridged but a complete bibliography to the subject has been annexed.

The author discusses the fundamentals of forest-disease control in chapter XXI under 2 categories, namely the control of native diseases and the control of introduced ones. The basic principles involved being different in the two cases, the methods evolved have been different. With the native diseases, sound sylviculture, selection of sites, adjustment and judicial composition of the crop at the time of restocking, preference to mixed in place of pure stands in the second rotation, or second cut stands, selection of seed, acclimatization of the species when introduced from one region to another, and lastly, the breeding of forest trees to improve resistance have been considered and the merits and demerits discussed. The attempts to introduce exotics in the place of susceptible native species have been condemned. Introduced diseases are infinitely more destructive and require more expensive direct measures. The author has quoted glaring instances of half a dozen pests introduced into the United States during the last 50 years. The lack of judgement in the initial stage to estimate the harming capacity of potential parasites and delay in taking action against them have given them time to establish themselves on native American hosts. The slow method of investigating and cruising (estimating) the diseases at the time when they are first noticed have also been criticized. Finally, the principles governing control of introduced parasites have been summarized and the views of Ditwiller (1929) and Fracker (1937) have been put forward. Public opinion has to be roused in support of effective sanitation and destruction of diseased stock, and the need for passing legal enactments when persuasive methods fail, has been emphasized.

Although the methods described relate to the checking of pests, both endemic and introduced, of native American trees, the basic principles discussed are applicable to any country faced with similar situations. We have fortunately no record of an introduced tree disease that has developed into dangerous stage. The diseases of our evergreen and deciduous hardwoods and of the conifers in the Himalayas so far recorded are endemic, and endemic diseases never develop to dangerous proportions. Furthermore, against native parasites direct control is rarely necessary or justifiable. We have a large number of resistant hardwood species. As regards conifers, the Himalayan species, though few, are sufficient for our home requirements, and except as a trial on an experimental basis, there is little need to introduce exotics in India.

During the last world war, it was difficult to safeguard any country against the introduction of new diseases. We have records of *Trametes serials* and *Poria monticola* reaching our shores in a consignment of sitka spruce and Doughlas fir timber. There is every likelihood that other parasites have invaded the country through agricultural and forest products. Professor Boyce's remarks on quarantine regulations as an effective barrier against new pests are particularly apparent.

This book, which is the only one of its kind, will be invaluable to students and teachers of forestry, the forester, the silviculturist and the private owner of forest land. It will be equally handy to the lumberman and to everyone interested in the timber trade. The complete bibliography is invaluable to students, as no other complete and up-to-date reference list is available in this country.

K.B.

Elements of Electronics, G. Windred (Chapman & Hall Ltd., London), 1949, pp. 197. Price 15*s*. net.

MR. WINDRED'S BOOK ENTITLED " ELEMENTS of Electronics " covers, in an elementary fashion, a wide field of the science of electronics. The text is divided into 15 chapters and deals with the principles of electronic charge measurement, thermionic emission, conduction *in vacuo* and gases, photo-electric effects, thermionic and gas-filled valves and circuits, X-rays, cathode ray tube and circuits, television, sound film, electron optics, cyclotron and radio location. The book is entirely non-mathematical and as such should appeal to those interested in electronics but without sufficient mathematical knowledge. The author's method of exposition is lucid and this he has ably maintained right up to the end.

However, in attempting to provide simple explanations for all subjects, the author has been rather vague in several places. For example, in the explanation of the working of the split-anode type magnetron, the statement on page 35, " in the unelectrified or neutral state a piece of matter contains a definite number of negative electrons, such that two pieces of matter in this condition will experience no mutual electric forces " is confusing. So also is a statement made in the paragraph which follows. In Figs. 57 and 58, the output terminals shown have no bearing with the text and as such are misleading. The grid bias arrangement and its measurement by voltmeter " V " in Fig. 65 is also wrongly shown. There are a few printing mistakes in the book.

But these criticisms are of a minor character and do not in any way detract from the value of the book. The author deserves congratulations for writing so clearly on a subject of wide interest like electronics.

G. SANYAL

Fibres from Peanut Proteins

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THE successful use of soyabean proteins and milk casein as raw materials for the production of artificial textile fibres led to the investigation of the suitability of groundnut proteins as another possible raw material for the same purpose. Experimental work on the production of obtaining fibre from groundnut proteins was carried out in the laboratories of the *Council of Scientific & Industrial Research* with promising results.

The *Imperial Chemical Industries* have succeeded in producing textile fibre from groundnut proteins on a commercial scale. It is of interest to review the developments in this field in view of the work carried out by the author.

Groundnut, or peanut as it is known in America, is grown in India, China, Manchuria, Nigeria, French Sudan, Dutch East Indies, Spain and Argentina. India produces 180,000 tons of peanuts a year. China and Manchuria together produce about as much, and the United States rank third among the producing countries.

The process of fibre production from casein, soyabean and peanut proteins is similar to that employed in rayon manufacture, and can be considered under four main heads ; viz.: (1) preparation of proteins ; (2) preparation of the spinning solution ; (3) spinning of fibre; and (4) hardening and after-treatment.

Preparation of Proteins

The first step consists in the preparation of the oil-free cake from the residue obtained after pressure expulsion of oil, through solvent extraction. The final cake has the following average composition :

	%
Nitrogen	10·5
Oil	1·5
Water	8·0
Ash	4·5

Peanut proteins are made up of the two globulins, arachin and conarachin, soluble in saline or alkali solutions. Extraction of proteins from the oil-free cake is one of the most critical operations in fibre production and demands extreme care for ensuring uniform batches. One method of extraction consists in soaking the powdered cake (of definite fineness) in 5 to 10 per cent saline at 15° to 20°C. for 6 to 12 hr. The quality of the finished proteins is influenced by the clarity of the extract, and a Sharple's supercentrifuge is employed for securing the desired clarity. The proteins are precipitated by adding to the extract freshly prepared, dilute sulphurous acid solution, or by slowly bubbling sulphur dioxide through the extract at 15° to 20°C., till a *p*H of about 5 is attained. The precipitated proteins are filtered, washed and dried at 30° to 40°C. The relative ratio of salt solution to the cake is also important. Ten parts of salt solution are usually employed for extracting the protein from one part of the cake. High temperatures during extraction lower the yield. Rate of stirring, concentration of sulphur dioxide, *p*H at precipitation, extent of washing and temperature of wash liquor, and sunlight are other factors which influence the physical properties of the proteins precipitated.

In another method, a dilute solution of sodium hydroxide (0·1 to 0·5 per cent) is used for extracting the proteins. The dissolved proteins are precipitated by sulphur dioxide at a *p*H of about 5. The use of lime has also been suggested[3]. The final product is cream-white in colour, and the yield varies from 20 to 30 per cent on the weight of the cake.

Preparation of Spinning Solution

The second step in fibre manufacture is the preparation of a viscous solution of high solid content. On account of the tendency of proteins to form a gel at high concentrations, it is difficult to prepare solutions containing more than 20 to 30 per cent solids. The solutions are then aged and " ripened ", during which reorientation and partial denaturation of protein molecules occur. Denaturation is controlled and is allowed to proceed only to the extent of eliminating side chains leaving the main long-chain molecule intact. Thomsan and Traill[5] have shown that at higher solution temperatures, lower concentrations of alkali are required to uncoil the globular molecules to a linear configuration and later break them down to short chains. A viscosity between 40 to 300 poises[6] provides a solution which can be spun into fibres.

To impart the necessary pliability, softness and elasticity to the finished fibre, it is desirable to incorporate certain plasticizers in the spinning solution. One per cent oleic acid, linseed oil acids, Turkey-red oil, sodium glycerol phosphate, sodium tartarate, glycol phthalate, ethyl glycollate, butyl tartarate, amyl acetate, sodium maleate and urea (in the order of their effectiveness) are a few of the plasticizers in use.

As strengthening agents (for increasing the tensile strength of the fibre), salts of aluminium, calcium, barium and, to a less degree, magnesium are recommended. While using calcium and barium salts it is necessary to use the sulphonates of alcohols, fats or aromatic radicals, with or without hexa-metaphosphate, to prevent premature precipitation of proteinates. Sodium lauryl sulphate and sodium oleyl sulphate are particularly effective. Of less value as strengthening agents are sodium silicate, sodium chromate, sodium tungstate and sodium stannate.

Technically, the preparation of spinning solution consists in soaking the proteins in water for 30 to 60 min., and grinding to a uniform paste. A small amount of alkali (1·0 to 1·5 per cent) is added slowly with continuous stirring when all the proteins go into solution. Plasticizers and strengthening agents are added and intimately mixed. The solution is then filtered in a stream-line filter and aged at 10° to 15°C. for 15 to 25 hr. The quality of different batches of the spinning solution depends upon the size of protein particles, rate of stirring, rate and order of addition of the additives and other factors.

Spinning consists in forcing the protein solution through spinnerets into an acid bath, and collecting the filaments on a bobbin as in the spinning of rayon.

The process may be conveniently described under two main heads :

Precipitation — Effective precipitants[7] are sulphuric acid, phosphoric acid and acetic acid (in 0·5 to 15 per cent concentration). Addition of small amounts of sulphates of calcium, aluminium and sodium and glucose to the bath provides a better control of the speed of coagulation and facilitates dehydration of the filaments. A small amount of formaldehyde is added to the bath for imparting strength to the fibre at an early stage.

Spinning Technique — The spinning solution, properly aged and freed from insolubles and air bubbles, is forced through fine nozzles dia. 10 microns) into an acid bath where the proteins are precipitated in the form of fibres, which are wound on a bobbin. Although horizontal baths are in general use, vertical baths may be more suitable. Stretching of the fibre during its precipitation is effected by pulling it through the acid bath over two glass pulleys revolving at different speeds. Stretching appreciably influences the physical properties of the finished fibre, unstretched fibres being brittle and weak.

When the spinning solution comes into contact with the bath solution, coagulation of the proteins occurs at the surface, leaving a core of uncoagulated solution within the fibre cylinder.

When the fibre is subjected to stretching, the rate of pull will be more on the coagulated solid layer than on the enclosed uncoagulated solution, i.e. a shear will be set up which brings about combing, alignment or orientation of the protein molecules. As the fibre leaves the acid bath (the length of the bath is about one metre), the enclosed solution has to be completely coagulated. It is obvious that to produce a good textile fibre, there should be a proper correlation between the speed of stretching and the rate of coagulation. The accompanying diagram (FIG. 1) illustrates the different speeds at the axis and circumference of the coagulating fibre. Spinning speeds up to 400 to 500 ft. per min. are generally employed[8]. Viscosity and temperature of spinning solution, speed of extrusion and stretching of the coagulated fibre, temperature, and composition of the acid bath, etc., are all factors which influence the final characteristics of the fibre.

The hardening of the fibre is brought about by a relatively long immersion (12 to 24 hr.) of the tow (thousands of tiny strands coming from the spinnerets) in a formaldehyde bath maintained at a definite temperature, concentration and *p*H. The use of more than one bath with increasing formaldehyde concentration is recommended[9]. Other hardening agents[7] include acetaldehyde, croton aldehyde and butrylaldehyde, but they are seldom used.

The amount of formaldehyde taken up by the fibre depends upon the *p*H of the solution. The presence of certain salts added to the bath also influences formaldehyde absorption.

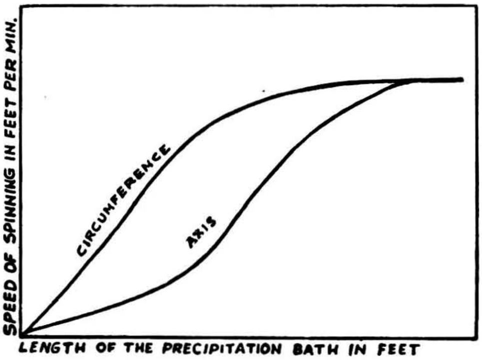

FIG. 1

Peanut fibre, unlike soyabean and casein fibres, forms a rod-like horny mass after the formaldehyde treatment and drying. To overcome this drawback, the hardened fibre is soaked in a hot solution of sulphurous acid in an organic solvent at 80°C. for 10 to 30 min., or in a solution containing sodium chloride and formaldehyde at 35° to 40°C. followed by treatment with a 5 per cent batching-oil emulsion, or sulphonated oil and soap[10]. This treatment imparts the feel, pliability and other properties required for a textile fibre. The product, finally washed and dried under controlled temperature and humidity conditions[11], resembles scoured wool and can be spun and woven by the conventional methods.

Properties of the Fibre

" Ardil ", the commercial name given to the fibre produced by *Imperial Chemical Industries Ltd.*, is white to light-cream in colour and has a soft wool-like feel. The crimp in the fibre and the consequent inclusion of air in fibre bundles gives the fibre a warm woolly handle. All " Ardil " and " Ardil "-wool fabrics give the same thermal insulation as an all-wool fabric of the same thickness. It has a low tensile strength (15,000 p.s.i., and an extension at break of 80 per cent) and poor resistance to abrasion. It absorbs moisture like natural wool and its affinity for dyes is comparable to that of other synthetic fibres. It does not felt like natural wool and fur. Knitting yarns are prepared from " Ardil " mixed with cotton and rayon.

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NOTES & NEWS

Synthesis of Vitamin A

VITAMIN A IN A HIGH STATE OF purity has been synthesized at the *Warner Institute of Therapeutic Research*. Complete identity between the natural and synthetic product has been established. A method suitable for commercial production has also been worked out.

Starting with a C_{18} ketone, first described by Heilborn, a mixture of vitamin A acid and ester was obtained by the addition of 2 more carbon atoms. This was reduced to the vitamin, employing lithium aluminium hydride. The final product had a purity of 50-75 per cent, a concentration higher than any vitamin A concentrate commercially available up to now.

The synthesis of vitamin A in the form of its acetate, starting from β-ionone, has been achieved on a sufficiently large scale (*Chem. & Eng. News*, 1949, **27**, 2106).

Alcohol from Bassia Flowers

OPTIMUM CONDITIONS FOR THE fermentation of bassia flowers to yield industrial alcohol, together with a plant design, are described (*Ind. & Eng. Chem.*, 1949, **41**, 1451).

Bassia latifolia flowers from C.P. and Berar containing, without hydrolysis 70 per cent reducing sugar as glucose, 5·9 per cent proteins (dry basis), and 18·4 per cent moisture, were used in these experiments. *Seagram yeast strain No.* 90 (*S. cerevisiae*) employed in all the experiments was acclimatized by serial transfer through bassia medium. 20, 25 and 30 gm. of flowers per 100 c.c. of water were macerated and the material heated at 180°F. for 30 min. Conversion of sugar to alcohol was highest at 20 per cent concentration of the mash. A fermentation efficiency of 98·3 per cent after 72 hr. incubation and a plant efficiency of 92·6 per cent were obtained. The alcohol (95 per cent) yield was 5·32 gal. per 100 lb. of flowers, dry basis, in contrast to yields of 4·89 and 4·58 gal. for 25 and 30 per cent concentrations respectively.

To determine the effect of temperature and time on extraction of sugar, whole flowers were extracted with agitation at room temperature and at 180°F. Cold extraction for 30 min. was found inferior to a similar hot extraction, as the former yielded 4·28 gal. as compared to 4·93 gal. 95 per cent alcohol in the latter case.

To augment the yield, various nutrients were added to 20 per cent mash which had been extracted for 30 min. at 180°F. Use of ammonium sulphate and ammonium phosphate at 0·2-0·6 per cent level yielded 5·05 gal. of alcohol 5 per cent. Urea was not found suitable as a nutrient.

A bassia flower distillery with a capacity of 2,000 gal. of industrial alcohol per day has been designed on the basis of this experimental work. The process consists of continuous automatic weighing of bassia flowers, water, and 5 per cent bassia stillage into a precooker. Bassia slurry prepared in the cooker after a holding time of 20 min. at 180°F. is passed on for wet milling followed by a 30 min. batch cooking operation at 180°F. 5 per cent (by volume) of the mash is diverted for yeast propagation while the remainder is pumped continuously through a single-tube heat exchanger where it is cooled to the fermentation temperature of 80°F. Four 25,000 gal. fermenters provide adequate space for the 60-70 hr. fermentation cycle required by a 20 per cent bassia flower mash.

The distillation unit consists of a direct steam-heated beer stripping still, containing 18 perforated plates and 2 bubble cap plates, a beer heater which acts as a partial condenser, and an alcohol-vapour fed rectifying column, containing 30 bubble cap plates.

With bassia flowers at $1.21 per cwt. *f.o.b.* the supplier, the cost of 95 per cent industrial alcohol manufactured by this process works out to $0.372 per gal.

Electrolytic Production of Azo-dyes

AN ELECTROLYTIC METHOD FOR the preparation of azo-dyes using an inexpensive iron anode in place of the usual expensive platinum anode has given satisfactory results in practice. The cell characteristics and the preparation of a few azo-dyes have been described (*Curr. Sci.*, 1949, **18**, 291). The iron anode is not only cheap ; it is corroded only to a negligible extent and does not promote nitrite oxidation. The yields of the dyes are also considerably improved.

The cell used is of the divided type, a porous alundum cup forming the cathode chamber and diaphragm. The anode is of heavy sheet iron, with the edges and corners rounded by filing. This anode is cleaned, heated to redness and chilled in water before being used ; the reaction is found to be unsatisfactory without this treatment. The anode is an iron oxide anode ; the thin layer of oxide formed helps in the passivation of the anode. A glass propeller-type stirrer is used for stirring.

On passing the current 3 times the theoretical length of time, a better yield of dye is obtained : 3·07 gm. or 61·4 per cent of the theoretical. The current efficiency, however, remains the same.

Ponceau G—This dye is obtained by the electrolysis of molar proportions of aniline, 2-naphthol-3, 6-disulphonic acid (disodium salt) and sodium nitrite in an alkaline electrolyte. The dye is formed in very poor amounts in neutral medium. 4·35 gm. R-salt, 1·16 gm. aniline, 0·90 gm. sodium nitrite and 0·50 gm. sodium hydroxide, in 150 c.c. of water are electrolysed at a current density of 5 amps./dm.^{2} for 20 min. The dye is recovered from the anolyte by salting out. The precipitated dye is filtered at the pump, dried and weighed. Yield of dye : 1·15 gm. or 20·3 per cent of the theoretical. The colour on silk and wool is bright and of a good shade.

Ponceau 2R—4·35 gm. of R-salt, 1·50 gm. m-xylidine and 0·90 gm. of sodium nitrite are electrolysed in 150 c.c. of water, with the modifications mentioned above, at a current density of 5 amps./dm.^{2}. The anolyte is coloured dark red at the end of the run. The dye is precipitated from solution on the addition of 25 gm. of sodium chloride with good stirring. The precipitated dye is filtered, sucked nearly dry and dried in an air oven at 80°C.

The yield of the dye is 95·5 per cent of the theoretical if the electrolysis is carried on for 2 hr. The quality of the dye is found to be satisfactory.

Glass Sand Beneficiation Process

A TECHNIQUE OF ATTRITION GRINDing that makes possible the production of a high-quality glass sand from a low-grade sandstone has been described (*J. Amer. Cer. Soc.*, 1949, **32**, 237).

The sandstone used is from Pottsville near Falls Creek, Pennsylvania. A typical sample analyses to : SiO_2, 98·75 ; Fe_2O_3, 0·12 ; Al_2O_3, 0·67 ; TiO_2, 0·10 ; CaO, 0·07 ; MgO, nil ; Na_2O, nil ; K_2O, 0·09 ; and ignition loss, 0·037, expressed as percentages. Petrographic examination of thin sections of specimens showed that about 60 per cent of the quartz was free and loose-grained ; the remainder consisted of aggregates of quartzite, chert and iron-bearing minerals. The sand also contained about 1·5 per cent of a ferruginous clay as a coating on the grains.

To obtain a suitable sand for the manufacture of flint glass, the Fe_2O_3 and TiO_2 content had to be reduced to about 0·025 per cent or less. The remaining impurities only needed to be made constant and it was not necessary to eliminate them.

The first beneficiation tests were started with impact grinding and washing. A sample of crushed rock, 40-50 mesh, was given washing and desliming treatments after which the sand contained 0·056 Fe_2O_3, 0·061 TiO_2 and 0·07 per cent Al_2O_3. The remaining iron was present as a limonite stain on the particles and as discrete mineral particles with or without titania. This was followed by flotation and magnetic separation which were not found effective. A hot acid-leach was then tried with hydrochloric acid and stannous chloride. It was found that the iron content with 50 mesh sand could be purified to 0·023-0·026 per cent iron. The remaining iron was assumed to be held inside the quartz grains as it could not be removed. Acid-leach test with SO_2 as the solvent also gave a similar product, although it took a longer time.

Large-scale beneficiation carried out with these methods proved complicated, economically unfeasible and unsatisfactory. Further study and microscopic work revealed that the sand had to be reduced in size by attrition grinding rather than by uncontrolled impact grinding. The pebbles had to slide over the surface of the sand grains to remove the limonite stain. The ratio of sand charge to pebble charge for maximum efficiency was found to be 1·1 to 8 for attrition grinding compared with 3 to 8 for impact grinding. The lower ratio permitted the reduction of sand by attrition to a certain size in one-half the time required to reduce the same amount of sand to the same fineness by standard impact grinding methods.

Attrition grinding resulting in a −150-mesh product (95 per cent) was found to be most economical and dependable. The beneficiated product contained 0·04 per cent TiO_2. Though this amount was not so low as the standard set originally, it was found harmless for the glass colour.

In the beneficiation plant described, sandstone from the quarry is subjected to primary crushing in jaw crusher and gyratory crusher and further reduced in a rod mill to −10 mesh. This material passes through a wet magnetic separator to the tube mills in a closed circuit with a bowl classifier, for attrition grinding to −150 mesh. This is freed from slime by a hydro-separator and passed on to a thickener, a filter and a drier successively. The water content of the product is less than 1 per cent.

Contact Catalysts

CONTACT CATALYSTS MAY BE PREpared by incorporating a suitable catalyst with a refractory carrier which is bonded by ethyl silicate. The preparation of contact catalysts by supporting a suitable catalytic agent on a refractory material is a common practice and usually the catalyst is prepared in pellet form. For example, a vanadium pentoxide catalyst supported on pumice pellets, bonded with sodium silicate, may be used to catalyse the oxidation of sulphur dioxide to sulphur trioxide. As a binder, sodium silicate has the drawback of impairing the refractory properties of the supporting material and also makes it difficult to obtain intricately shaped catalytic masses. If an organic silicate is used as the binding material, these drawbacks may be overcome. Ethyl silicate is generally chosen for this purpose. Suitable solutions can be prepared from ethyl silicate by hydrolysis with aqueous ethyl alcohol using hydrochloric acid as a catalyst. Since non-volatile inorganic bases are absent, the refractory properties of the supporting materials are unimpaired. Catalysts containing vanadium pentoxide have been prepared in this manner.

Ethyl silicate containing up to 5 per cent of piperidine by volume may be slip-cast to give intricately shaped objects and after air-drying and firing between 700°C. and 1000°C., a hard, bonded refractory object is obtained, uncontaminated by inorganic bases.

By incorporating a suitable catalytic agent in the slurry during the above bonding process, the preparation of contact catalysts having complex shapes can be carried out, provided that the bonding process does not destroy the activity of the catalyst, or that the presence of the catalyst does not prevent the setting of the slurry. Instead of the direct incorporation of the catalytic agent in the mixture, a precursor which forms the catalyst on ignition may be added. Sillimanite blocks containing vanadium pentoxide and platinum catalyts have been prepared by this method. To prepare the vanadium catalyst, powdered hydrated vanadic oxide was mixed with the graded sillimanite. The block containing the platinum catalyst was prepared by suspending freshly prepared ammonium platinichloride in the aqueous ethyl alcohol required to gel the ethyl silicate-piperidine mixture. The blocks were air dried and fired as described above.

After preparation, the catalyst blocks were granulated and loaded into a hard-glass combustion tube. Catalytic activities were assessed qualitatively by heating to redness and passing over the hot material a mixture of equal volumes of sulphur dioxide and oxygen. In both cases much sulphur trioxide was formed. A control experiment, performed with catalyst-free material, resulted in the formation of very little sulphur trioxide. The vanadium catalyst block was also found effective in the oxidation of ethyl alcohol. In the temperature range 100°-150°C., acetaldehyde was obtained whilst at 200°C., appreciable quantities of acetic acid were also formed. Tentative experiments indicated that the vanadium pentoxide catalyst is also effective in the oxidation of naphthalene to phthalic anhydride.

The above results indicate that vanadium pentoxide and platinum catalysts may be incorporated in sillimanite blocks bonded by an ethyl silicate and piperidine mixture, and retain their oxidative catalytic activity (*Chem. & Ind.*, July 1949, p. 500).

Oxidizing Agent for Organic Compounds

GLYCOLS ARE OXIDATIVELY CLEAVed to carbonyl compounds by sodium bismuthate in an acid medium, the reagent thus behaving in a similar way to periodic acid and lead tetra-acetate ; hydroxy acids are degraded to the lower aldehydes.

Two principal procedures are used : (a) The reagent is added to a solution of the glycol in glacial acetic acid and stirred or shaken at, or slightly above, room temperature, until the colour is discharged and a clear solution results (taking a few minutes or several hours) ; this procedure is also suggested as a convenient test for these classes of organic compounds. (b) The reaction is similarly carried out with the glycol in aqueous (purified) dioxan solution in the presence of phosphoric acid, the initially yellow-orange suspension being ultimately replaced by pure white bismuth phosphate.

The following substances have been oxidized and the products isolated (in parenthesis) in good yield (70-100 per cent) : ethylene glycol (formaldehyde) ; diethyl tartrate (ethyl glyoxylate) ; *cis*- and *trans*-cyclohexane 1 : 2-diol (adipic aldehyde) ; hydrobenzoin (benzaldehyde); lactic acid (acetaldehyde) ; mandelic acid (benzaldehyde). The reaction has been successfully applied for the preparation of ethyl glyoxylate and adipic aldehyde, and also for pelargonic aldehyde and azelaic aldehyde acid from 9 : 10-dehydroxystearic acid (*Nature*, 1949, **164,** 185).

Corrosion Test for Plastics

A LABORATORY METHOD TO EVALuate chemical resistance of various grades of plastics has been evolved by *Haveg Corporation*, U.S.A. (*Chem. Eng.*, 1949, **56,** 98).

The weight loss test used for measuring corrosion in metals is not applicable to plastics which may lose weight due to evaporation of the plasticizer or gain by absorbing solutions, on account of their porous structure.

The new test procedure is based on 4 tests involving changes in the appearance of the sample and the solution (acid or alkali solution in which the specimen is kept immersed) and changes in weight and volume of the sample. All these data have to be considered together to arrive at a conclusion.

The tests are described with particular reference to *Haveg* (a proprietary product by *Haveg Corporation*) but are adaptable to all plastic materials. Further, in order to provide a semi-quantitative system of reporting results, a system of ratings on a percentage basis has been worked out. The final rating depends on the average of the 4 individual ratings for the 4 tests. This procedure also makes possible the comparison of results of different workers and prediction of field results with considerable accuracy.

The sample under test, cut into strips of 3/8″ × 3/8″ × 3/4″ strips, of known weight and original volume are placed in a 3/4″ dia. test-tube, 6″ long. Stainless steel containers are advised for strongly alkaline solutions. For hydrofluoric acid and its compounds at elevated temperatures, *Haveg* 48 or a plastic tube made from pure phenolic resin is recommended. The end of the tube is then drawn to a capillary tube in a blast lamp. The tube and the sample are put in a beaker of test solution with capillary down, in a vacuum desiccator. The vacuum is applied and released, filling tube by suction to about 3/4″ depth over sample. The tube and the contents are then warmed to the required test temperature. The capillary is sealed and the tube is maintained at test temperature by keeping in an oven, or water or oil bath for the duration of the test which may be up to 3 months. At the end of this period, the solution is observed for colour, cloudiness or sediment and rated accordingly by reference to the relevant table. The sample is then taken out of the tube, wiped dry, and weighed immediately. It is observed for any visual disintegrative changes and rated. The final volume of the sample is obtained by calculating quickly the specific gravity, preferably using a Jolly balance. From this data are obtained the changes in volume and weight which, on reference to the standard graphs, give the respective ratings. When all the 4 ratings are obtained, they are averaged to give one percentage rating. This gives the value of the sample under the test conditions. The test being not precise enough for a purely quantitative comparison, the results are also frequently reported as excellent, good, fair or poor.

These results are checked and calibrated against known results for standard chemical-resisting grades, in order that they can be used for prediction of field results. A very close concordance has been found to exist between the laboratory and the field results.

Spectroscopic Identification of Timbers

A SPECTROSCOPIC METHOD FOR the identification of 16 varieties of Indian timbers is described (*Science & Engineering*, 1949, **2,** 114). Aqueous extracts of the specimen (5 gm.) was prepared by allowing the pieces of timbers to remain in 100 c.c. of a 5 per cent solution of sodium hydroxide for 24 hr. Alcoholic extracts were obtained by extracting 5 gm. of the wood in 100 c.c. of an alcoholic caustic soda solution containing equal volumes of rectified spirit and caustic soda solution (0·91 gm./c.c.) for 24 hr. The filtered solutions were examined in a Hilger spectroscope of the constant deviation type fitted with a wavelength scale. A 100-watt pearl lamp was used for illumination.

TABLE I

TIMBER	AQUEOUS EXTRACT, WAVELENGTH ABSORBED	ALCOHOLIC EXTRACT, WAVELENGTH ABSORBED
	Å	Å
Dysoxylum malabaricum	4239	4250
Terminalia belerica	4360	4405
Calophyllum tomentosum	4430	4270
Lagerstroemia lanceolata	4513	4275
Tetrameles nudiflora	4635	4350
Xylia xylocarpa	5770	4395
Terminalia paniculata	4790	4440
Mangifera indica	4742	4500
Dichopsis elliptica	5128	4600
Cedrella toona	5407	4710
Terminalia arjuna	4933	4850
Tectona grandis	5970	5295
Albezzia lebbek	5334	5445
Atrocarpus integrifolia	5282	5670
Atrocarpus hirsuta	5384	5960
Dalbergia latifolia	5685	...

The position of the absorption bands of the extracts were recorded and mapped.

The difference in the absorption values observed for the aqueous and alcoholic extracts of a specimen serves as an additional aid in identifying a timber.

Table I gives the wavelengths absorbed by the different timber extracts.

The results of these studies show that ranges of absorption are specific for each wood extract and serve to identify a particular type of timber.

New Sectioning Technique

THE APPLICATION OF THE ELECTRON microscope to many biological problems has been seriously hampered by the lack of a rapid practical method of cutting uniformly thin sections having adequate area and integrity of structure. Because of the very slight penetrating power of the beam in commercial electron microscopes and the great relative depth of field involved, specimen structure is difficult to interpret when sections are over a fraction of a micron in thickness.

The high-speed microtome has been of limited use in solving the problem. This precision instrument is not only expensive and complicated, but also produces a low percentage of useful sections. The recent method by Pease and Baker employs the standard histological technique of embedding the tissue in collodion and paraffin and a modified Spencer rotary microtome in which the unit of advance was reduced approximately to one tenth the calibrated value used to produce sections as thin as $0{\cdot}1\mu$. This technique is not, however, handy because of the exacting demands made on the microtome-advancing mechanism and the difficulty involved in obtaining thin sections with standard embedding media.

A promising new procedure for obtaining extremely thin sections uses a methacrylic resin as an embedding medium, a thermal expansion device for advancing the specimen in a commercial microtome, and metallic shadowcasting for increasing observable detail in some of the sections. These techniques form the basis of the new method for producing thin sections suitable for obtaining transmission images at the higher magnifications in the conventional light, phase-contrast and electron microscopes. Polymerization of n-butyl methacrylate provides a rapid and simple means for embedding the biological material in a solid resin. This gives an optically clear matrix from which the sections are cut, one at a time. Smooth, continuous advance of the embedded specimen toward the knife is effected by the thermal expansion of a brass specimen holder, which permits the microtome, with its advancing mechanism disengaged, to cut ultra-thin sections having uniform thickness, large area and integrity of tissue structure.

Before embedding, the tissues are fixed and then dehydrated by the usual cytological techniques. From absolute alcohol, they are transferred to a solution containing equal volumes of absolute alcohol and pure monomeric n-butyl methacrylate from which the inhibitor has been removed. After about 1 hr. in the alcohol-monomer mixture, the tissues are put in the monometer alone for an equal period. To ensure removal of the alcohol, they are then placed in two additional changes of monomer for at least 1 hr. in each.

No. 00 gelatin capsules are convenient embedding moulds. The main body of the capsule is set upright in a wooden block or other base and filled with the monomer, to which has been added 1 per cent (by weight) of a catalyst (2, 4-dichlorobenzoyl peroxide). After the tissue is placed in the mixture, the capsule lid is slipped on to retard evaporation, and the assembled capsule is placed in an oven kept at a temperature of 45°-50°C. For even heating, the capsules are suspended by strips of cellophane tape and good air circulation is maintained in the oven.

At the end of 6-8 hr., the monomer is polymerized into a solid matrix containing the tissue embedded at the bottom of the clear plastic. An additional period of several hours at this temperature will ensure complete cure. After soaking in water, the gelatin capsule may be peeled off the resin.

The device for holding and advancing the embedded specimen consists of a brass block with a hole threaded at one end to receive a standard ⅜″ brass pipe plug. A cavity drilled into the face of the plug provides a seat for the embedded specimen. Behind the plug is a needle valve, which admits compressed carbon dioxide. As the gas undergoes a large change in volume, it cools and contracts the assembly. Stopping or reducing the flow of gas allows the apparatus to approach room temperature again and thus provides continuous advance of the embedded tissue toward the cutting edge.

In practice, the embedded specimen is first cemented into the mounting block with a mixture of pure gum-rubber and paraffin. Then, with the device clamped in the jaws of the microtome head, the entire assembly is cooled below room temperature. Upon the appearance of a thin layer of frost on the metal, the knife is adjusted so that the specimen just misses it on the cutting stroke. The specimen is then mechanically advanced at 2 or 3μ increments until the first slice is made. At this point the mechanical advancing mechanism of the rotary microtome is disengaged by setting it to zero and the gas flow is reduced or stopped. After a few seconds, the specimen can be cut again. Because the specimen is advancing continuously, a quick chopping stroke involving one complete revolution of the hand wheel is necessary. With a little experience, one can soon judge the necessary time interval between cuts. Some control of the rate of specimen advance can be obtained by bleeding the carbon dioxide at various reduced rates into the expansion chamber.

Although polybutyl methacrylate has excellent cutting properties, the sections usually are found to be somewhat folded. They are lifted from the knife with a dry camel-hair brush, picked up with a dissecting needle and placed on a water surface warmed on a hot plate to about 35°C. After a period ranging from a few minutes to an hour or more, many will flatten out on the surface and exhibit bright interference colours. These sections are then floated on to clean microscope slides and allowed to dry flat. Sections prepared for phase contrast microscopy are placed in acetone or toluene for about ½ hr. to remove the matrix and are mounted in Canada balsam. For ordinary light microscopy, the matrix is dissolved off and the section stained in the usual way.

In preparing material for the electron microscope, the sections are floated from the water on to clean glass slides and dried flat. The matrix is then dissolved out by placing the slide in acetone, toluene, or amyl acetate.

A dilute solution of collodion in amyl acetate is allowed to flow over the slide bearing the tissue, which is then permitted to dry at room temperature. The collodion film containing the section is floated from the slide on to water, and the specimen-mounting screens of the electron microscope are placed over the area of the film containing the section in the usual manner.

This process of wide application in electron microscopy produces a three-dimensional aspect as well as greater contrast in structural details of the tissue.

Although this new method for obtaining very thin sections has given satisfactory results, it possesses certain limitations. Knife sharpness, for example, is of vital importance. The tilt of the knife during sectioning is not particularly critical. The greatest chance for failure appears to lie in the polymerization of the embedding mass. Use of low temperature catalyst and maintenance of a curing temperature of 45°-50°C. will usually prevent the formation of insoluble resins. Occasionally, tissues are injured during the polymerization reaction and such tissues, which are easily detected, can be promptly discarded. For this reason, the use of fixing solutions with good hardening properties is recommended. While fixation artefacts remain problems for serious consideration, the new technique provides an inexpensive, practical method for producing ultra-thin sections of tissue in almost a routine fashion (*Science*, 1949, **110,** 66).

Recent Advances in Instrumentation

A WIDE VARIETY OF NEW LABOratory instruments has been developed and announced in the U.S.A. during recent months. These range from new instruments to detect atomic energy radiations to a new vacuum-tube voltmeter for measuring voltage in electronic and other low-energy circuits (*Chem. Age*, 1949, **61,** 155).

Resistance Thermometer—A new thimble-size platinum resistance thermometer using the same temperature-sensitive element as the standard platinum thermometer but $\frac{1}{8}$th its size, measures temperatures in the region of 10°K and higher in calorimetric experiments. The instrument can also be employed as a primary standard for resistance thermometry between $-190°$ C. and $+500°$C. With the 2-G Mueller bridge temperature, measurements can be made to an accuracy of $\pm 0{\cdot}01$°C.

The thermometer has a platinum protecting tube, about 48 mm. long and 5·6 mm. outside dia. and can be readily mounted entirely inside a calorimeter by casting in low-melting alloy. Being helium-filled, it is usable down to 5°K. Four-wire current and potential leads are brought out through a glass seal. Nominal resistance at 0°C. is 25·5 ohms.

Potentiometer — The K-2 potentiometer of *Leads & Northrup Company* combines precision with facility of operation so essential for studying tiny high-speed particles constituting beta rays. Its chief features are the addition of a third and lower range, elimination of exposed electric conducting parts and standardization on any of its 3 ranges (0-0·0161, 0-0·161 and 0-1·61). The last $\frac{1}{10}$th, $\frac{1}{100}$th or $\frac{1}{1000}$th volt is spread over a 5 meter slide-wire, making it easy to follow a small fluctuating voltage with accuracy not ordinarily possible where several switches must be manipulated within the range of fluctuations.

The potentiometer can be used for various measurements of potential difference, hydrogen-ion concentration, oxidation reduction, polarization, temperature, and other measurements.

Consistometer — A new apparatus, the " consistometer ", with a simple design and high efficiency, for measuring the flow characteristics of lubricating greases at constant temperature as the grease is being mechanically worked has been devised. It can similarly determine the apparent viscosity of rubber solutions at different shear and temperature. The principal advantages of the apparatus are its flexibility, wide range of shear and consistency measurements, short test cycle and provision for characteristics flow data while working under controlled conditions.

The consistometer is composed essentially of two coaxial steel cylinders mounted vertically, with a capillary-type shearing element in between through which grease under test is forced in either direction by close-fitting pistons. Dead weights on the down-stroke and air pressure on the up-stroke are used on the pistons while making flow measurements. Provision is also made for automatic mechanical working. Possible rates of shear cover a rather wide range, from about 10 to 100,000 reciprocal seconds.

Calibrating Hygrometers—A new humidity test apparatus for research, calibration and testing of hygrometers and various humidity instruments at low temperatures has been developed at the *U.S. National Bureau of Standards.*

A current of dry air is divided into 2 streams (by a proportionating valve) one of which is maintained dry, while the other is completely saturated over a series of trays containing ice. The 2 currents are then mixed in a mixing chamber and allowed to escape through a test chamber into the atmosphere. All the 3 chambers — the saturator, mixing chamber, and test chamber — are kept immersed in a constant temperature bath. Essential functional units of the apparatus are : the drying system, the proportionating system, the humidifying system, the test chamber, the cooling system, and the thermoregulating system for temperature control.

The instrument fills a basic need in meteorology an is expected to find application in refrigeration, air-conditioning and other fields in which humidity plays an important role.

Freezing Point Determination Apparatus — A precision apparatus involving a platinum resistance thermometer and bridge for evaluating purity of hydrocarbons by measuring their freezing points has been developed by the *U.S. Bureau of Standards.* Procedure employed in the determination utilizes time-temperature observations of the liquid solid equilibrium in freezing and melting experiments.

5 c.c. of the sample to be examined are placed in an inner vacuum flask surrounded by a refrigerant and stirred by an overhead electric motor. Observations of time and temperature are made, starting with the temperature of the liquid above the freezing point and continuing until stirring becomes difficult. By plotting these values, using a suitable scale on the temperature axis, it is possible to determine graphically the " zero time " at which crystallization would have begun in the absence of undercooling. The corresponding temperature value is then the true freezing point. The purity is

calculated from the measured freezing point as well as the value of the freezing point for zero impurity and the cryoscopic constants.

Determination of freezing points for substances such as ethylbenzene, which do not normally yield time-temperature freezing curves, is made from melting curves. The procedure is practically the same as for freezing experiments except that when stirring becomes difficult, the cooling bath is replaced by an appropriate warming bath.

Air Meter — Development of " Anemotherm ", a three-way air meter providing air velocity, air temperature and static pressure readings at the turn of a knob is reported. The meter is expected to find wide application in adjusting and testing equipment used for heating, ventilating and air-conditioning.

The operation of the instrument on self-contained batteries and provision of the small probe attached to a long flexible cable make the instrument portable and permit readings even in the neck of the air diffuser. The meter measures air velocity from 10′ per min. to 500′ per min., provides air-response measurement of temperatures from 30°F. to 155°F. Static pressure, either negative or positive, may be read directly in in. of water from 0·05 to 10 positive, and 0·05 to 4 negative. The new instrument detects even the slightest draughts and measures air velocity accurately regardless of direction of air flow.

Raman Spectrograph — This instrument, designed by the *Esso Laboratories*, is based on the discoveries first made by Sir C. V. Raman in 1928.

The photo-electric recording spectrograph, which is capable of measuring the amount of light from a candle 5 miles distant, has found application in the analysis of petroleum products in the laboratories of the *Standard Oil Development Co.*

Measurement of Thickness of Microtome Sections

A SIMPLE AND ACCURATE MEANS for measuring the thickness of very thin microtome sections has been described (*Science*, 1949, **110**, 73).

Several optical methods are available for measuring thin films but their use is difficult when the area of the section is of the order of a sq. mm. A much simpler way is to measure, under the microscope, the area of the section, then to melt the section into a sphere from whose diameter the volume of wax can be calculated. To obtain the sphere, the section is caught on a very thin (less than 10μ) glass fibre, and the fibre is mounted in the field of a microscope. A small loop of electrically heated wire is carefully guided close to the section, which is slowly melted. If the glass fibre is thin enough, an almost perfect single sphere will result; for greater accuracy, one may prefer to calculate its volume as an ellipsoid. It is important to watch the heating under fairly high magnification, and to heat slowly, as too high a temperature will cause evaporation.

Estimation of Fatty & Rosin Acids

A SWEDISH METHOD FOR ACCURATE estimation of fatty and rosin acids by complete esterification of the fatty acids is described (*Chem. Age*, 1949, **61**, 184).

The mixture is esterified with *n*-butanol and benzene and the water formed is separated. Complete esterification of the fatty portion takes place but at the same time rosin acids are also partially esterified. To reduce this, as far as possible, a suitable catalyst (benzene sulphonic acid) is used in low concentration, which is constant, and the temperature of the reaction is kept down during treatment. The concentration of the catalyst being constant, a definite amount of rosin acids is esterified per time unit. Thus, it is possible to introduce an exact correction for the fraction of rosin acids converted during cooking.

The unesterified rosin acids in the reaction, mixture are determined titrimetrically. If the acids are of varying molecular weights, gravimetric methods have to be applied.

Palm Oil Substitute

A SYNTHETIC SUBSTITUTE FOR THE expensive African and East Indian palm oil, which is extensively used in the manufacture of hot-dip tin-plate, has been developed at the *Armour Research Foundation*, Chicago (*Chem. Age*, 1949, **61**, 185).

In making hot-dipped tin-plate, sheets of pickled steel are passed through a flux into a bath of molten tin and through an 18″ layer of hot (450°F.) palm oil. The palm oil substitute has therefore, to be one which makes smooth, bright and quality tin-plate, drains rapidly and freely from the sheet, neither oxidizes nor polymerizes readily and has low viscosity at 100°-200°F. so that it may be pumped or poured readily. Moreover, the oil has to be non-toxic, odour-free and preferably edible; should not become rancid during storage, or interfere with lithographing, lacquering or soldering procedures and should retard atmospheric corrosion.

It has been found that dimerized linoleic acid (dimer acid) consisting of 2 linked molecules of linoleic acid has the best possibilities. Its flash point (600°F.) is even higher than that of palm oil (470°F.) and its volatility is one-third of the African product. A mill test on this material was encouraging except that the acid tended to increase in viscosity.

A New Industrial Amine

A PRIMARY AMINE—ROSIN AMINE D, of great industrial utility—has been developed by the *Hercules Powder Co.* Derived from modified rosin, the amine is a pale-yellow, viscous liquid having a density of 0·997 at 25°C. Commercial qualities have a purity of 88-92 per cent. The amine is soluble in most organic solvents but is practically insoluble in water. With most of the film formers and resins, it is compatible. At room temperature, it forms salts with mineral acids and organic acids of low molecular weight. With high molecular-weight organic acids, the reaction takes place at 70°-95°C. Carbon dioxide combines with the amine to form a white crystalline unstable salt. Within a specific range of concentrations of 0·02-0·4 per cent, the salts of the low molecular-weight organic acids exhibit hazy or cloudy solutions with distilled water.

Inorganic salts of Rosin Amine D are practically insoluble in water or organic solvents. The *p*H values of their dispersions and of dilute solutions of soluble salts range from 5·4 to 6·8, the values decreasing with higher concentrations. The amine and its carboxylic acid salts give a positive Libermann-Storch test for rosin.

Non-ionic type emulsifiers such as Tweens and Renex can emulsify the amine and its salts in water solutions; ionic type emulsifiers, e.g. sulphonated castor oil, Naccanocol and rosin soaps are not suited on account of their reaction with the amine to form salts. Fatty acid soaps, if used with concentrated amine solutions, cause gel formations.

Rosin Amine D and its salts are stable up to 100°C. They darken slightly on ageing in air at normal temperatures. On heating at 100°C., slight decomposition with loss of nitrogen takes place, and higher temperatures induce further darkening and decomposition.

The amine does not cause any appreciable corrosion of metals. It is non-toxic; concentrated forms irritate the skin slightly due to its weak alkaline action.

The chemical is used as a flotation agent, rubber softner, corrosion inhibitor, herbicide in the control of crab grass, preservative and germicide, and as a chemical intermediate in the preparation of numerous derivatives. Its addition to asphalt, mastic cements improves adhesion. In ceramic inks its use gives better flow and spreading properties. The amine and its derivatives are expected to find wide application in breaking oil emulsions in the production of crude petroleum.

Synthetic Detergents

THE PRODUCTION AND THE USES of synthetic detergents in the United States are described (*C.T.J.*, 1949, **125**, 33).

The first commercial production of synthetic detergents was in Germany during the early 1930's, when the extreme shortage of fats and oils led to the development of a series of sulphonated alcohols which had superior detergent properties. The better types of synthetic detergents possess the following general properties: (1) excellent wetting ability; (2) neutrality in solution (non-hydrolysing); (3) solubility and effectiveness in cold water; (4) ready rinsability; (5) resistance to hard and sea water and acid and alkaline solutions; (6) excellent foaming properties; and (7) tastelessness.

One important technical weakness was recognized early for most synthetic detergents, that is, that they are not equal to soap for the removal of heavy oils, particularly from cotton fabrics. Whether a synthetic can compete with soap in soft water as well as hard water depends on the following considerations: (1) quality and price believed to be satisfactory; (2) ability to wash heavily soiled cottons: not yet comparable with low-cost detergents, but potentially so; (3) capability of being made into bars: not yet satisfactory; (4) supplies of chemical raw materials: capable of eventual solution.

Listed below are the chief types of synthetics falling in the anionic and non-ionic groups:

Anionic — (1) Alkyl aryl sulphonates; (2) sulphated fatty alcohols; (3) miscellaneous sulphates and sulphonates: (a) alkyl sulphonates; (b) sulphated esters and acids; (c) amide sulphates and sulphonates; (d) sulphated and sulphonated oils, fats and waxes.

Non-ionic — (1) Fatty esters of glycerol and glycol and other polyhydric alcohols, such as sorbitol; (2) condensation products of ethylene oxide with fatty acids, alkyl phenols and mercaptans.

Within the types of compounds mentioned, there are literally hundreds of possibilities.

The alkyl aryl sulphonates, because of their excellent properties and low price, have been pushed as a competitor for soap in household cleaners, especially in hard-water areas. In addition they have wide industrial usage.

Sodium lauryl sulphate represents the bulk of the sulphated fatty alcohol detergents, and is prepared from coconut oil. At present the bulk of this type of product is marketed as household powders.

There has been considerable interest in the past few years in the non-ionic synthetic detergents because of their good detergency and low foam. The principal drawback to these materials are their inherent high price and liquid form.

With the use of "builders", sodium tripolyphosphate and tetrasodium phosphate, it is possible to produce an ideal household detergent using synthetics.

Prevention of Corrosion in Metals

RECENT METHODS FOR PREVENTING metal corrosion were reviewed at the recent U.N. Scientific Conference on the Conservation and Utilization of Resources (UNSCCUR). Humidity control, de-aeration inhibitors, cathodic protection and alloying materials were some of the methods discussed.

Humidity — At relative humidity below 30 per cent, corrosion is negligible. Air-conditioning in large spaces and suitable moisture absorbing substances such as silica gel with packaged apparatus can accomplish this. When the package is a metal container, the inside air can be replaced with an inert gas such as nitrogen.

De-aeration — Removal of atmospheric or other oxygen from the environment, e.g. the use of a de-aerator in the treatment of water for boiler-feed and steel pipe lines inhibits corrosion.

Inhibitors — Certain compounds which function mostly as chemically or physically absorbed films either alter the electro-chemical characteristics of the metal or serve as mechanical barriers to the normal corrosion processes. Reactions of various chemical inhibitors are described.

Cathodic protection provides the most effective means of arresting corrosion. It is based on the principle that most corrosion in practice is electro-chemical in nature and results from the flow of current through an electrolyte between areas of different potential which may exist on the surface of the single metal or between two or more different metals. The solution is to bring all surfaces to the same potential. This is done by introducing a current to offset the one produced by the difference in potential, i.e. by discharging current on the more cathodic surfaces, so as to achieve their cathodic polarization to the potential of the adjacent anodes. The method finds the most important application in connection with miles of underground oil, gas and water pipe lines and power and communication cables (*Sci. News-letter*, 1949, **56**, 4).

Liquid Oxygen Converter

A NEW AUTOMATIC AND COMPACT liquid oxygen converter for aviation, industrial and medical purposes has been developed at the *U.S. National Bureau of Standards* (*Chem. Age*, 1949, **61**, 255).

The equipment, 25″ high and 18″ dia., weighs 60 lb. and holds 62 lb. oxygen enough for 10 men for 10 hr. It is sturdy, simple in design and operation, attains pressure rapidly, consumes less oxygen and delivers warm gas. It consists of a standard 25-litre metal Dewar flask modified by the addition of a bottom drain, together with 2 coils, one for build-up of pressure and the other for warming the gas as it is delivered. After it is filled with liquid oxygen, the flask can be sealed off from the atmosphere, To build up pressure, a valve is opened to allow liquid to flow through a drain tube and liquid trap at the bottom of the flask and into the build-up coil where it is evaporated and warmed by the atmosphere. The warm gas rises through the coil by thermal convection until it enters the top of the flask where it mixes with the gas above the liquid. Here the gas that condenses on the liquid furnace is immediately replaced by freshly warmed gas and the cycle continues, the gas pressure rising quickly. The fact that it is not necessary to heat the entire mass of liquid to raise the pressure constitutes an important difference between this and earlier types of liquid oxygen converters.

The equipment delivers gas at a temperature differing by only about 5°C. from the atmospheric temperature for flows up to 150 litres per min. When the container is full, pressures from 0 to 65 lb. p.s.i. are built in 10 sec. and are quite stable. The equipment has successfully withstood severe heat, cold and vibration tests. Operation is reported to be satisfactory at altitudes up to 40,000′.

Black Stainless Steel

A NEW TYPE OF STAINLESS STEEL, the " shineless stainless steel ", is finding application for many industrial purposes. Like the conventional bright stainless steel, this black variety has a thin chromium oxide coating and the blackening of the surface is achieved by placing the metal in a molten bath of dichromate at a temperature of 730°-750°F. for 15-30 min. The black surface, which is resistant to the same, degree as the ordinary stainless steel because of its non-reflecting surface is finding application in military requirements. It is also used in certain types of jewellery (*Sci. Newsletter*, 1949, **55**, 394).

New Uses for Rubber

A NEW INDUSTRY FOR MANUFACTURING rubber hydrochloride from natural rubber and hydrochloric acid is being developed in Holland. A simpler and far more economical process than the American for the manufacture of rubber hydrochloride (Pliofilm) has been perfected. The process will enable natural rubber to compete with synthetic rubber and plastics.

Rubber hydrochloride has been found very suitable for packing purposes especially where water-clear transparency and very low moisture permeability are required. Another important use of natural rubber announced is the manufacture of ebonite tanks for storage of solid and liquid corrosive chemicals. These tanks are strong, stable and inexpensive (*Chem. Age*, 1949, **61**, 122).

Fire Resistance & Sound Insulation of Walls

THE STRUCTURAL STABILITY, FIRE resistance and sound insulation requirements of " party walls " between houses are dealt with in the *National Building Studies Special Report No.* 5 published by the *Department of Scientific & Industrial Research.*, U.K. (*H.M.S.O.*, London, Price 2*s*.). The report discusses the twofold problem : firstly to determine standards of performance in respect of fire resistance, and secondly to find ways of assessing whether particular forms of construction will attain the requisite standards.

Until the middle of the 19th century, the need for fire protection was the major consideration in the design of " party walls " apart from the obvious requirements of structural stability. A solid wall of 9″ of brickwork was the accepted and adequate form of construction. Since then the noise problem has so increased as to render this wall inadequate as a barrier to noise, while new methods of construction introduced in recent years are impracticable because of their high cost.

The design of the wall should be such as to meet the following specification : A party wall should provide a complete separation between houses, from foundation level to the underside of the roof covering. It should afford at least 1 hr. fire resistance and should provide an average sound reduction of 55 db. between the living rooms in one house and the living or bed-rooms in another house ; and 45 db. between other rooms.

Higher grades of fire resistance should be provided between houses of combustible construction and at defined intervals in terrace houses. In the latter case, the wall should, in addition, be self-stable.

Of the single and multiple-leaf systems of construction, the four-leaf system in which the individual leaves are insulated and have a total weight in leaves of 12-15 lb./sq. ft., appears to give an average sound reduction of 55 db.

To avoid reduction of fire resistance by perforation of the wall with floor joists, the joists should preferably run parallel with the party wall. This is also a useful measure in respect of sound insulation.

Indirect sound transmission due to common floor beams and wall plates can be overcome by using a double-frame system of construction which can be more easily adapted to give the necessary standards both of fire resistance and sound installation than single-frame systems.

Weight for weight, such materials as clinker concrete, foamed slag and wood-wool appear, on present evidence, to be better sound insulators than the denser materials such as dense concrete or brickwork.

Indian Pulp & Paper

THE ANNIVERSARY NUMBER OF this journal (July 1949 ; price Rs. 3), in addition to the usual features, contains some excellent and instructive articles. Articles of interest include : Research for the Paper Industry ; Nature of Bamboo Lignin ; Paper ; Origin, History and Manufacture ; Photography as an Aid to the Paper Mill Chemist ; Water Marks, etc.

Electronic Measuring

THE *Philips Laboratories*, EINDHOVEN, Holland, have issued 9 numbers of the second volume of their technical bulletin entitled *Electronic Measuring* in which are discussed the applications of electronic measuring technique. The bulletin gives an idea of the significance of new techniques and developments in electronic measurements. The publication is supplied *free* of charge on request to *Philips Electrical Co.* (*India*), *Ltd.*, " Philips House ", 2 Heysham Road, Calcutta 20.

REPORTS FROM STATES & PROVINCES

(*Continued from page* 463)

160,000 seedlings to plant about 2,000 acres. Seednuts from selected trees having all the desirable characters will be collected mainly in the months of February to June and the supply of seedlings will commence in July and continue throughout the monsoon months.

Reports from States & Provinces

MYSORE

Sugar Factory for Shimoga

MYSORE STATE, ONE OF THE MAIN sugar-producing areas, will have another sugar factory situated at Shimoga, in the Malnad area. The factory is estimated to cost Rs. 50 lakhs on capital expenditure and Rs. 15 lakhs as annual recurring expenditure. The necessary licence for obtaining the plant and machinery has already been obtained.

Fertilizer Factory

CONSTRUCTION WORK ON THE Government chemical and fertilizer project estimated to cost Rs. 3 crores and capable of producing 25,000 tons of ammonium sulphate and 25,000 tons of nitro-chalk annually is to commence by the end of this year. The necessary import licence for the plant has been obtained and plans are being finalized in consultation with the *Chemical Construction Corp. Ltd.* of New York.

RAJASTHAN

Beryl Resources in Ajmer-Merwara

THE ANNUAL OUTPUT OF BERYL from the mica pegmatites in Bihar and Madras would probably be 5-10 tons during mica mining. The principal source will be Ajmer-Merwara, where the mineral occurs more extensively than in other parts of the Indian Union ; these sources are said to have supplied about 40 tons a month during the war years.

Further detailed mapping in parts of Assam, Orissa, Bihar and Central Provinces, Hyderabad and Madras, may expose some beryl-bearing pegmatites. On a conservative estimate a few hundred tons per annum may be raised in the Indian Union.

In Ajmer tehsil, the most important beryl workings are located near Lohagal and Makrera, the latter with a total production between 500 and 1,000 tons. Promising localities are Foya Sagar Lake, Qazipura, Boraj, Barla and Rajosi. Indications are noticed in the neighbourhood of Chamunda, Palran and other localities. In Beawar region, the localities from where large quantities of beryl were hitherto obtained are within 3-4 miles from Jawaja. Dhoti Danti in Kharwar is a noticeable locality. In Kekri area the important localities are Bogla, Gulgaon, Para, Gonda, Kacharia, Kalera, Khawas, Khoda, Nemode and Bisundini. The total production from Bisundini mine is known to have varied from 500 to 1,000 tons. The promising occurrences in Nasirabad area are Tihari, Tilara, Tantuti, Shokla, Loharwada, Hanwantia and Kanpura. The deposits in Ajmer-Merwara are by no means exhausted and large quantities of beryl are yet to be forthcoming under favourable conditions of mining industry. On an average, about 50-100 tons per annum may be easily obtained from Ajmer-Merwara.

The systematic survey of the beryl occurrences in Ajmer-Merwara has shown that usually large workable deposits of beryl are not present with good quality ruby mica. Large-scale mining for low grade mica, may yield large quantities of beryl as by-product.

MADRAS

Breeding of Improved Varieties of Millets

Rust Resistant Strain of Panicum italicum (Korra)—This millet crop, common in the ceded districts, is invariably susceptible to the attack of a rust, characterized by rusty, brown spots on the leaves. This rust disease is responsible for poor yields. A selected strain, S.L. 3756, evolved at the Millet Breeding Station, Coimbatore, has been found to be comparatively resistant to the rust and yields a bigger harvest.

Improved Strain of Irrigated Sorghum vulgare (Cholam) — A high-yielding strain, K. 2, has been found suitable for cultivation in two seasons—January/February and April/May — in certain districts. The strain matures earlier than the local varieties. The cultivation of the strain not only saves the cost of one irrigation but also gives extra produce valued at Rs. 37-8-0 per acre at the present price for the millet.

New Eleusine Corocana (ragi) strain — K. 1, a *ragi* strain, yielding 18 per cent more than the local variety (288 lb. per acre) isolated at the Agricultural Research Station, Koilpatti, is now available for distribution. A net profit of Rs. 36/- per acre is expected out of its cultivation.

Hybrid Varieties of Pennisetum typhoideum (Cumbu) Two new hybrid varieties of *cumbu*, X. 1 and X. 2, have been recently released for trial from the Millet Breeding Station, Coimbatore. They have been produced by crossing promising pure lines which exhibited the maximum hybrid vigour when crossed. Trials conduced in Tiruchirapalli district have been encouraging.

Green Manure — An acre of paddy field requires 25-30 lb. of small-sized green manure seed like *Pillipesara*, *Sesbania speciosa*, etc. Efforts to produce seed of this green manure crop on the paddy-field bunds at the several agricultural research stations show that it is easily possible to obtain 25 lb. of seed from *Sesbania speciosa* planted on the well-trimmed field bunds of an acre of land immediately after planting paddy in July-August. Planting of seedlings is to be preferred to dibbling seed directly on the bunds. Nursery of *Sesbania speciosa* should be sown on a small high-level plot 4-5 weeks in advance of the completion of paddy planting.

Coconut Seedlings

IN ORDER TO SUPPLY THE PUBLIC with selected seedlings at comparatively low price, a comprehensive coconut nursery scheme financed by the Government and the *Indian Central Coconut Committee* was sanctioned by the Government of Madras in October 1948, and was put into operation on 10th November 1948. Nurseries have been started at the 8 research stations, viz. Anakapallee, Samalkot, Marutera, Tindivanam, Pattukottai, Coimbatore, Pattambi and Nileshwar. Under this scheme, it is proposed to produce annually

(*Continued on page* 462)

INDIAN PATENTS

The following is a list of Patent Applications notified as accepted in the *Gazette of India,* Part II, Section 1, for September 1949. The patents from the *Council of Scientific & Industrial Research* are indicated by asterisks.

Paints, Varnishes & Lacquers

*40752. SIDDIQUI, SARIN & SINGH : Conversion of bhilawan shell liquid to a non-vesicating drying product for the manufacture of lacquer varnishes, stoving enamels, water resistant and flame resistant paints : *Treating bhilawan shell liquid with chlorine until chlorine content is at least* 25-30 *per cent.*

Organic Chemicals

41029. UNITED STATES RUBBER COMPANY : Process of making DDT (22nd July 1948) : *Characterized in using fluosulphonic acid as condensing agent in a continuous process.*

Miscellaneous Chemicals

40983. SOLVAY & CIE : Process for oxidizing substances with chlorite solutions : *The substance is immersed in a suspension of activating agent and then it is dipped in the chlorite bath.*

40984. SOLVAY & CIE : Process of activating chlorite solutions and bleaching materials with these solutions : *Introducing into the chlorite solution a hydrolysable organic solvent.*

Instruments (Professional, Scientific & Controlling)

41665. ROY : An improved balance : *Balance provided with ball bearings at one or more points of movement.*

Food & Kindred Products

39650. DIJK : Improved method, process and means for decorticating cashew nuts and like products : *Freezing and cracking the nuts and then separating shell from kernel.*

40835. PITHWALA & SREENIVASAN : Preparation of a modified tamarind seed polyose suitable for food industries : *Tamarind seed polyose is subjected to the action of citric acid.*

39384. THE DOOR CO. : Improvements in clarification of sugarcane juice : *Passing lime into admixture with mud in the lowermost compartment of a clarifier.*

40949. ANHEUSER-BUSCH, INCORP. : A process for producing riboflavin, yeast or vitamins of the B complex : *Adding a culture to the nutrient medium, containing the culturing and separating the cellular material.*

Drugs & Pharmaceuticals

38838. N. V. PHILIPS' GLOEILAMPENFABRIEKEN : Methods of preparing medicinal vitamin C tablets: *Mixing* 10 *gm. of vitamin C per gram of a mixture of cocoabutter, sugar and cocoa.*

41357. GLAXO LABORATORIES LTD. : Preparation of liver extracts : *Passing the aqueous extract of liver through a silica gel column and thereafter eluting the active principle.*

41358. GLAXO LABORATORIES LTD. : Preparation of liver extracts : *Subjecting an extract of liver to a counter current extraction process between water and a suitable solvent as defined herein.*

40472. CHAUDARY : Improved driving arrangement for cycles : *Another free wheel on opposite side of rear wheel axle, carrying a length of chain.*

Metal & Metal Products

40929. DESAI : Extraction of metal copper from malachite or ores containing copper carbonates: *Reacting ferrous sulphate of* 5N *strength with malachite to form copper sulphate to which scrap iron is added.*

Rubber & Rubber Products

41067. SINGH : An improved process of manufacturing rubber balloons : *Liquid latex is allowed to fall from the orifice of a container into a rotating former.*

Textile & Textile Products

41119. INDIAN JUTE MILLS' ASSOCIATION RESEARCH INSTITUTE : Preparation of a fast-to-light substantially white jute (Addition to No. 37868) : *Treating bleached jute with acetic anhydride at a temperature below its boiling point in the presence of an alkali metal acetate.*

Miscellaneous

39131. DEWAR & MCLEOD & CO. LTD.: Tea-breaking machines : *Breaking effected by pressure and bending applied to the belt and to tea on the belt.*

9660. CHAUDARY : An improved plough : *Two tilling points spaced apart from one another and at different levels.*

*40112. NARAYANA RAO & SREENIVASAYA : A process for production of distillery yeasts of high potency : *Securing a highly active zymase complex within it and elaborating protective agents within the cell, which stabilize the zymase complex.*

40600. SINGH : Typewriters for Hindi, Devnagri and like scripts : *Half characters provided on type bodies and vertical stroke provided on a linear adjacent to guide, typebar actuating the lever when half character key is depressed.*

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CONTENTS

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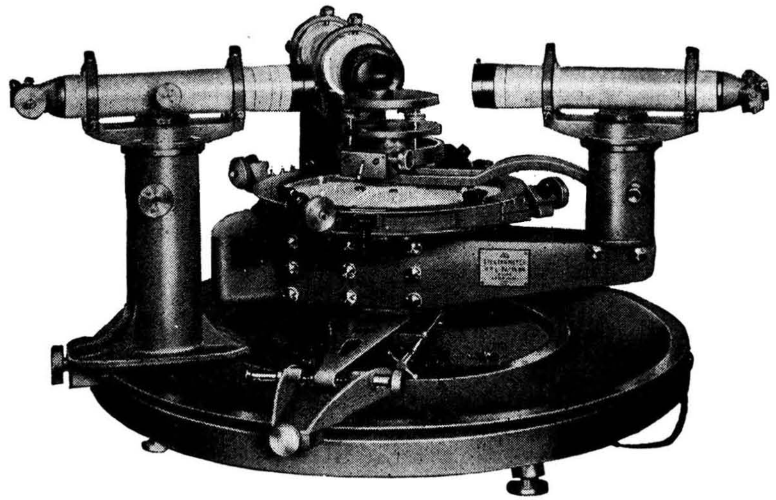

Studies in the 3-phenyl Coumarin Series: Part I—Synthesis of Some New Members of the Series

PRITHVI RAJ BHANDARI, JOGENDRA LAL BOSE &
SALIMUZZAMAN SIDDIQUI
Chemical Laboratories, C.S.I.R., Delhi

WHILE 2- and 3-phenyl-γ-benzopyrones, namely flavones and isoflavones on the one hand, and simple coumarins and furo-coumarins on the other, form important groups of natural products, the phenyl-α-benzo-pyrones, namely 3- and 4-phenyl coumarins have not so far been found to occur in nature. This is particularly striking in reference to 3-phenyl coumarins and isoflavones, as in both cases the 3-aryl radical is attached to the α-carbon atom with respect to the carbonyl group of the pyrone ring, and an interchange of the position of —CH= and —CO— with the corresponding adjustment of the double bond would convert an isoflavone to a 3-phenyl coumarin, or the latter into the former. This is illustrated by the interesting work of Baker[1] relating to the attempted conversion of the isoflavan derivative, anhydro-catechin tetramethyl ether, to a tetramethoxy-isoflavone by oxidation, which actually resulted in the formation of a tetramethoxy-3-phenyl coumarin. A further illustration of the same point was given by the attempted synthesis of 7-hydroxy isoflavone through the condensation of α-formyl-phenyl-acetonitrile with resorcinol resulting in the formation of 7-hydroxy 3-phenyl coumarin[2].

The work embodied in the present paper was started in the first instance to exclude the possible 3-phenyl coumarin structure for biochanin A, one of the main constituents of Bengal gram (*Cicer arietinum* Linn.) germ[3], the constitution of which was later established as 4′-methoxy 5, 7-dioxy isoflavone on the basis of degradation experiments[4], and confirmed by its synthesis by Shriner and Hull[5], in connection with their work on the constitution of prunetin. Studies in the synthesis of 3-phenyl coumarins were subsequently extended further, in view of the lack of any systematic work in the field and also in consideration of the fact that some members of the series have been found to possess high haemostatic activity.

Since the first synthesis of the parent compound of the series, namely 3-phenyl coumarin by Oglialoro[6], no work was done in the field for a long time until Bargellini and co-workers[7] synthesized a number of 3-phenyl coumarins in connection with their work on the constitution of some α-benzopyrones, synthesized by Jacobson and Ghosh[8] and presumed by them to be γ-benzo-pyrones. Later Bargellini, by the synthesis of 4′, 5, 7-trioxy and trimethoxy 3-phenyl coumarins, which proved to be different from prunetol, the demethylated product of prunetin and methyl prunetin respectively, excluded the possible 3-phenyl coumarin constitution for prunetin, the isoflavone constitution for which was subsequently established by a synthesis of prunetol (genistein) by Baker and Robinson[9].

Meerwein and co-workers[10] have also recently synthesized a few 3-phenyl coumarins in the course of their studies on the reaction of aromatic diazo compounds with α-β-unsaturated carbonyl compounds.

Out of the three general reactions available for the synthesis of 3-phenyl coumarins, namely (1) Perkin's reaction ; (2) Knoevenagel's reaction ; and (3) the reaction of aromatic diazo compounds with coumarin, the first was found in the present studies to be most suitable for the synthesis of oxy-3-phenyl coumarins. In this method the oxy-coumarins are initially obtained as acetoxy derivatives, and the method of deacetylation had, therefore, to be carefully chosen for ensuring good yields. In this connection deacetylation with concentrated sulphuric acid at 0°C. was found to be better than either deacetylation with dilute sulphuric acid at higher temperatures or with alkali.

All the hydroxy and methoxy-3-phenyl coumarins synthesized in the course of the present work were found to exhibit strong fluorescence even in high dilutions in various solvents as well as in alkali and concentrated sulphuric acid. The fluorescence data of the coumarins along with their melting points are given in Table I.

Experimental

4′-Acetoxy-3-phenyl coumarin — A mixture of salicylaldehyde (0·3 gm.), sodium salt of *p*-hydroxyphenylacetic acid (0·5 gm.) and acetic anhydride (6 c.c.) was refluxed in an oil bath for 25 hr. at 150°-160°C. After cooling to the room temperature, the reaction mixture was poured in an excess of cold water and left overnight. The crude product (0·35 gm.) was collected and repeatedly crystallized from dilute alcohol (charcoal) when it was finally obtained in colourless prismatic rods, m.p. 183°C. It is soluble in alcohol, methanol, acetone, ether, ethyl acetate, chloroform and benzene and very sparingly soluble in petroleum ether.

Found : C, 73·2 ; H 4·6 ; required for $C_{17}H_{12}O_4$: C, 72·9 ; H, 4·3 per cent.

4′-Oxy-3-phenyl coumarin — 4′-Acetoxy-3-phenyl coumarin (0·2 gm.) was taken in a small flask and cooled in ice. Cold concentrated sulphuric acid (0·5 c.c.) was added to it, drop by drop, mixing the acid with the solid coumarin thoroughly after the addition of each drop when a dark, homogeneous, viscous mass was obtained. After keeping for 10 min., crushed ice was added to the reaction mixture and the separated coumarin was filtered and thoroughly washed with water. The crude product (0·16 gm.) was crystallized a number of times from alcohol (charcoal) when finally the pure coumarin was obtained in colourless prismatic rods, m.p., 202°C. It is soluble in alcohol, methanol, acetone, ether, ethyl acetate and chloroform, sparingly soluble in benzene and insoluble in petroleum ether.

Found : C, 75·7 ; H, 4·2 ; required for $C_{15}H_{10}O_3$: C, 75·6 ; H, 4·2 per cent.

On methylation with methyl iodide in acetone solution in presence of freshly desiccated potassium carbonate, it yields *p*-methoxy-3-phenyl coumarin, m.p., 143°-144°C. There was no depression in the melting point on admixture with a sample of 4′-methoxy-3-phenyl coumarin prepared from salicylaldehyde and sodium homoanisate.

4′,7-Diacetoxy-3-phenyl coumarin — A mixture of resorcylaldehyde (0·5 gm.), sodium salt of *p*-hydroxy phenyl acetic acid (0·72 gm.) and acetic anhydride (10 c.c.) was refluxed for 30 hr. at 170°-180°C. On working up in the usual manner, 0·5 gm. of the crude product was obtained. It was crystallized a number of times from acetone-methanol mixture and was finally obtained in small colourless needles, m.p., 213°-214°C. It is soluble in methanol, alcohol, acetone, ether, ethyl acetate, chloroform and benzene, and insoluble in petroleum ether.

Found : C, 67·2 ; H, 4·2 ; required for $C_{19}H_{14}O_6$: C, 67·4 ; H ; 4·1 per cent.

TABLE I — FLUORESCENCE CHARACTERISTICS OF THE VARIOUS COUMARINS

No.	Coumarin	M.P. °C.	Acetone	Alcohol	Ether	Concentrated sulphuric acid	5% Potassium hydroxide (aqueous)
1.	4′-acetoxy-3-phenyl coumarin	183	Nil	Nil	Nil	Yellow	Yellow
2.	4′-oxy-3-phenyl coumarin	202	Blue with violet tinge	Bluish violet	Bluish violet	Pale yellow	Yellow
3.	4′, 7-diacetoxy-3-phenyl coumarin	213-214	Very faint bluish violet	Faint violet	Very faint violet	Yellow	Yellow
4.	4′, 7-dioxy-3-phenyl coumarin	320-321	Bluish violet	Greenish yellow	Violet	Yellow	Greenish yellow
5.	4′, 7-dimethoxy-3-phenyl coumarin	182	Violet	Violet	Violet	Yellow	Yellow
6.	4′-methoxy-7-acetoxy-3-phenyl coumarin	178	Bluish violet	Bluish violet	Bluish violet	Yellowish green	Greenish yellow
7.	4′-methoxy-7-oxy-3-phenyl coumarin	232	Bluish violet	Bluish violet	Bluish violet	Yellow	Greenish yellow
8.	4′-methoxy-5, 7-diacetoxy-3-phenyl coumarin	182	Bluish violet	Blue	Blue	Pale yellow	Yellow
9.	4′-methoxy-5, 7-dioxy-3-phenyl coumarin	280-282	Bluish violet	Blue	Bluish violet	Deep yellow	Yellow

4′,7-Dioxy-3-phenyl coumarin — 4′,7-Diacetoxy-3-phenyl coumarin (0·2 gm.) was deacetylated with concentrated sulphuric acid (0·5 c.c.) at 0°C. On working up in the usual manner, 0·15 gm. of the crude product was obtained. It was crystallized from alcohol (charcoal) and then a number of times from methanol-acetone mixture and was finally obtained in aggregates of colourless needles, m.p., 320°-321°C. (with decomposition). It is soluble in methanol, alcohol, acetone, ether and ethyl acetate and insoluble in chloroform, benzene and petroleum ether.

Found : C, 70·5 ; H, 4·1 ; required for $C_{15}H_{10}O_4$: C, 70·9 ; H, 3·9 per cent.

4′,7-Dimethoxy-3-phenyl coumarin — 4′,7-dioxy-3-phenyl coumarin (0·1 gm.) was dissolved in dry acetone (10 c.c.) and freshly desiccated potassium carbonate (0·2 gm.) was added to it followed by methyl iodide (0·5 c.c.). The mixture was refluxed at 60°C. for 4 hr. and was left overnight at the room temperature. The solvent was removed *in vacuo*, and the residue was washed with water and crystallized from alcohol (charcoal) when the methoxy coumarin was obtained in colourless needles, m.p., 182°C. ; yield, 0·06 gm. It is soluble in alcohol, methanol, acetone, ether, ethyl acetate, chloroform and benzene and very sparingly soluble in petroleum ether.

Found : C, 72·1 ; H, 4·7 ; OCH_3, 21·3 ; required for $C_{17}H_{14}O_4$: C, 72·3 ; H, 4·9 ; OCH_3, 21·9 per cent.

4′-Methoxy-7-acetoxy-3-phenyl coumarin — A mixture of resorcylaldehyde (0·35 gm.), sodium homoanisate (0·5 gm.) and acetic anhydride (5 c.c.) was refluxed for 20 hr. at 160°-170°C. in an oil bath and the reaction product was poured into water and left overnight. The crude crystalline product was collected and repeatedly crystallized from acetone when the pure coumarin was finally obtained in aggregates of colourless needles, m.p. 178°C., yield, 0·3 gm. It is soluble in alcohol, methanol, acetone, ether, ethyl acetate, choloroform and benzene and insoluble in petroleum ether.

Found : C, 69·6 ; H, 4·7 ; required for $C_{18}H_{14}O_5$: C, 69·7 ; H, 4·5 per cent.

4′-Methoxy-7-oxy-3-phenyl coumarin — 4′-Methoxy-7-acetoxy-3-phenyl coumarin (0·2 gm.) was deacetylated with concentrated sulphuric acid (0·5 c.c.) at 0°C. After keeping at the room temperature (24°C.) for 5 min., crushed ice was added to the solution when the deacetylated coumarin separated out. On repeated crystallizations from methanol, it was finally obtained in colourless needles, m.p., 232°C. ; yield, 0·15 gm. It is soluble in alcohol, methanol, acetone, ether and ethyl acetate, sparingly soluble in choloroform and insoluble in benzene and petroleum ether.

Found : C, 71·5 ; H, 4·5 ; OCH_3, 11·0 ; required for $C_{16}H_{12}O_4$: C, 71·6 ; H, 4·5 ; OCH_3, 11·6 per cent.

On methylation of the coumarin with methyl iodide in the usual manner, a product identical with 4′,7-dimethoxy-3-phenyl coumarin, m.p., 182°C., was obtained.

4′-Methoxy-5,7-diacetoxy-3-phenyla coumarin — A mixture of phloroglucinol aldehyde (0·5 gm.), sodium homoanisate (0·75 gm.) and acetic anhydride (10 c.c.) was refluxed at 160°-170°C. for 30 hr. After cooling, water was added to the reaction mixture and left overnight. The solid which separated out was crystallized from alcohol (charcoal) and then from slightly diluted acetone when the coumarin was finally obtained in colourless silky needles, m.p., 182°C. ; yield, 0·43 gm. It is soluble in alcohol, methanol, acetone, ethyl acetate, benzene, ether and petroleum ether and sparingly soluble in chloroform.

Found : C, 64·9 ; H, 4·4 ; required for $C_{20}H_{16}O_7$: C, 65·2 ; H, 4·3 per cent.

4′-Methoxy-5,7-dioxy-3-phenyl coumarin — 4′-methoxy-5,7-diacetoxy-3-phenyl coumarin (0·1 gm.) was deacetylated with concentrated sulphuric acid at 0°C. The deacetylated coumarin crystallized from methanol-acetone mixture in small pale-yellow needles, m.p., 280°-282°C., yield, 0·07 gm. It is soluble in alcohol, methanol, acetone, ether and ethyl acetate, sparingly soluble in chloroform and benzene and insoluble in petroleum ether.

Found : C, 67·2 ; H, 4·1 ; OCH_3, 10·6 ; required for $C_{16}H_{12}O_5$: C, 67·6 ; H, 4·2 ; OCH_3, 10·9 per cent.

On methylation of 4′-methoxy-5,7-dioxy 3-phenyl coumarin with methyl iodide 4′, 5, 7 trimethoxy 3-phenyl coumarin crystallizing from alcohol in small colourless needles was obtained. It melted at 162°-163°C., as against 163°-165°C., recorded in the literature.

The phenolic aldehydes required for the synthesis were prepared by Karrer's method[11], namely by the action of cyanogen bromide on phenols, and homoanisic acid and *p*-hydroxy phenylacetic acid by hydrolysing the corresponding derivatives of phenylacetonitriles.

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Ultrasonic Absorption in Unassociated Liquids

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THE absorption in the case of polyatomic gases can be attributed to the excitation of vibrational levels of the molecules by ultrasonic waves, to the lag between the excited molecules returning to their normal levels and to the time period of vibration of ultrasonic waves, known as the relaxational phenomenon. In 1938 Kneser[1] explained the phenomenon of absorption in the case of liquids by assuming the same mechanism ; that is, he assumed that in the case of liquids also the vibrational levels are excited. The vibrational contribution at any particular temperature is calculated from spectroscopic data, viz. the frequencies of the most intense Raman lines and infra-red bands. It is an observed fact that the intensity of Raman lines decreases when the temperature is raised[2]. It follows, therefore, that with the rise of temperature the number of excited molecules increases and these molecules, when exposed to ultrasonic waves, will not be in a favourable condition to take up energy from the incident waves, i.e. the absorption coefficient will decrease. This leads to a negative temperature coefficient of absorption which is not verified by experiment.

The authors are of the opinion that the mechanism of absorption in the case of liquids is not entirely due to vibration of atoms, but that the structural changes in the quasi-crystalline state of liquids play an important part in the absorption of ultrasonic waves, and suggest that the phenomenon of absorption and viscosity are related and that both can be described in terms of " hole theory " of liquid state formulated by Furth[3] and Frankel[4] and developed by Auluck and Kothari[5]. Briefly, the conception of the liquid state may be described in the following manner.

The simplest model suggested by Furth is that the liquids also, like crystals, have short-distance order arrangement and geometrical regularity. In this lattice arrangement there are certain sites which are unoccupied by molecules. These unoccupied places are filled by molecules from outside. Similarly, in the lattice arrangement a molecule jumps out and causes a vacancy. At a given temperature a statistical equilibrium is maintained. These unoccupied places or sites have been given the name " holes ". Increase of temperature results in the destruction of holes and the geometrical structure and at the boiling point the structure of the holes is completely deranged. Hence it follows that the quasi-crystalline liquid structure must be endowed with a certain amount of energy when the holes are present. The energy can be calculated if the geometrical structure is known, otherwise we have to calculate it from other data. For instance, the energy at any temperature can be calculated from the rate of evaporation :

$$a = S\sqrt{\frac{kT}{2\pi m}} \quad e^{-\frac{A}{RT}}$$

where a is the number of molecules escaping from the surface of the body per unit time and under unit surface into the empty space, S the number of molecules per unit volume, m the mass of the molecules, k the Boltzman constant, T the absolute temperature and A the work function required to remove N molecules from the interior of the body to infinite distance.

In the case of ultrasonic waves, it is suggested that the statistical equilibrium at a particular temperature is disturbed and the number of holes increase. The required energy to increase the number of holes is taken from the incident sound waves ($\Delta T \sim 11°$C. for a sound intensity 10 W/cm² at 1 Mc.).

If during the cycle the sound wave completes one period, the holes return to their equilibrium value and no absorption will result. If, on the other hand, there is a lag, then the energy taken away by the holes will not be given back to the sound waves and this will amount to loss of energy from the sound waves.

Having in mind the structure of a liquid described above, we have to calculate the values of the work function of the holes in the geometrical configuration which we have postulated. The phenomenon of viscosity of liquids is explained on the basis of the quasi-crystalline structure of the liquid state and the thermal motion of particles with transference of momentum on collision. This represents the "hole" theory of viscosity. Writing A to represent the work function for the holes per gram molecule of liquid, the viscosity is given by

$$\eta = B\frac{T}{T_m} \cdot e^{\frac{A}{RT}} \tag{1}$$

where η is the viscosity coefficient, T and T_m the absolute temperatures (T_m melting temperature) and

$$B = 09{\cdot}45\frac{RT_m}{V_m}\sqrt{\frac{m.}{\sigma}}$$

where σ is the surface tension and R the gas constant.

If, therefore, η and η' are the values of viscosity at two different temperatures T and T'

$$A = R\frac{T\,T'}{T'-T}\log\frac{\eta T'}{\eta' T} \tag{2}$$

for a gram molecule of liquid. We assume the same type of structure in the liquid state under normal condition, and consequently the same work function in determining the increase in the number of holes when sound waves are incident upon the liquid. It is apparent that if the sound waves do not possess the requisite amount of energy, no increase in the number of holes will result. While, if the frequency of the sound wave is large, the same result will again follow.

Table I gives the value of A for various liquids from the viscosity data as calculated from formula (3). Viscosities at various temperatures have been taken from *International Critical Tables*[6] and *Physical & Chemical Constants* by Kaye and Laby[7].

Table II shows the values of the internal structural specific heat C_i as calculated from the formula

$$C_i = R\left(\frac{A}{RT}\right)^2 e^{-\frac{A}{RT}}$$

where A and R are as usual work function and gas constant respectively.

In applying the reaction rate theory, we assume that the excited condition is represented by the creation of holes in the liquid ; Bauer's formula[8] (equation 14) leads to the result

$$\frac{d}{N^2} = \pi\frac{C'}{C^2}\,\frac{\gamma-1}{C_p}\cdot\frac{C_i}{N_m}\,\frac{1}{\left\{1+\left(\frac{N}{N_m}\right)^2\right\}} \tag{3}$$

where d is the absorption coefficient, N is the frequency of sound waves C^1 is the velocity of sound when ultrasonic absorption takes place and C^2 the same without absorption, γ the ratio of specific heats, C_p specific heat at constant pressure, C_i the

TABLE I

LIQUID	A cal./mole
Carbon tetrachloride	3330
Benzene	3148
Chlorobenzene	2771
Chloroform	2291
Acetone	2250
Toluene	2207
Ethyl bromide	1812
Carbon disulphide	1802

TABLE II

LIQUID	C_i cal. deg.$^{-1}$ mole^{-1}
Carbon disulphide	0·8937
Benzene	0·2769
Carbon tetrachloride	0·2297
Chloroform	0·6446
Chlorobenzene	0·4295
Toluene	0·9181
Acetone	0·6486
Ethyl bromide	0·8640
Acetic acid	0·2423

internal structural specific heat and N_m the dispersion frequency.

The maximum absorption per wavelength at $N = N_m$ is given by

$$2\mu_m = \pi \frac{\gamma'}{\gamma} \frac{\gamma-1}{C_p} \cdot C_i \tag{4}$$

where γ' is the ratio of specific heats when ultrasonic absorption takes place and γ the same without absorption.

The dispersion frequencies have been calculated from the relation

$$N_m = \frac{2\mu_m}{C} \left[\frac{1}{d/N^2}\right] N << N_m \tag{5}$$

The dispersion frequencies have been determined for different liquids by substituting the experimental values of $\frac{d}{N^2}$. These results have been recorded in Table III. The values of absorption and the sound velocity c as shown in columns 2 and 3 of Table III have been taken from Bauer's paper[8] (TABLE I) and that of acetic acid from Bazulin[9] and Pinkerton[10]. It is to be noted that a very small change in the values of viscosity at a particular temperature has a large effect upon the specific heat C_i and consequently upon the maximum absorption frequency.

TABLE III

LIQUID	$d/N^2 \times 10^{17}$ sec.^{2} cm.$^{-1}$	$c \times 10^5$ cm./sec.	$2\mu_m$	N_m (Mc.)
Carbon disulphide	10,800	1·14	0·2789	23
Benzene	830	1·31	0·0979	90
Carbon tetrachloride	513	0·91	0·0652	140
Chloroform	380	0·987	0·1806	480
Chlorobenzene	123	1·302	0·1540	963
Toluene	84	1·31	0·2278	2070
Acetone	60	1·17	0·2301	3278
Ethyl bromide	56	0·932	0·2608	5067
Acetic acid	12,000	1·2	0·0701	4·87

The experimentally determined value of maximum dispersion frequency in the case of acetic acid is 2·5 Mc. while the hole theory gives 4·87 Mc. If we assume that there are vibrational levels in addition, the requisite value of C^i comes out to be 0·246 cal. deg^{-1} mole^{-1} and A is 7211 cal./mole. Subtracting from this the hole energy 3291 cal./mole, we get 3920 cal./mole — the additional vibrational energy corresponding to the frequency 1382 cm.$^{-1}$. This corresponds to frequency 1381 ± 5 cm.$^{-1}$ for monomer acetic acid[11].

It seems, therefore, that the energy levels of hole may have quantized values as suggested by Auluck and Kothari in their paper[5] " that the discrete energy levels of a hole may reveal their presence in the ultrasonic phenomenon ".

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Evaluation of Red Lead Pigments: Part III — Autocatalysis in Paint Sedimentation

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THE object of this paper is to report an interesting case of autocatalytic behaviour exhibited by a sample of " superfine " red lead paint during sedimentation. Sedimentation studies with a view to determine the particle-size distributions in red lead pigments were already reported by the author in an earlier paper[1]. Numerous samples of " ordinary ", " non-setting ", " high-disperse " and " superfine " samples of red lead were examined in the course of those investigations, both in the presence and also in the absence of an added dispersing agent such as aluminium stearate. A typical sedimentation curve and the size-distribution diagram deduced from the above by the graphical method of Svedberg are shown in Figs. 1a and 1b. All the samples behaved in the normal way in respect of the sedimentation velocity. The " superfine " red lead sample, however, when made into a paint paste with 0·75 per cent of aluminium stearate in the usual mixture of raw and double-boiled linseed oils and dispersed in white spirit for the purpose of determining the rate of sedimentation, was found to give a curve which was distinctly S-shaped and which called for an explanation.

The autocatalytic behaviour disappeared when the paint dispersion was thoroughly shaken in the same sedimentation tube after 24 hr. and the experiment repeated. Figs. 2, 3 and 4 show the sedimentation curves for " superfine " red lead paint in the

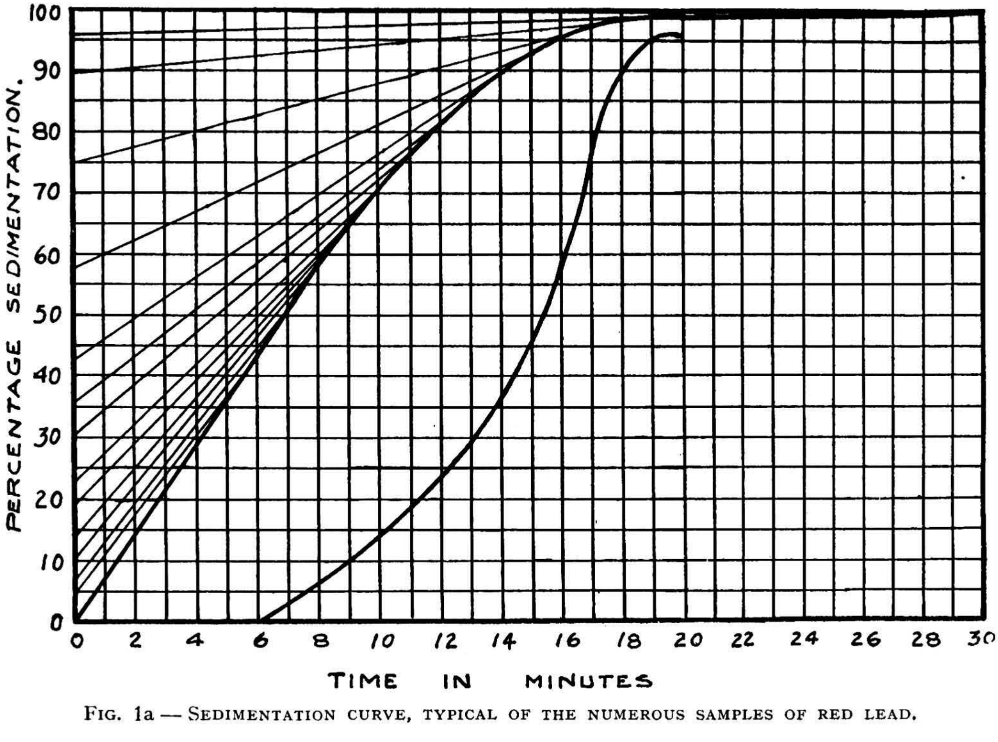

FIG. 1a — SEDIMENTATION CURVE, TYPICAL OF THE NUMEROUS SAMPLES OF RED LEAD.

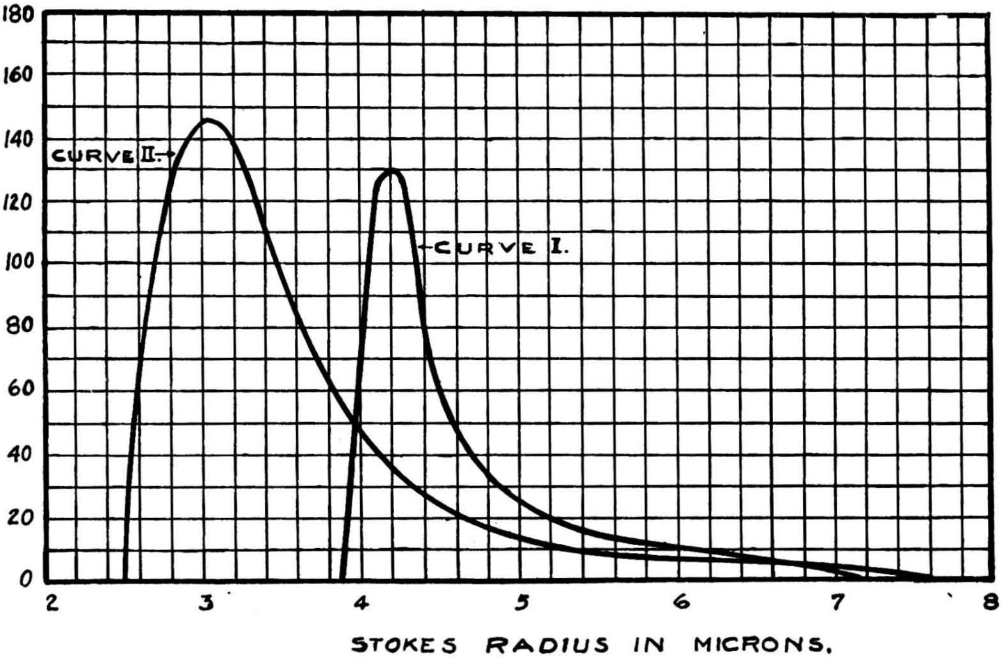

FIG. 1b — CURVE I : SIZE-DISTRIBUTION DIAGRAM, DEDUCED FROM ABOVE. CURVE II : SIZE DISTRIBUTION DIAGRAM FOR THE SAME PAINT, BUT WITH ALUMINIUM STEARATE AS A DISPERSING AGENT.

absence of aluminium stearate, in the presence of aluminium stearate and in the presence of the same when the experiment was repeated after 24 hr. in the same tube, respectively. The amount of aluminium stearate used was 0·75 per cent of the weight of the pigment.

The experiments were repeated in order to make sure that the autocatalysis was genuine and did not arise due to any flaw in the experimental procedure adopted, and the results were confirmed. None of the other samples so far examined exhibited this type of behaviour. The appearance of autocatalysis (FIG. 3) and its disappearance when the same system was examined after 24 hr. was somewhat curious. In order to track down the cause of such a behaviour, further experiments were carried out using fresh quantities of the same sample of red lead and repeating the experiment on the same system in the same sedimentation tube by shaking it vigorously at stated intervals of time before determining the rates of sedimentation. This gave interesting results which made it possible to offer a fairly satisfactory explanation regarding the causes for the autocatalysis and the latter's significance.

Experimental Results

The apparatus used, the general experimental procedure and the method of calculation of the rates of sedimentation and the particle-size distribution diagrams were described in an earlier paper[1]. The sedimentation curve shown in Fig. 1 is typical of those obtained for numerous samples of red lead pigment of different qualities and description either when used as such or after incorporation of a small percentage of aluminium stearate. The " superfine " sample itself, without aluminium stearate, gave the same type of curve that is normally obtained, as can be seen from Fig. 2. It was the incorporation of 0·75 per cent of aluminium stearate, along with the " superfine " red lead while making the paint, that changed the character of the sedimentation, as will be seen from the distinctly S shape of the curves in the initial stages of sedimentation (FIG. 3). When observations were taken on the same paint system after a lapse of 24 hr. by shaking the tube for thorough

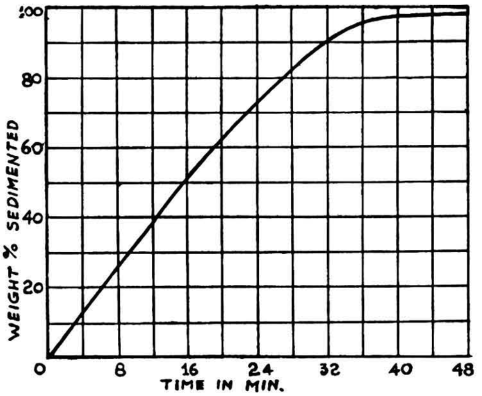

FIG. 2 — SEDIMENTATION CURVE FOR "SUPERFINE" RED LEAD MADE INTO PAINT WITHOUT ALUMINIUM STEARATE.

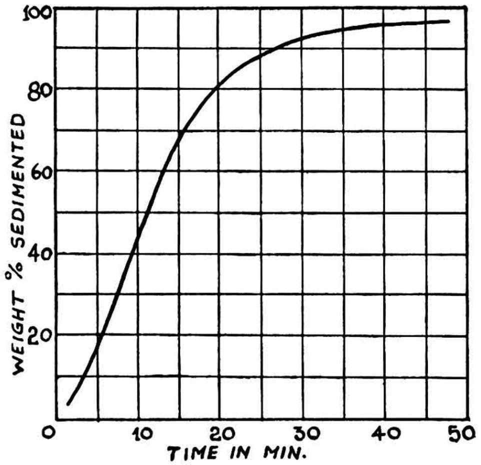

FIG. 3 — SEDIMENTATION CURVE FOR THE SAME SAMPLE WITH 0·75 PER CENT ALUMINIUM STEARATE.

re-dispersion and noting the rates of fall, the autocatalytic behaviour disappeared altogether as will be seen from the sedimentation curve shown in Fig. 4. Similar experiments were repeated, starting with fresh samples of "superfine" pigment made into paint with the usual mixture of raw and double-boiled linseed oil and 0·75 per cent aluminium stearate, and dispersing in white spirit. The only important difference was that after the formation of the paint and dispersing in white spirit, instead of setting the systems aside for 24 hr. before taking the observations, they were set aside for stated intervals of time, viz. 20 min., 1 hr., 6 hr., and 47 hr., and the rates of sedimentation were determined. These sedimentation curves, especially for the initial stages of settling, are shown in Fig. 5. It will be seen from the curves that the autocatalytic behaviour which characterizes this system tends to disappear gradually, as more and more time is allowed to elapse after the first formation of the paint. The sedimentation curve for the 6 hr. interval system actually shows only a slight inflexion. The sedimentation curves for the same system, determined after 24 hr. and 30 hr., showed the normal behaviour without disclosing autocatalysis or other novel feature. When the observations were made after a still longer time-interval, namely 47 hr., the rate of sedimentation again failed to conform to the normal type, and indicated the existence of

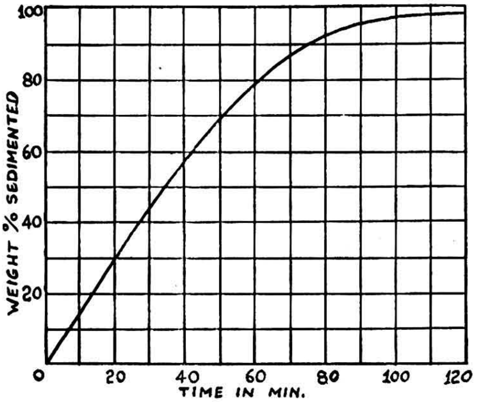

FIG. 4 — SEDIMENTATION CURVE FOR THE SAME SYSTEM AS IN FIG. 3, BUT 24 HR. AFTER THE PREPARATION OF THE PAINT.

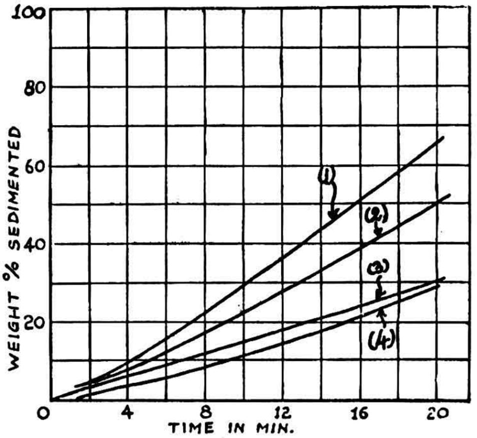

FIG. 5 — SEDIMENTATION CURVES FOR THE SAME SYSTEM AS IN FIG. 3, BUT AFTER THE PREPARATION OF THE PAINT: (1) 20 MIN.; (2) 1 HR.; (3) 6 HR.; (4) 47 HR.

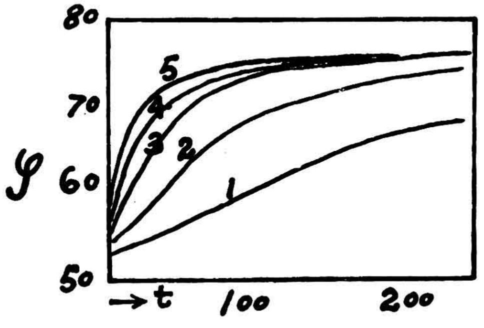

FIG. 6 — SHOWS COAGULATION VELOCITY CURVES OF Al_2O_3 SOL BY KCl SOLUTION.

certain complicating factors arising from the precipitation of gels of lead soap, which could be distinctly noticed as thin flakes floating in the supernatant white spirit and almost forming a layer on the surface of the sedimented particles of red lead. In this connection it may be recalled that in the preparation of double-boiled linseed oil, sulphates, borates, oxides and other salts of lead, manganese, cobalt, cerium, etc. (driers), are added to the oil before heating. These bases form soaps by reaction with the fatty acids of the oil. These soaps, especially of lead, get separated out as gels either on standing or on dilution with thinners such as white spirit or turpentine. The disturbing effect of the flakes of gel on the normal course of sedimentation of the pigment particles so as to make it deviate from Stokes' law is, therefore, quite obvious and explains the type of curve obtained in the case of the system set aside for 47 hr., referred to above.

Discussion

The curious nature of the sedimentation curves described above, which characterizes only the " superfine " red lead sample, seems difficult of explanation. The extreme fineness of the particle-size of this pigment, on careful consideration, appears to provide the necessary clue to a proper understanding of all the observed facts.

It is well-known that autocatalysis is a common feature in the " slow " coagulation of colloids. With increasing concentration of the coagulating electrolyte and the consequent change to " rapid " coagulation, the autocatalysis also gradually vanishes. Freundlich mentions a number of examples of autocatalytic behaviour, viz. the coagulation of aluminium oxide sol by potassium chloride solution, the sedimentation of kaolin and barium sulphate suspensions, etc.[2] Figs. 6 and 7 (reproduced from Freundlich) clearly show the effects of increasing the concentration of the coagulating electrolyte on the coagulation velocity of aluminium oxide sol and on the rate of sedimentation of barium sulphate. At low concentrations of the coagulator, the curves are distinctly autocatalytic, but as the concentration is gradually increased, the initial S shape of the curves also likewise tends to vanish and when the coagulator-concentration is made sufficiently high, the curves tend to assume a straight-line shape. It is explained that in such cases the original primary particles form coarser flakes or aggregates under the influence of the coagulator, and that they settle more quickly only when the time up to their formation, namely the " aggregation time " of Oden, has elapsed.

The foregoing examples indicate that in dealing with paint systems of the type provided by " superfine " red lead, one is confronted with " colloid phenomena ", the significance of which, in so far as it concerns the field of practical applications, needs careful elucidation, since it is not unlikely that it may lead to conclusions of far-reaching importance not easily apparent or understandable when considered in the orthodox manner of treatment.

In a previous paper[3] (Part II of the *Evaluation of Red Lead Pigments*), the author has drawn attention to the fact that the " standard specifications " are generally deficient in respect of the prescribed particle-size characteristics, and that one is liable to over-estimate the usefulness of a paint if he bases his judgement on the particle-size properties stated in the specifications. The

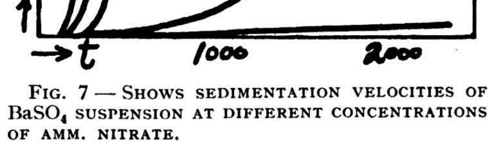

FIG. 7 — SHOWS SEDIMENTATION VELOCITIES OF $BaSO_4$ SUSPENSION AT DIFFERENT CONCENTRATIONS OF AMM. NITRATE.

defect of the standard specifications seems to lie in the rough upper limit which alone is usually stated, without mentioning anything about the lower limits of the particle size. It is stated, for example, that not more than 1 per cent of the pigment should constitute particles retained on a 325-mesh sieve (44 μ). This specification merely helps in ensuring that the pigment does not contain coarse particles of dirt, grit or other foreign matter. As will be seen from the particle-size data presented by the author[1,4], none of the 15 samples examined indicates the presence of particles approaching 44 microns diameter and almost all of them are confined to the region below 14-16 microns diameter. While the conditions laid down in the specifications appear to be fulfilled in the case of *all* these pigments, it must be noted that the different samples possess properties widely varying in their practical usefulness such as covering power, non-setting nature, protecting power against corrosion, etc.

The importance and need for specifying a *lower* limit too becomes obvious from the significant behaviour shown by the " superfine " sample. Of all the samples, this one appears to be almost the best in respect of particle fineness, peak size, specific surface, dry bulk, oil-absorption, density, etc. One is, therefore, apt to consider this to be the most suitable and satisfactory pigment. In fact there is an almost universal over-emphasis on the particle-fineness desired, with the result that it is considered by most people that the finer the pigment, the more it is to be preferred. Actual exposure tests, on the contrary, have shown that this argument is only of limited validity. Appearance of rust on test panels painted with " superfine " red lead occurred much earlier than in the case of panels painted with a comparatively coarse pigment. Thin layers of small pigment particles are more readily affected by light than tinctorially equivalent layers of large particles[5]. For this reason alone, there appears to be adequate justification for fixing or specifying the " lower " limit of particle size.

A second consideration of far-reaching significance arises from the observations of autocatalysis recorded in this paper. Normally, the use of dispersing agents of the type of aluminium stearate may be advocated to counteract the " setting " tendency of paints to procure certain obvious advantages in the preparation and storage of the so-called " non-setting " red lead paints. When this procedure is adopted in the case of the " superfine " type of samples, the effect will be to transform the paint into a near " colloidal " or actual colloidal system with all its logical consequences.

Ordinarily, the " dispersion stability " of these systems can be gauged from determinations of rates of sedimentation, under conditions of extreme dilution, by an inert solvent such as white spirit. In the case of paints comprising of near-colloidal systems, the inert diluent is no longer inert, but acts as a coagulator causing flocculation or aggregation. This is shown by the autocatalytic type of sedimentation curves obtained in " superfine " red lead plus aluminium stearate system. When the experimental method adopted, which is supposed to be valid only for systems consisting of particles freely falling in an infinite, inert medium in accordance to Stokes' law, is thus vitiated by flocculation or aggregate formation, it becomes difficult to draw quantitative conclusions as regards the particle-size characteristics.

Secondly, in the practical field as well, the presence of colloidal systems, easily liable to flocculation under actual conditions of application of the paints, very much detracts from the usefulness of the paint under question, as far as rust-prevention, hiding power, etc., are concerned. The " superfine " red lead paint, when slightly thinned and applied on the surface which it is desired to protect, will " coagulate " in the same manner as happens when it is suddenly diluted with white spirit and shaken in the sedimentation tube (as described in this paper). This will lead to segregation of the pigment particles in streaks or spots or in other irregular manner, causing a number of lacunae, devoid of the pigment layer, to appear on the painted surface. These " open " unprotected spaces will be the first to be attacked by agencies causing corrosion.

It will be seen from what has been stated above that the "quality" of pigments cannot be increased indefinitely by increasing the " fineness " of particles by continued or improved techniques of grinding or other means. Incidentally, the results laid down in this paper lend weight to the author's supposition stated in previous papers that " superfine " red lead is nothing but " orange lead " or " orange mineral " of very fine particle size, no doubt, but also, at the same time, of doubtful usefulness. The possibility and the easy scope for the formation of these paints into near-colloidal systems will be easily understood if it is remembered that " orange mineral " is made not from massicot but from white lead " tailings " which form as a froth in the grinding and levigating operations, and is, hence, of an extremely soft, light and fine texture.

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Evaluation of Red Lead Pigments: Part IV — Improvement of Dispersion Stability of the Paints

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AN assessment of the hard-setting tendency of red lead paints can be made normally by tests conducted in the storage container after different periods of storage. But this procedure involves long waiting before an opinion can be pronounced on the samples under test. It was pointed out in the previous papers[1] how the setting quality of a paint can be estimated by an examination of the dispersion stability. Measurement of the latter could be done by determining the rates of sedimentation of the paint particles under conditions aggravating the flocculation tendency brought about by extreme dilution with an inert solvent, namely white spirit in this case. The resistance of the pigment particles to sedimentation under these conditions gives an idea of the dispersion stability. Advantage was taken of this procedure in setting studies of numerous samples of red lead. A similar procedure was reported to have been adopted at the Paint Research Station, Teddington, in the evaluation of other pigments. The hypothetical particle-size distribution having the same rate of sedimentation as the pigment particles settling down under conditions of extreme dilution, as in the case of these experiments, would give a very useful picture of the pigment quality. In experiments previously reported (loc. cit.), the particle-size distributions in different types of red lead, both as such and also when the paint was sought to be improved by the incorporation of a small percentage of aluminium stearate as a dispersion agent, indicated the great improvement conferred by aluminium stearate in respect of the natural setting tendency.

In this paper are reported the results of further studies involving the use of certain other dispersing agents. Protection of pigment particles, it has been pointed out, may arise either by the formation of an adherent envelope of long-chain polar molecules or by a cohesive envelope such as that produced by polymerized oils, not strongly polar in character and consequently not present as an absorbed layer on the pigment surface, but as a sort of coherent skin around the particles in the same manner as colloidal sols of gold are stabilized by gelatine and other protective agents. It will be seen that a careful examination of the size-distribution diagrams gives some insight into the mechanism of the anti-setting property conferred by different dispersing agents.

Experimental Results

All the experiments were conducted on the same sample of fairly satisfactory red lead pigment purchased from the market. In each experiment, 10 gm. of the pigment were formed into stiff paint with 3 c.c. of a mixture of raw and double-boiled linseed oil

(1 : 2 by volume), except where stated otherwise. The duration of mixing and the detailed procedure adopted in forming these paints were maintained the same throughout. The paint so formed was thinned to a volume of 300 c.c. using white spirit in the sedimentation tube (modified Kelly's tube) and the rates of sedimentation were determined by observing the fall of the meniscus in the manometric limb by means of a cathetometer. Calculation of the particle-size distributions was made by the graphical method employed by Svedberg.

The results obtained are shown in Figs. 1 to 10. The description of the paint system in the case of each experiment, the peak radius, the size range and the type of size distribution are indicated in Table I. There are different ways of expressing the particle size of heterogeneous or polydisperse materials by a single figure. The usual types of diameters referred to by statisticians are the mean particle diameter, d_1 or d_{me}, the median particle diameter, d_{med}, the Modal particle diameter, d_m, the length average, the mean volume-surface diameter, the weight mean diameter, the mean surface diameter, the mean volume diameter, etc. Of these, the most frequently used averages are the first three only. The first one, namely d_{me}, as its name implies, is only the arithmetical average diameter given by the expression

$$d_{me} = \frac{\Sigma nd}{\Sigma n}$$

The second, d_{med}, represents the diameter of a particle so chosen that the number of particles of larger diameter is the same as those of smaller diameter. The third, d_m, the Modal particle diameter, represents the

TABLE I

No.	Description of paint system	Peak radius microns	Range microns	Type of distribution	Ref. to Fig.
1	No dispersing agent	4·9	3-6	Slight left-skew	1
2	With 0·25% Al. stearate	4·7	3-6·2	Symmetrical	2
3	With 0·5% Al. stearate	3·6	2·5-6	Nearly symmetrical	3
4	With 0·75% Al. stearate	3·7	2·5-5·8	Symmetrical	4
5	With stand oil + raw linseed oil	4·25	3-6	do	5
6	With stand oil alone	3·6	2-5·6	do	6
7	Repetition of 6, with fresh pigment sample	3·6	2·8-6·5	Slight right-skew	7
8	With 2% plaster of Paris	4·3	3-6	Nearly symmetrical	8
9	With 5% plaster of Paris	3·7	2-6	Symmetrical	9
10	With oleic acid + raw linseed oil	5·3	3·2-7·5	do	10

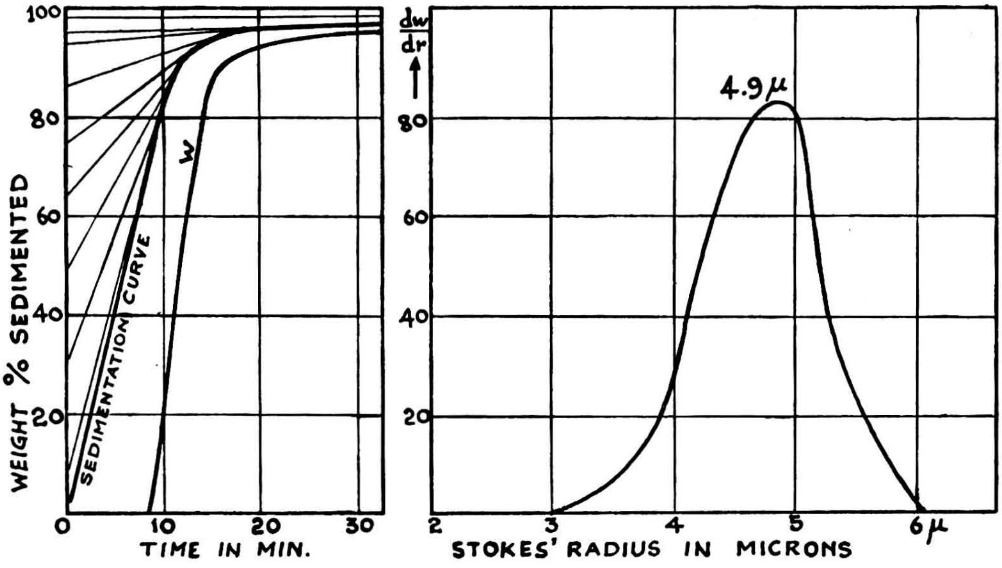

FIG. 1 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM USING SVEDBERG'S GRAPHICAL CALCULATION METHOD ; RED LEAD WITHOUT ANY DISPERSING AGENT.

most probable diameter for a particle in a mixture of many sizes, that is to say, that it is the diameter of the largest number of particles. It is considered that an idea of d_m will be the most significant in the studies reported in this paper. The value of d_m is given by the peak of the size-distribution diagram and in Table I, the peak radius is, therefore, the Modal particle radius.. This is also the same as the arithmetical mean particle diameter d_{me} or the median diameter d_{med}, in cases where the size-distribution is symmetrical.

The first 4 experiments reveal progressive improvement in respect of dispersion stability, brought about by increasing use of

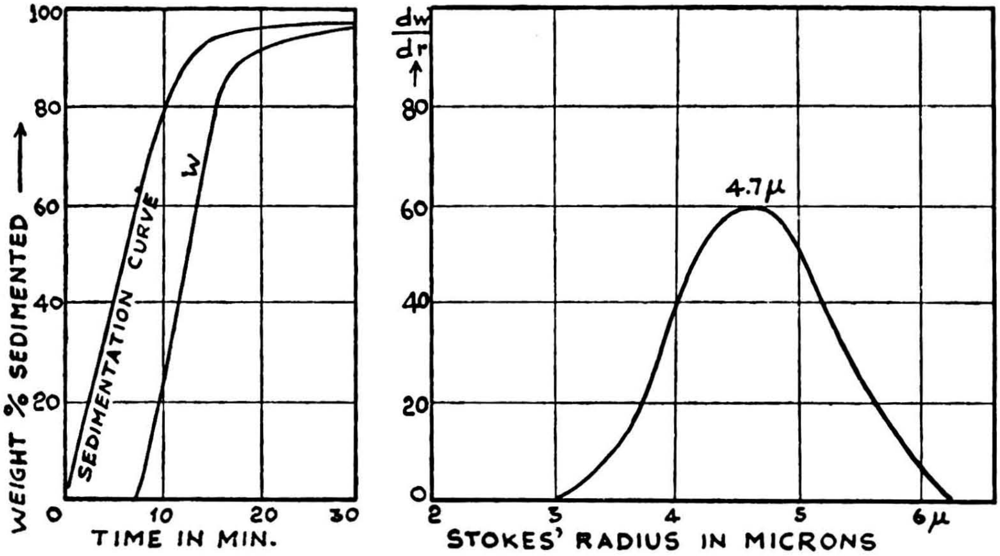

FIG. 2 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM ; RED LEAD + 0·25 PER CENT ALUMINIUM STEARATE.

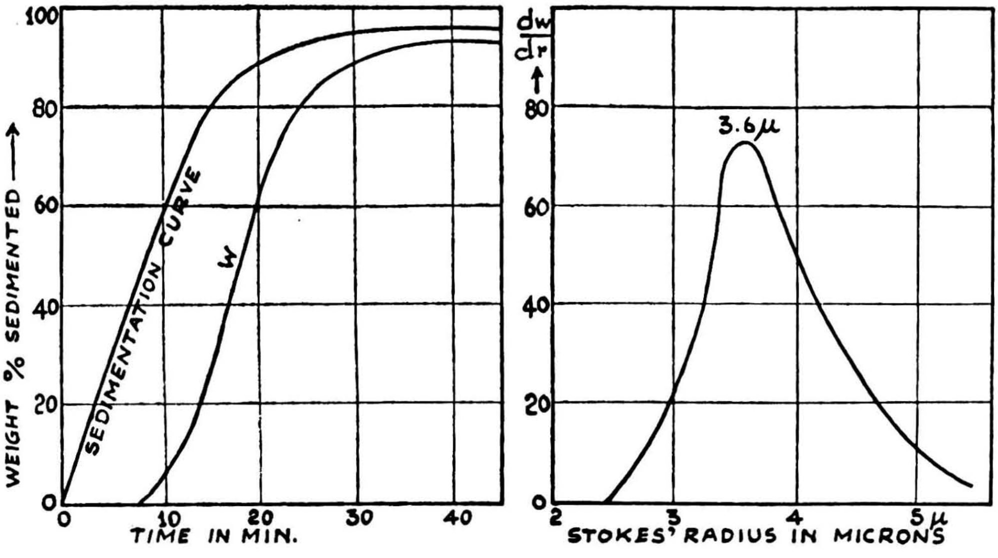

FIG. 3 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM ; RED LEAD + 0·5 PER CENT ALUMINIUM STEARATE.

aluminium stearate as a dispersing agent. This is clearly brought out by the progressive decrease of the peak radius given in column 3 and also by the narrowing down of the size range given in column 4 as one proceeds from experiments 1 to 4 (FIGS. 1 to 4). 0·5 per cent or 0·75 per cent of aluminium stearate on the weight of the pigment seems to be the optimum for securing the best dispersion stability.

In experiments 5, 6 and 7 (FIGS. 5 to 7), the effect of using stand oil, either in partial or total replacement of the linseed oil usually employed, is shown. In experiment 5, 10 gm. of the pigment under test were mixed with 1·5 c.c. of raw linseed oil and 1·5 c.c. of

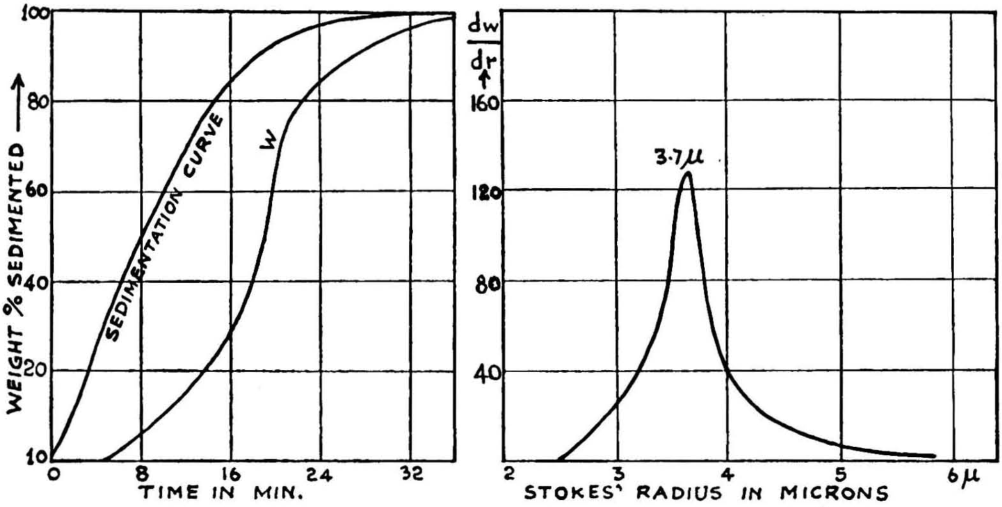

FIG. 4 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM ; RED LEAD + 0·75 PER CENT ALUMINIUM STEARATE.

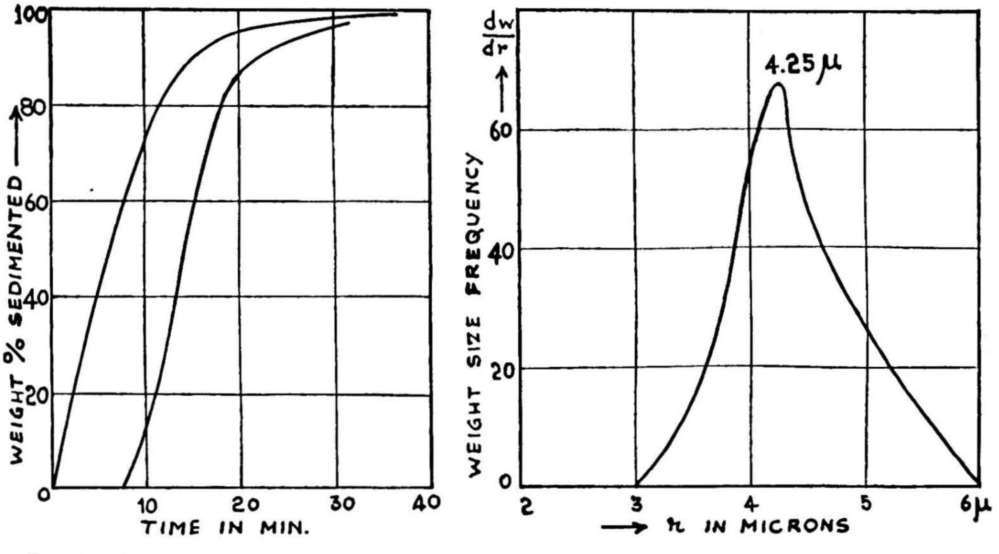

FIG. 5 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM ; SAME SAMPLE OF RED LEAD BUT HALF OF THE 30 PER CENT (BY VOLUME) OF RAW AND DOUBLE-BOILED LINSEED OIL REPLACED BY AN EQUAL VOLUME OF STAND OIL, THE OTHER HALF BEING RAW LINSEED OIL.

stand oil for forming the paint. In experiment 6, no linseed oil was employed, and instead, 3 c.c. of stand oil alone were used. Experiment 7 is a repetition of experiment 6 using 3 c.c. of stand oil with a fresh batch of pigment drawn from the same original stock. This experiment indicates the extent of reproducibility of the results. In experiments 8 and 9 (FIGS. 8 and 9), the paints were formed by using 2 per cent and 5 per cent, by weight, of plaster of Paris respectively. The usual mixture of raw and double-boiled linseed oils (1 : 2 by volume), employed in the case of experiments 1 to 4, was used in forming the paint. The plaster of Paris was pre-heated for 4 hr. at 125°C. before incorporation in the paint. In experiment 10, 10 gm. of the pigment was mixed with 1·5 c.c. of oleic acid and 1·5 c.c. of raw linseed oil. It will be seen from the distribu-

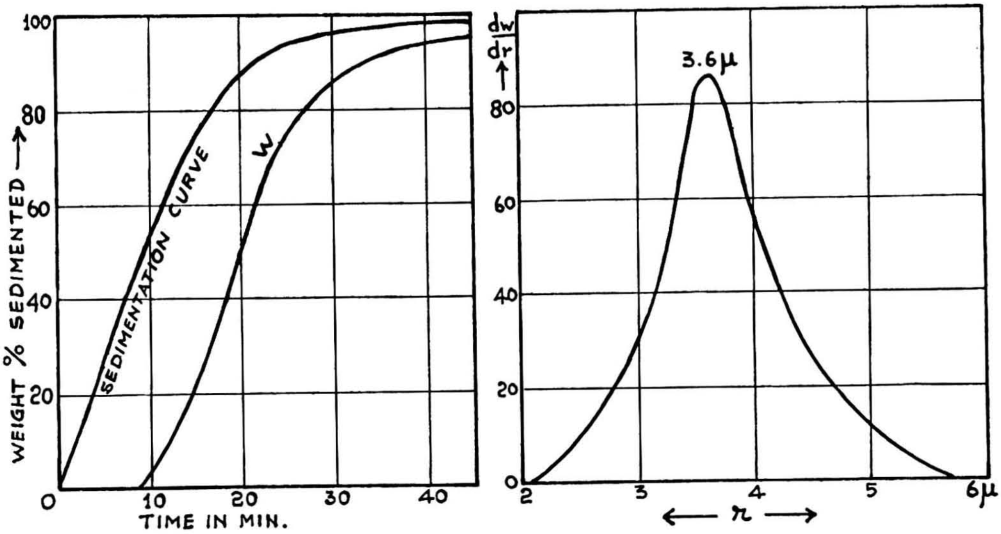

FIG. 6 — RED LEAD MADE INTO PAINT WITH AN EQUAL VOLUME OF STAND OIL IN TOTAL REPLACEMENT OF 30 PER CENT (BY VOLUME) OF LINSEED OIL.

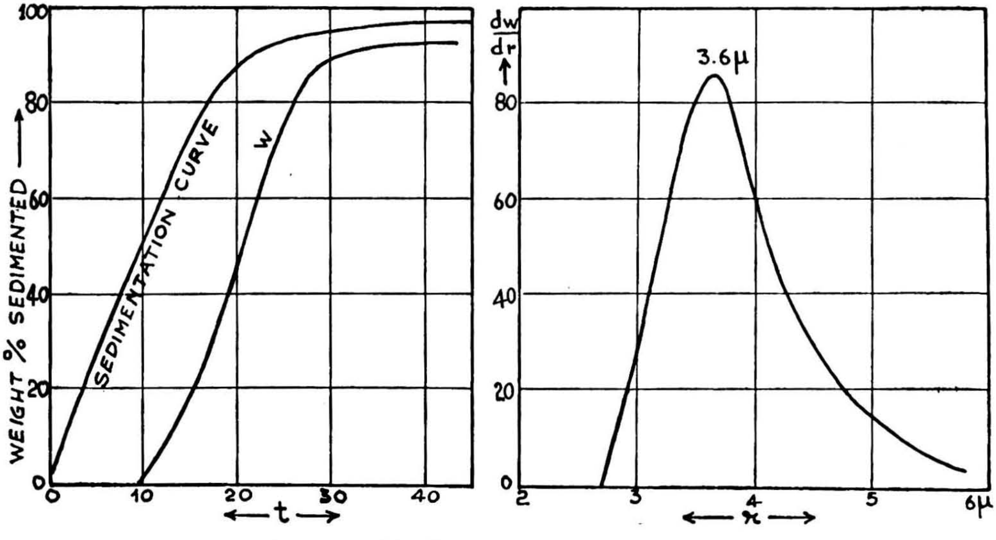

FIG. 7 — SAME AS IN FIG. 6, BUT WITH A FRESH BATCH OF PIGMENT.

tion diagram (FIG. 10) that far from acting as a dispersing agent, oleic acid has actually reduced the dispersion stability thereby leading to more rapid setting. The peak radius as well as the size range (cols. 3 and 4) are higher in this case than the values for experiment 1 where the pure test sample as such was employed without any dispersing agent.

It may be concluded from the results given above that if the red lead paints are formed by total replacement of the usual linseed oil mixture by stand oil, the quality of the paints in respect of setting tendency and dispersion stability is improved to the same extent as can be secured by the incorporation of an optimum percentage of aluminium stearate. The same may be

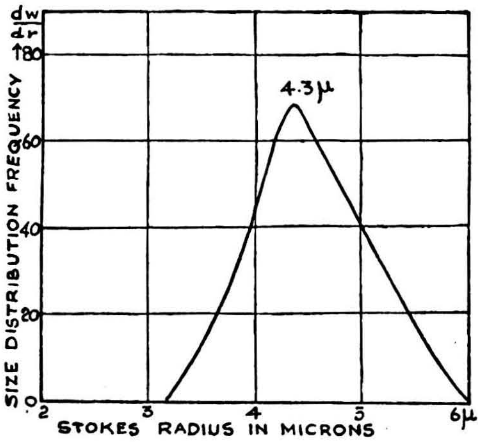

FIG. 8 — RED LEAD WITH 2 PER CENT PLASTER OF PARIS.

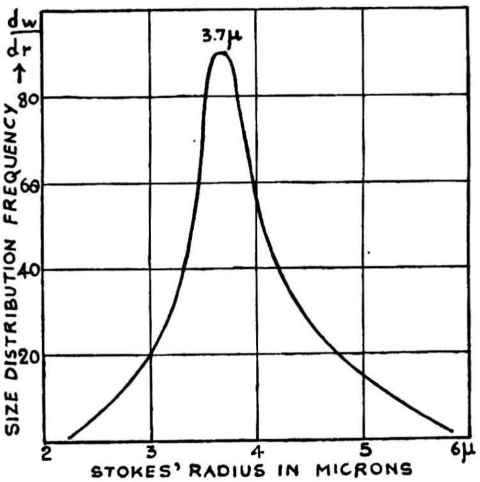

FIG. 9 — RED LEAD WITH 5 PER CENT PLASTER OF PARIS.

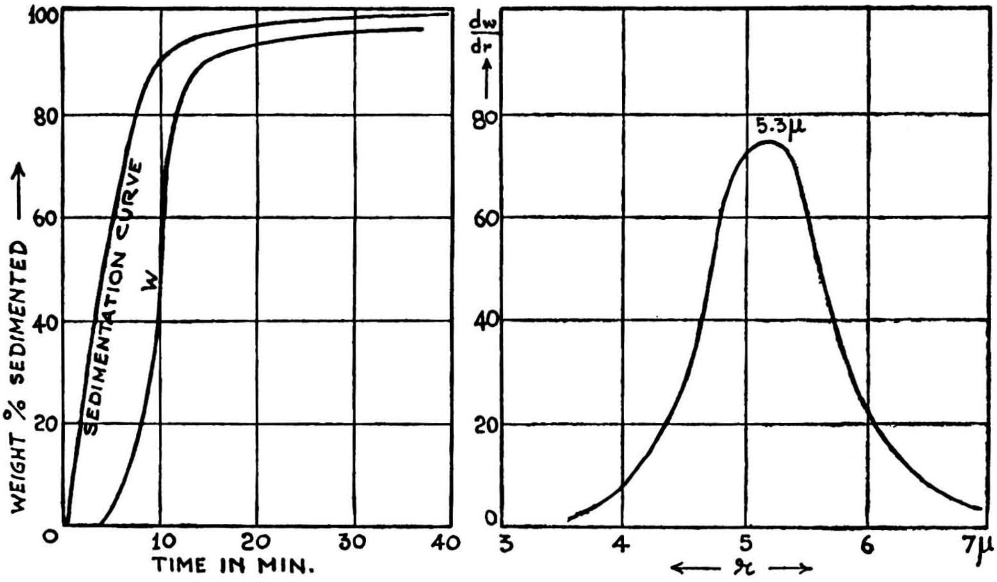

FIG. 10 — RED LEAD; HALF OF THE 30 PER CENT (BY VOLUME) OF LINSEED OIL IS SUBSTITUTED BY AN EQUAL VOLUME OF OLEIC ACID AND THE REMAINING HALF BEING RAW LINSEED OIL.

said about the use of plaster of Paris (5 per cent by weight) on the pigment sample. A few experiments carried out using talc, barytes and other substances as extenders or suspending agents gave unsatisfactory results. Although some of them appeared to slightly retard the setting, the results obtained were not considered satisfactory for extended investigations. As a matter of fact, barytes increased the *settling* tendency which is also an undesirable feature for any paint sample.

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Electrolytic Reduction of *ortho*-Nitrochlorobenzene to *meta*-Chlor-*p*-Aminophenol

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THE electro-reduction of *o*-nitrochlorobenzene in acid emulsion to *meta*-chlor-*p*-aminophenol does not appear so far to have been reported in literature. Indeed, no reference to the electro-synthesis of halogen-substituted aminophenols is found in literature and investigations even on the chemical methods of their preparation appear to be scarce. This accounts partly for the general lack of recognition of such compounds as possible dyestuff intermediates. Apart from their value as potential dye-intermediates, a study of these compounds would be of considerable interest in throwing light on the influence of such halogen substitution on the stabilization of the molecule against oxidative changes thereby conferring on them better keeping qualities. This is a factor of not little importance in making them suitable for employment as photographic developers in tropical climates.

m-Chlor-*p*-aminophenol was first decribed in a *D.R.P.*[1] as the product of reduction of 3-chlor-4-nitrophenol by iron and hydrochloric acid at 80°-100°C. and the m.p. was given as 160°C. It was also mentioned as a possible photographic developer. Later, Hurst and Thorpe[2], in the course of their work on the preparation of phenacetin by the reduction of *p*-nitrophenetole using tin and conc. hydrochloric acid, obtained a chloro-phenetidine and its acetyl derivative was de-ethylated by them in the presence of aluminium chloride to a product which was identified as *m*-chlor-*p*-acetyl-aminophenol (crystallized from chloroform, m.p. 121°C.). Hodgson and Kershaw[3] had also prepared this compound by the reduction of 3-chloro-4-nitrophenol in alkaline solution using sodium hyposulphite for the purpose (m.p. 160°C. ; N-acetyl derivative, m.p. 121°C.). More recently, Kiewict and Stephen[4] obtained the diacetyl derivative of the compound (3-chloro-4-acetamido-phenyl acetate, m.p., 127°C.) by reducing 3-chloro-4-nitrophenol in acetic anhydride with solid stannous chloride. In all these cases, the products have been obtained by devious means and the yields too have not been specified.

When *o*-nitrochlorobenzene is subjected to cathodic reduction in strongly acid media, according to accepted principles, the initially formed β-chloro-phenyl hydroxylamine undergoes isomerization in part to the corresponding aminophenol, viz. *m*-chlor-*p*-aminophenol and also reduction in part to *o*-chlor-aniline, the relative proportions of the two depending upon the conditions of the experiment. Previous experience in regard to the preparation of aminophenolic derivatives by electro-methods had shown that these conditions which determine the maximum formation of the aminophenolic body are mainly concerned with the employment of suitable cathodic material, either an alloy or simple metal, in conjunction with catalysts which suppress amine formation and simultaneously help the speed of isomerization of the hydroxylamino compound. Apart from these primary considerations, other factors like strength of acid, temperature of reduction, current density and depolarizer concentration also play a part in determining the nature and extent of the reduction. These latter factors, however, have been to some extent standardized, so that the experiments in the reduction of *o*-nitrochlorobenzene in acid medium were conducted only under the most favourable conditions determined previously, and were confined mainly to the study of the influence of variations in cathodic material and catalysts on the yields of the products.

Experimental

The experimental procedure involved in both the reduction and isolation of the products in this investigation is similar to that worked out in connection with that of *o*-nitro-toluene[5]. One exception, however, has been in the use of a solvent like xylene since only by its employment the depolarizer could be brought to the top of the catholyte, thereby facilitating agitation and subsequent emulsification which otherwise would have been hampered, owing to the denseness of the nitrochlorobenzene and its consequent tendency to settle to the bottom in spite of vigorous agitation of the mass. The use of a volatile solvent, especially in an open cell, made it necessary to maintain the temperature of reduction rather low (about 50°-55°C.), consistent with efficient working, so as to reduce loss. Moreover, it was found that neither a higher temperature nor the passage of current in excess of that required by theory helped to improve the yield of the products. Only the theoretical amount of current, viz. 64 amp./hr. was, therefore, employed for the reduction.

As regards cathodic material, monel and copper (pure and amalgamated), and lead were employed in this study. Copper was found to be the most suitable in providing the maximum yield of the chlor-aminophenol (29·1 per cent of theory) and lead for the chloraniline (34·6 per cent of theory), in conjunction with copper sulphate as catalyst. Amalgamation of copper did not improve the yield of the aminophenol, although the current efficiency rose from 54 per cent to 65 per cent. Apart from copper sulphate, other catalysts such as sulphates of mercury, calcium, zinc and ammonium and iodide of potassium were also tried. A low yield (10 per cent) of aminophenol resulted when mercuric sulphate was used in the presence of copper cathode, while the rest of the catalysts were to be found to be quite unsatisfactory. Calcium and ammonium sulphates were employed as a modification of the suggestion by Brand Strache[6] who found a mixture of the chlorides to be effective in the chemical reduction of nitrobenzene to phenyl-hydroxylamine. According to Khirkhgof[7] presence of iodides in the catholyte greatly increased the speed and efficiency of reduction of nitro compounds by acid electrolysis, but we are unable to confirm this in the present case.

The optimun conditions worked out and the results obtained so far for the preparation of *m*-chlor-*p*-aminophenol by acid electrolysis of *o*-nitrochlorobenzene are given below :

Conditions — Cathode : copper ; catholyte : 100 gm. of *o*-nitrochlorobenzene dissolved in 100 c.c. of xylene and kept in 400 c.c. of 30 per cent sulphuric acid ;

anode : lead ; anolyte : 50 c.c. of 30 per cent sulphuric acid ;
catalyst : 10 gm. of copper sulphate ;
temperature : 50°-55°C. ;
current strength : 20 amp./hr. ;
current density : 5·99 amp./ sq. dm. ;
total current passed : 64 amp. hr. (theoretical).

Results — Current efficiency : 54 per cent ; *o*-chlor-aniline : 24 per cent of theory ; *m*-chlor-*p*-aminophenol : 29·1 per cent of theory, m.p., 160°C. ; diacetyl derivative, double crystallized from alcohol and from chloroform, m.p., 131°C. ; reduces ammoniacal silver nitrate to a fine mirror of silver and colours ferric chloride violet.

Discussion

The amounts of chloraniline and chloraminophenol account for only a little over half (28·7 gm.) of the nitrochlorobenzene reduced. On titrimetric analysis of the mother liquor for the aminophenol remaining in solution, according to a modification of Brigham and Lukens' method[8], another 8·8 gm. of the used up depolarizer could be accounted for. Even after this there still remained about 30 per cent of the reduced nitro compound to be accounted in terms of converted products.

Although it is known that in a not too strongly acid medium a suspension of a nitro compound, when electrolytically reduced, yields, besides an amine and aminophenol, azo and azoxy benzenes and even benzidine[9], these side reactions are known to occur only slowly[10] and cannot, therefore, account for all the 30 per cent of the nitro compound reduced, nor can losses due to evaporation explain this. Since it is very probable that an equivalent amount of the hydroxylamino compound had been formed, a plausible explanation would be found in the assumption that it remained as such in solution, not having a chance, under the conditions of the experiment, either to be isomerized to the aminophenol owing to steric hindrance or to get reduced further to the amine. It, therefore, becomes necessary not only to study the course of the reaction under different conditions but also to identify the various products formed before any attempt is made to improve the yields of the aminophenol. With this end in view, a set of experiments is planned and details will be published separately.

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Miscibility of Castor Oil with Some Petroleum Products

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TESTS for the purity of castor oil such as the determination of the Frabot Number[1] and Benzine point[2] have been based on its partial miscibility with petroleum ether. The *British Pharmacopoeia*[3], for identification of castor oil, states that it " mixes completely with half its volume of light petroleum (B.P. 50° to 60°C.), and is only partially soluble in two volumes ". Atkins[4] studied critical solution temperatures of castor oil with petrols of different sources and observed that the presence of aromatics and naphthenes lowers the miscibility temperatures of the paraffins present. The values obtained by different workers for the critical solution temperature have differed mainly on account of the uncertain composition of the petroleum fractions used, and the wide boiling ranges of these fractions.

There is a general impression that castor oil and petroleum ether are immiscible at room temperatures usually prevalent (*c.* 25°-30°C.). This is not entirely correct since the two mix completely at certain volume ratios, irrespective of the type of the petroleum ether[5]. The present work was undertaken with a view to studying the behaviour of petroleum ether-castor oil binary mixtures over the the whole composition range. In addition, the miscibility of castor oil with kerosene, technical white oils and lubricating oils has also been studied.

Experimental

Materials Used — Castor oil : pure cold-drawn filtered oil with the following characteristics was used :

Sp. gr. at 29·5°/29·5°C. 0·9557
Ref. index at 29·2°C. 1·4780
Acid value, 1·9
Sap. value, 183·0
Hydroxyl value, 158·3

The solvents used in this study are given in Tables I and II.

Procedure — Mixtures of castor oil and the various solvents were made on volume basis and their turbidity temperatures were determined in the usual way. Mixtures which were clear and homogeneous at room temperature were cooled down gradually in a water bath and the temperature at which faint turbidity appeared was noted. Mixtures which were immiscible were heated gradually in the water bath until they were homogeneous and then were allowed to cool down slowly until faint turbidity appeared.

TABLE I

SOLVENT	QUALITY	BOILING RANGE, °C.	ANILINE POINT, °C.
Petroleum ether A	C.P.	98-118	53·7
Petroleum ether B	C.P.	87- 94	57·2
Petroleum ether C	C.P.	65- 70	58·1
Kerosene	Commercial	170-250	71·1

TABLE II

SOLVENT	QUALITY	SP. GR. AT 30°/30°C.	REF. INDEX AT 29·5°C.	VISCOSITY IN CENTIPOISES AT 35°C.	ANILINE POINT °C.
White oil I	Technical	0·8593	1·4727	14·59	97·5
White oil II	Technical	0·8653	1·4753	12·46	91·3
Shell BD_2 oil	Lubricating	...	...	...	82·2

TABLE III

CASTOR OIL VOL. %	PETROLEUM ETHER C (65°-70°C.)	
	Turbidity temperature, °C.	Clearance temperature, °C.
100·0	−12·0*	...
95·0	−14·0*	...
90·0	−15·0*	...
75·0	−14·0*	...
60·0	−11·0*	...
54·5	5·0*	6·0*
50·0	13·4-·2	13·6-·7
37·5	28·4-·2	28·3-·4
25·0	33·4	33·4-·5
12·5	36·2	36·3-·6
5·0	32·4-32·0	32·2-·4
1·25	20·6-·4	20·6-·8

*Approximate

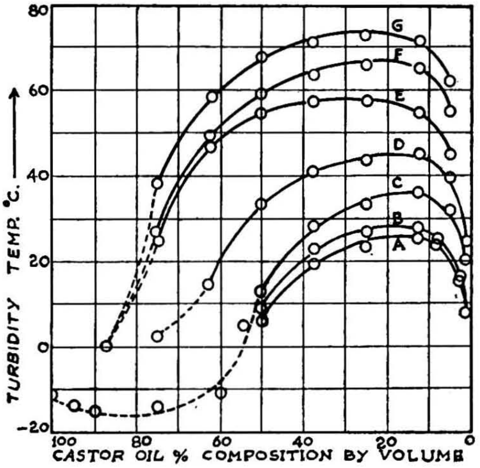

FIG. 1 — MISCIBILITY OF PETROLEUM PRODUCTS : CURVES A, B AND C REFER TO PETROLEUM ETHERS ; D : KEROSENE ; E : SHELL BD_2 LUBRICATING OIL ; F AND G : WHITE OIL I AND II.

In all the cases, the temperatures at which turbidity just disappeared were also noted.

Results — Although in most cases the appearance of turbidity was sharp and repeated checks gave fairly concordant values (difference not greater than 0·2°C.), there were instances where the turbidity was either difficult to mark or where no white turbidity appeared, but " liquid droplets " separated on cooling. In such cases, check readings differed by as much as 0·5°C.

Table III gives the experimental results for one binary system. The values for this and other systems are represented graphically in Fig. 1.

Discussion

The turbidity curves are remarkably similar. In all the cases, mixtures miscible at room temperature are obtained only in high concentrations of castor oil. As the proportion of solvent is increased, the mixtures become miscible only at elevated temperatures. They again become homogeneous at room temperature in very low concentrations of castor oil. The curves show that the 3 kinds of petroleum ethers, normally used in the laboratory, are completely miscible with castor oil at room temperature in equal proportions or in any larger proportion of castor oil. Although curve C only has been studied over the whole range, it will be seen that the other curves follow a course almost parallel to it over the partial range that has been studied. Castor oil is liable to be mixed either with technical white oils if it is being sold as a hair oil or with kerosene or lubricating oils if it is being sold for lubricating purposes. The curves show that these mineral oils, when mixed with castor oil up to 10-20 per cent of its volume, cannot be visually detected, since such mixtures remain perfectly clear at average room temperature.

The miscibility of castor oil with a petroleum product appears to be related to the Aniline point of the latter. In Fig. 1, the lower-most curve is for petroleum ether having an Aniline point of 53·7°C., the topmost curve is for white oil with an Aniline point of 97·5°C. The 3 petroleum ethers, A, B and C, were found to be almost free from unsaturated components and may be supposed to contain octanes, n-heptane and n-hexane respectively as their major components. Had they been of the highest purity, their Aniline points should have been 71·7°C., 69·7°C. and 68·6°C. respectively. These not only differ considerably from the values obtained for the 3 ethers used, but their order is also reversed. This indicates that even the so-called " chemically pure " petroleum ethers are not only mixtures of straight-chain paraffin isomers, but they may as well contain cyclo-paraffins and naphthenes. That the miscibility of castor oil depends on the Aniline point, i.e. the paraffinic nature of the solvent, was verified in the case of another lubricating oil with an Aniline point of about 105°C. This oil appeared to be much less miscible than the white oil with an Aniline point of 97·5°C.

Summary

Miscibility of castor oil with 3 types of petroleum ethers, kerosene, white oils and lubricating oils has been studied. It has been found that all of them are miscible at average room temperature with castor oil in high as well as in very low concentrations of the oil. The miscibility in the intermediate regions depends on the Aniline point of the petroleum product ; the lower the Aniline point, the higher is the miscibility.

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Chemical Examination of the Seeds of *Cucumis sativus* Linn.

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CUCUMIS sativus Linn. (Hindi : *Khira*) belongs to the natural order *Cucerbitaceae.* Its seed kernels are edible and are used in confectionery in India. In order to evaluate their nutritive value, a detailed chemical examination of the seed kernels has been undertaken, and the results obtained are recorded in this paper.

A few of the characteristics of the oil have been reported by Hooper[1]. The seed kernels contain about 42·5 per cent oil with the following fatty acid composition :

	%
Acids lower than palmitic	0·63
Palmitic acid	4·14
Stearic acid	16·42
Linoleic acid	40·11
Oleic acid	38·70

The kernels contain about 42 per cent proteins. The ash is rich in phosphates (0·62 per cent P_2O_5).

The unsaponifiable fraction of the oil has been crystallized from alcohol in white needles (m.p. 161°-162·5°C.). It gives a well-defined Libermann-Burchard colour test for sterols. Its acetate melts at 162°-163°C.

The oil belongs to the semi-drying class. In view of the fatty acid composition, high percentage of protein and appreciable quantity of phosphates, the seed kernels of *Cucumis sativus* should have appreciable food value.

Experimental

The kernels form 75 per cent of the seeds. The powdered kernels were extracted 4 times with petroleum ether (B.P. 40°-60°C.). After removing the solvent, 42·50 per cent of a clear, light-yellow, bland oil having the following characteristics was obtained :

		According to Hooper
Sp. gr.	0·9130 (40°C.)	0·9240 (15°C.)
Viscosity at 40°C.	27·32 centistokes	...
Ref. index at 25°C.	1·4605	...
Acid value	0·22	11·5
Sap. value	193·0	195·2-196·9
Iodine value (Wijs)	114·9	117·7-118·5
Acetyl value	3·1	...
Hehner value	94·86	94·4
Soluble fatty acids calculated as butyric acid, %	0·4	...
Hexabromide value	nil	...
Unsaponifiable matter %	0·91	...
Saturated fatty acids (modified Bertram method)[2]	20·76%	...

200 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 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.

TABLE I

FATTY ACIDS	QUANTITY %	NEUTRALIZATION VALUE	IODINE VALUE
Total	...	202·7	116·1
Soluble lead salt	79·49	204·4	138·18
Insoluble lead salt	20·51	201·3	8·1

TABLE II

Weight of esters distilled, 55·0 gm.

FRACTION	TEMPERATURE RANGE °C.	PRESSURE, mm.	WT. OF FRACTION gm.
L-1	150-180	6·0	1·48
L-2	180-194	6·0	6·89
L-3	186-190	4·0	9·03
L-4	190-194	4·0	7·41
L-5	194-198	4·0	8·55
L-6	198-210	4·0	8·73
L-7	210-219	4·0	2·47
L-8	Residue	...	9·70
			54·26

The acids (150 gm.) were dissolved in 95 per cent alcohol (750 c.c.), the solution boiled and mixed with a boiling solution of lead acetate (110 gm.) in 95 per cent alcohol (750 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°C. were crystallized from alcohol containing 1·5 per cent glacial acetic acid and recrystallized from ether at 0°C. 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 ether and alcohol mother liquors. The quantities of soluble and insoluble lead salts of fatty acids and some of their characteristics are given in Table I.

TABLE III

FRACTION		CORRECTED WT. gm.	IOD. VALUE	SAP. VALUE	MEAN MOL. WT.	WEIGHT OF ESTERS					
						Methyl caprylate gm.	Methyl caprate gm.	Methyl myristate gm.	Methyl palmitate gm.	Methyl linoleate gm.	Methyl oleate gm.
L-1		1·50	107·09	220·42	254·0	0·17	0·12	...	...	0·67	0·54
L-2		6·98	129·97	195·06	287·6	...	...	0·09	0·04	3·70	3·15
L-3		9·15	138·92	190·46	294·6	...	...	...	...	5·62	3·53
L-4		7·51	140·28	190·37	294·7	...	...	...	...	4·73	2·78
L-5		8·67	142·99	190·26	294·8	...	...	...	...	5·73	2·94
L-6		8·85	140·76	189·78	295·6	...	...	...	...	5·62	3·23
L-7		2·51	120·97	189·77	295·6	...	...	...	...	1·02	1·49
L-8		9·83	91·33	189·36	296·3	...	...	...	...	0·65	9·18
	Total	55·00	...	...	...	0·17	0·12	0·09	0·04	27·74	26·84

Soluble Lead-salt Fatty Acids

The soluble lead-salt fatty acids, on oxidation with potassium permanganate (modified Bertram method[2]), gave 1·0 per cent saturated acids.

The liquid acids were converted into methyl esters, and a known weight was fractionally distilled under reduced pressure[3]. The results obtained are given in Table II.

The loss in distillation (0·74 gm.) was 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. The results are given in Table III.

The acids from L-3 and L-5 were oxidized with dilute alkaline potassium permanganate when tetrahydroxy stearic acid (m.p. 172°-173°C.) and dihydroxy stearic acid (m.p. 130°-131°C.) were isolated from both the fractions. Fraction L-8 gave dihydroxy stearic acid by this treatment. The composition of the soluble lead-salt fatty acids is follows :

	%
Acids lower than palmitic	0·67
Palmitic acid	0·06
Linoleic acid	50·44
Oleic acid	48·83

The bromine addition derivatives of the soluble lead-salt fatty acids were prepared according to the method of Eibner and Muggenthalor[5] as described by Jamieson and Boughmann[6]. The results are as follows :

Soluble lead-salt acids taken for analysis	3·1563 gm.
Saturated acids from Bertram's method	0·03 ,,
Linoleic tetrabromide insoluble in petrol ether, m.p. 113°-114°C.	1·5841 ,,
Residue (tetrabromide, dibromide and saturated acids)	4·2352 ,,
Bromine content of the residue	43·33%
Linoleic tetrabromide in the residue	1·8292 gm.
Oleic dibromide in the residue	2·3760 ,,
Total tetrabromide found	3·4133 gm.
Linoleic acid equivalent to tetrabromide =	1·5930 or 50·47%
Oleic acid equivalent to dibromide =	1·5164 gm. or 48·04%

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

	%
Acids lower than palmitic	0·79
Palmitic acid	0·07
Linoleic acid	50·46
Oleic acid	48·68

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 IV.

TABLE IV

Weight of esters distilled, 22·0 gm.

FRACTION	TEMPERATURE RANGE 0°C.	PRESSURE mm.	WT. OF FRACTION gm.
S-1	150-175	6·0	5·60
S-2	175-183	6·0	5·65
S-3	176-183	5·0	4·93
S-4	Residue	...	4·86

The loss in distillation (0·96 gm.) was added proportionately to each fraction. The iodine values, saponification values and mean molecular weights of all the fractions were determined. The amounts of various acids in the different fractions were calculated according to the method of Jamieson and Boughmann[6] (TABLE V).

The acids from fraction S-1, on repeated crystallization from alcohol and acetone, gave a product melting from 60° to 67°C., showing it to be a mixture of palmitic acid (m.p. 63°C.) and stearic acid (m.p. 69°C.). The acids from fraction S-4, on crystallization from alcohol, gave stearic acid.

TABLE V

FRACTION	CORRECTED WT. gm.	IOD. VALUE	SAP. VALUE	MEAN MOL. WT.	WEIGHT OF ESTERS		
					Methyl palmitate gm.	Methyl stearate gm.	Unsaturated esters gm.
S-1	5·85	2·59	195·13	287·6	2·25	3·48	0·12
S-2	5·91	3·43	193·04	290·6	1·61	4·15	0·15
S-3	5·16	4·04	189·47	295·4	0·39	4·61	0·16
S-4	5·08	8·10	188·38	297·9	0·01	4·76	0·31
Total	22·00	...	...	...	4·26	17·00	0·74

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

	%
Palmitic acid	19·96
Stearic acid	80·04

From the above analysis, the total fatty acid constituents of the oil from the seed kernel of *Cucumis sativus* Linn. are :

	%
Acids lower than palmitic	0·63
Palmitic acid	4·14
Stearic acid	16·42
Linoleic acid	40·11
Oleic acid	38·70

The composition of the oil, therefore, is :

		%
Glycerides of	Acids lower than palmitic	0·63
	Palmitic acid	4·12
	Stearic acid	16·30
	Linoleic acid	39·62
	Oleic acid	38·42
	Unsaponifiable matter	0·91

Seed Kernel Cake

The cake obtained from the seed kernels, after complete removal of the petrol-ether soluble fraction, was analysed according to the method described in the *Official and Tentative Methods of Analysis*. The following results were obtained :

	%
Moisture	8·13
Proteins (N × 6·25)	72·53
Ash	9·7
Crude fibre	1·0
Carbohydrates (by difference)	8·64

The ash, on analysis, gave 11·17 per cent phosphate calculated as P_2O_5.

Acknowledgement

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Letters to the Editor

A NOTE ON PULSED EMISSION FROM OXIDE-COATED CATHODE

MECHANISM OF THERMIONIC EMISSION FROM oxide-coated cathode as also that of conduction through oxide coating had been a subject of controversy for a long time. The situation, however, became simpler when Fowler-Wilson[1] theory of semi-conductors based on statistical mechanics was applied to the problem. Electronic energy levels inside a semi-conductor (n-type) are shown in Fig. 1. With the values as shown in the figure, the expression for conductivity σ of the semi-conductor can be shown to be of the form :

$$\sigma=\sigma_0\ exp.\ (-eQ_{1/2kT}) \quad (1)$$

where e is electronic charge, k is Boltzmann's constant, T is the temperature of the cathode in °K, σ_0 is a constant directly proportional to electronic charge and mobility and is very slightly dependent on temperature. $Q_{1/2}$ is called the activation energy of the semi-conductor. The expression for saturated thermionic current i_m can be written as

$$i_m=\alpha\, AT^2\ \exp.\ [\,-e\,(Q_{1/2}+\phi)/kT\,] \quad (2)$$

where α is a constant slightly dependent on temperature, A is the usual constant of Dushmann's equation and is independent of temperature. This approach to the problem helped to explain many peculiarities observed in an oxide-coated cathode and could predict emission of the correct order of magnitude. In recent years, however, peculiar effects were observed in an oxide-coated cathode operated with pulsed voltage of microsecond duration on the anode. It was found that surprisingly large amount of peak current could be drawn from a cathode which, under condition of d.c. operation, gives a much lower magnitude of current. This effect aroused a lot of speculation among the workers and it has led to the suspicion that Fowler-Wilson theory of semi-conductor is perhaps not the correct approach to the problem, at least under pulsed operation. For example, Blewett[2] has suggested that the large emission observed is probably connected with some non-equilibrium condition inside the cathode and, therefore, statistical-mechanical approach, which is valid only under condition of equilibrium, may not be applicable to such a case. He and Hahn have, therefore, advocated a new approach based on mass motion. An interesting suggestion due to Coomes[3], however, does not require such a radical revision of idea. He has suggested that electrons which are thermally excited to conduction level during the period between the successive pulses are swept away by the pulse, thus causing a momentary flow of a large amount of current. This suggests that expression for conductivity of the oxide layer as also of thermionic emission current should be given by expressions of the type (1) and (2). Experimental data available to date are not, however, sufficient for an outright decision in favour of one or the other suggestion. In a recent communication Danforth and Goldwater[4] have given some interesting experimental data for pulses of microsecond duration. They found that log 1/R plotted

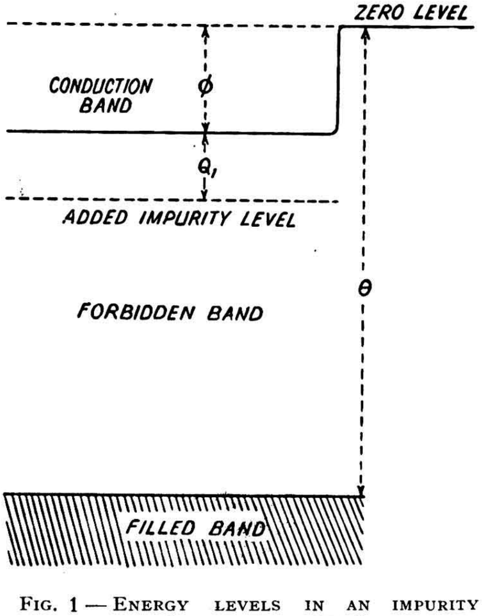

FIG. 1 — ENERGY LEVELS IN AN IMPURITY SEMI-CONDUCTOR (EXTRINSIC N-TYPE).

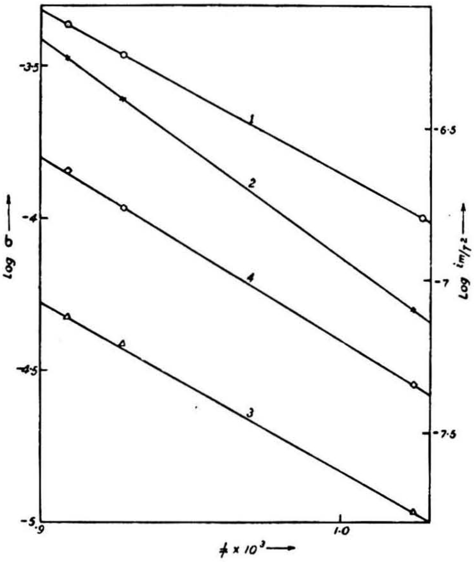

FIG. 2 — FIGURE SHOWING CONDUCTIVITY AS FUNCTION OF TEMPERATURES (CURVES 1, 2, 3 AND RICHARDSON PLOT OF EMISSION CURRENT (CURVE 4).

against 1/T gives a straight line whose slope, within experimental accuracy, lies near about the value of Q_1 obtained by other workers under condition of d.c. operation by using relation of the type (1) (R being the resistance of the cathode). In their experiment they used a probe embedded inside the oxide coating to determine R. An interesting method of measurement which avoids complications due to the embedded probe was used by Loosjes and Vink[5] to determine the characteristics of an oxide-coated cathode for pulses of duration 10^{-4} sec. They found that surprisingly large amount of potential drop exists across the oxide layer under such condition of operation. They have not, however, examined if the data obtained by them obey relations of the type (1) and (2). This has been done and is being presented in this note. It is found that there is strong evidence that the Fowler-Wilson theory of semi-conductor might be applicable in pulsed operation and, therefore, suggestion offered by Coomes deserves serious consideration.

Current voltage characteristics (*i-v*) of the cathode given by Loosjes and Vink show the following 3 distinct regions :

1. A very small linear region at the origin.
2. A curved region beyond (1).
3. A nearly linear region for high value of V.

Curve (1) in Fig. 2 has been drawn for the first region. Conductivities were calculated by drawing a tangent at the origin of the characteristic. It is seen that the curve is essentially linear with a slope of 1·05 volts. Curve (2) has been drawn for the second region mentioned above. Conductivities were calculated for $V = 20$ volts. Here also $\log \sigma - 1/T$ is found to be linear. Slope of the curve is about 1·4 volts. Curve (3) has been drawn for the third region mentioned above. The curve is linear with a slope of 1·1 volts. It is seen that the slope of $\log \sigma - 1/T$ curve for region (2) differs from the other two values. Conductivities calculated for this region from the values of *i* and *v* at point should, however, be interpreted carefully. Characteristics is curved in this region, which shows that a non-ohmic element in the cathode might be playing an important rôle in this region. It is well known that non-ohmic contacts give rise to a potential barrier having the property of rectification. Presence of a fairly thick barrier of this type in the core/oxide interface of an oxide-coated cathode has been definitely established by Fineman and Eisenstein[6], Wright[7] and Coomes[3]. Resistance of this barrier may be high compared to that of the semi-conductor. The linear relationship obtained for this region in curve (2) also indicates that the combined effect of a potential barrier at the core/oxide interface and the semi-conducting oxide layer is to give rise to a resistance having a higher activation energy. This is also expected from theoretical expression given by Mott[8] for the current flowing through a potential barrier of the type mentioned above. Therefore, leaving the value for the curved region out of consideration we can take 1·1 volts as the activation energy for the semi-conducting oxide layer. This does not agree well with the value of ·825 volt given by Nishibori and Kawamura[9] for d.c. operation, but agrees well with the value of $1{\cdot}2 \pm 1$ volt found by Mahlman[10]. With 1·1 volts as the activation energy of the semi-conductors, and hence $Q_1 = 2{\cdot}2$ volts, the impurity level must be about 2·2 volts below the bottom of the conduction band and, therefore, the energy separation between the top of the filled band and the bottom of the conduction band is at least 2·2 volts. Calculation of Wright[11] on the basis of the treatment of Mott and Gurney[12] for alkali halides gives for BaO a value of θ (FIG. 1) the energy separation between the zero level and the topmost level of the filled band as 2·6 volts. This shows that ϕ should have a value of ·4 or less. According to Wright, the maximum value that ϕ can have is ·6 for BaO, but it may be zero or or even negative if the bottom of the conduction band lies above the zero level. Supposing, therefore, that the value of ·4 is quite admissible, one finds that the maximum value of thermionic work function is

$$Q_{1/2}+\phi=1\cdot5 \text{ volts}$$

This value can be roughly checked with the help of the data given by Loosjes and Vink. From the values of plate voltage and thermionic current given, it is possible to determine i_m the maximum possible emission for the cathode at a particular temperature without Schottky effect. This is done by simply plotting log i against $\sqrt{\text{plate voltage}}$ and then finding the intercept on the log i axis obtained by producing the linear portion of the curve. The following data were thus obtained:

Temp.	*log* i_m
976°K	−1·35
1078°K	−·70
1100°K	−·56

Curve was drawn with log i_m/T^2 as ordinate and 1/T as the abscissa and curve (4) in Fig. 2 was obtained. This is a straight line as required by equation (2). From the slope of the line the value of thermionic work functions is found to be 1·19 volts. This is within the limit set by previous consideration. This value can be compared with the values of 1-1·5 volts obtained with d.c. operating voltage.

It is also possible to determine the value of "α A" from the intercept made on the log i_m/T^2 axis by curve (4). The value thus obtained roughly is ·08. This is much larger than the usual value of ·0013-·000013 found for oxide-coated cathode under d.c. operating conditions, but is only slightly greater than the value of ·03 found by Mahlman[10]. It is thus seen that the larger emission obtained under pulsed condition is due to a large increase in the factor "α A" and not at all due to any lowering of work function. Indeed, there are evidences which show that for the same cathode the work function is probably larger under condition of pulsed operation.

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Supplement

AROMATIC PLANTS OF INDIA

PART XI

BY

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FAMILY

42. Saxifragaceae
43. Crassulaceae
44. Hamamelidaceae
45. Combretaceae

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AROMATIC PLANTS OF INDIA

Family XLII — SAXIFRAGACEAE

(Saxifrage Family)

THIS family comprises 90 genera and 750 cosmopolitan, chiefly temperate species of trees, shrubs or herbs. Its members are distinguished usually by the free bicarpellary ovary and by having as many or twice as many stamens as petals.

The family is of little importance from the economic point of view. Some species are cultivated in gardens, and among them may be mentioned *Hydrangea hortensia* Sieb. (garden hydrangea, Chinese guelder rose), *Philadelphus coronarius* Linn. (syringa), and species of *Saxifraga*, *Deutzia*, *Escallonia*, etc. Several species of *Ribes* yield edible fruits. The red currant (*Ribes rubrum* Linn.), black currant (*R. nigrum* Linn.), and the gooseberry (*R. grossularia* Linn.) are found wild in India and elsewhere, but are not cultivated in India for their fruits, as is done in some foreign countries.

The leaves of some species of *Ribes*, such as *R. nigrum*, *R. rubrum*, and *R. grossularia*, contain hydrocyanic acid. Tannins have been reported from the roots of some species of *Saxifraga*, saponins from the leaves of some species of *Saxifraga* and *Philadelphus*, and glycosides from some species of *Hydrangea* and *Dichroa*.

Essential oils have been isolated from the buds of *Ribes nigrum* and flowers of *Philadelphus coronarius*. The leaves and bark of *Itea rosmarinifolia* Poir. of Cochin-China contain an essential oil, but the Indian *I. macrophylla* Wall., *I. chinensis* Hook. & Arn., and *I. nutans* Royle have not been examined so far ; the Indian species have glandular-toothed leaves. The rhizomes (*pakhan-bed*) of *Bergenia ligulata* (Wall.) Engl. (*Saxifraga ligulata* Wall.) of the Himalayas are said to contain an odorous principle (Dymock, Warden & Hooper, 1890. *Pharmacographia Indica*, **1** : 585) ; they are used in indigenous medicine as a tonic, in dysentery, and as a remedy for boils. The root bark of *basak* (*Dichroa febrifuga* Lour.) of the E. Himalayas is faintly aromatic (Dymock, Warden & Hooper, *loc. cit.* : p. 588), and is used in indigenous medicine as a febrifuge. *Deutzia corymbosa* R. Br., a shrub of the temperate Himalayas, bears corymbose terminal panicles of white, fragrant flowers $\frac{1}{2}$ to $\frac{3}{4}$ in. in diameter. *D. staminea* R. Br., another shrub of the temperate W. Himalayas, bears panicles of white, fragrant flowers $\frac{1}{3}$ to $\frac{1}{2}$ in. in diameter.

P-cymene is the only constituent which has been identified in the black currant bud oil.

1. PHILADELPHUS (Riv.) Linn.

(The name of a plant mentioned by Aristotle, which has not been identified by modern botanists ; from the Greek word *Philadelphon* — a sweet-flowering shrub.)

This genus comprises 20 species of shrubs of wide distribution in the north temperate regions. It is distinguished by the numerous stamens and inferior ovary. Several species with strongly fragrant flowers are cultivated as ornamental shrubs under the names of mock orange and syringa. In India it is represented by two species.

P. tomentosus Wall. (*P. coronarius* Linn., var. *tomentosus* C. B. Clarke), which is found in the temperate Himalayas, bears white, very fragrant, tetramerous flowers up to 1 in. in diameter.

Philadelphus coronarius Linn.

Fl. Brit. Ind., **II**, 407 (without description).

(Mock Orange, Syringa*)

This is an erect shrub with white, orange-scented flowers about 1 in. in diameter. According to the *Flora of British India*, this plant is not found in India, but Collet (1921. *Flora Simlensis* : p. 180) and some other authors report it from Narkunda and other areas in the Himalayas from Kishtwar to Sikkim at altitudes of 6,000 to 10,000 ft. It is also cultivated in some gardens in the hills. Parker (1924. *Forest Flora for the*

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* The common English name syringa should not be confused with the botanical name *Syringa*, which is a genus belonging to the family Oleaceae and includes the lilac *Syringa vulgaris* Linn.

FIG. 42 — *Ribes nigrum* Linn.

Punjab with Hazara and Delhi : p. 237), who calls it a European plant, states that this differs from *P. tomentosus* Wall. in having a septicidal (not loculicidal) capsule.

Treff, Ritter & Wittrisch (1926. *J. prakt. Chem.*, **113** : 355) extracted the flowers of various species of *Philadelphus* with petroleum ether. They reported 0·237 per cent. of a concrete and 0·124 per cent. (52·2 per cent. of the concrete) of an absolute. The absolute on steam distillation yielded 0·006 per cent. of a yellowish distillate having a powerful and characteristic odour. The distillate had the following characteristics : sp. gr. $^{15°}$ 0·947, [α] 0°, acid val. 28, ester val. 73, and ester val. after acetylation 224. It probably contained some methyl anthranilate. Farmiloe (1929. *Perfum. essent. Oil Rec.*, **20** : 321), who investigated the flowers of *P. coronarius*, obtained 0·25 per cent. of a petroleum ether concrete, which yielded 38·2 per cent. of an absolute. The odour of the absolute approximated to that of jonquil and narcissus.

Igolen (1938. *Parfums de Fr.*, **16** : 92) recovered 0·144 to 0·179 per cent. of a reddish-brown petroleum ether concrete of hard consistency. This concrete gave 25 to 27·2 per cent. of a reddish-brown absolute of thick consistency, having a penetrating, fruity odour suggestive of the syringa flowers. From the absolute, Igolen obtained 9 per cent. of a steam distillate, which had the following characteristics : sp. gr. $n^{15°}$ 0·912, $[\alpha]$ $+3{\cdot}5°$, $n^{20°}$ 1·4668, acid val. 25·2, and ester val. 95·2.

The natural perfume is not extracted, or, if so, only in insignificant quantities. The synthetic perfume of this name is usually composed of terpineol, linalool, hydroxycitronellal, and methyl anthranilate.

2. RIBES Linn.

(From the Arabic name *ribas*, meaning a plant with an acid juice.)

This genus comprises about 60 species of shrubs out of which 8 are represented in India. Its representatives have small, racemose, variously coloured flowers with 4 or 5 scale-like petals, 4 or 5 stamens, 2 styles, and an inferior ovary. The fruit is a globose or ovoid berry. It includes the currants and the gooseberry which have been referred to in the account of the family.

Hydrocyanic acid and essential oils have been reported from some species.

Several Indian species are covered with resinous glands or glandular hairs. For example, the twigs of *R. orientale* Desf. of the W. Himalayas are glandular and sticky, and its pubescent leaves have coarse sticky-glandular hairs. *R. nigrum* Linn. has been investigated for its essential oil, and is dealt with below.

Ribes nigrum Linn.

Fl. Brit. Ind., **II**, 411.

(Black Currant, *Muradh*)

This is an erect aromatic shrub 6 ft. high. The young twigs are hairy and clothed with sessile yellow resinous glands which are also found on the petioles, lower surface of the leaves, inflorescence, and calyx. The heart-shaped 3- to 5-lobed leaves are scented. The flowers are greenish, on 2 to 3 in. long racemes which arise from leaf-producing buds. The berries are globose, $\frac{1}{3}$ in. in diameter, black, with a strong aromatic smell and taste. It is found in the W. Himalayas, from Kunawar to Kashmir, at altitudes of 7,000 to 12,000 ft.

The plant is often cultivated in Europe for its edible fruits. The Indian fruits from wild plants are said to be similar to the cultivated black currants.

The buds yield 0·75 per cent. of a pale, greenish oil, having the odour of cymene. Its characteristics are : sp. gr.$^{15°}$ 0·8741, $n^{20°}$ 1·4858, $[\alpha]$ +2·3°, and ester val. 5·6. The oil is soluble in 6·5 parts of 90 per cent. alcohol, with slight cloudiness, but is clear in 10 volumes (Schimmel & Co., 1907, Apr. *Rep.* : p. 106).

The leaves are said to contain an essential oil, quinic acid, and a very active oxydase (Huchard, 1909. *Pharm. J.*, **82** : 528).

Family XLIII — CRASSULACEAE

(Orpine and Houseleek Family)

This is a small family of succulent herbs or undershrubs, with free calyx, persistent petals, and fruit consisting of distinct carpels. It comprises 25 genera and 450 cosmopolitan species found chiefly in S. Africa.

A large number of plants of this family have smooth succulent leaves, and are able to grow in places where water is at times scarce. Life plant [*Bryophyllum pinnatum* (Lam.) Kurz, syn. *B. calycinum* Salisb.] is remarkable for forming new plants at the crenatures of the leaves when these are laid on damp ground. A few species are cultivated for ornamental purposes, for example, *Rochea coccinea* DC. of the Cape of Good Hope. Some species of *Cotyledon*, especially *C. ventricosa* Burm. *f.* and *C. wallichii* Harv. of S. Africa, are said to be concerned in the induction of a disease called " krimpsiekte " or cerebro-spinal meningitis, especially in small stock.

The only species reported to contain an essential oil is *Sedum telephium* Linn. (orpine) of middle Europe. Its leaves, stems, and roots contain an amorphous glycoside, which breaks up through the agency of emulsin into glucose and geraniol-smelling essential oil (Bridel, 1922. *J. Pharm. Chim., Paris,* **26** (7) : 289 ; 1922. *C.R. Acad. Sci., Paris,* **174** : 186 ; 1923. *Ber. Schimmel u. Co. Lpz.* : p. 213).

Family XLIV — HAMAMELIDACEAE

(Witch-hazel Family)

This family comprises about 20 genera and 50 species of shrubs and trees which are chiefly found in the subtropical region.

The family, though a small one, is important from the economic point of view. Some species, such as those belonging to the genera *Altingia*, *Bucklandia*, and *Liquidambar*, yield valuable timbers. Others, such as those belonging to the genera *Hamamelis*, *Corylopsis*, and *Distylium*, are grown as ornamental shrubs. The witch-hazel or hamamelis extract of trade, which is used in medicine as an astringent and to stop bleeding, is obtained from the leaves of witch hazel (*Hamamelis virginiana* Linn.) of N. America. The tough and flexible twigs of *Parrotia jacquemontiana* Decne. of N.-W. Himalayas are largely used in wickerwork in the same way as the willows, and also twisted into thick ropes, often 300 ft. long, which serve for crude suspension bridges across the hilly streams. The wood of this species is esteemed for walking sticks, tent pegs, rice pestles, etc.

The balsam storax or styrax, which is obtained from some foreign species of *Liquidambar*, is of great importance in perfumery, and is also valued in medicine as a parasiticide in scabies and other parasitic skin affections. The Indian *Altingia excelsa* Noronha also yields a balsam, rasamala, which, though differing from the genuine storax in some respects, may be used as a substitute.

The leaves and branches of *Hamamelis virginiana* on steam distillation yield a soft, sticky, green oil of strong odour. When this is distilled with steam, an yellowish oil of a powerful odour is obtained. The bulk of this oil consists of a sesquiterpene, an alcohol (7 per cent.), a small quantity of ester, and a wax (about 70 per cent.).

Among the constituents of essential oils isolated from members of the family may be mentioned : β-phellandrene and other sesquiterpenes, dipentene, α- and β-pinene, camphene, borneol, styrene (styrol, phenylethylene), cinnamic alcohol, cinnamic acid, cinnamyl cinnamate, coumarin, vanillin, benzaldehyde, cinnamaldehyde, and unidentified aldehydes and ketones.

1. ALTINGIA Noronha

This genus comprises three arboreous species which are found in Assam, S. China, and southwards. In India it is represented by only one species, *A. excelsa* Noronha, which is the source of a product called Burmese storax. Tonkin storax is derived from *A. gracilipes* Hemsl.

Altingia excelsa Noronha

Fl. Brit. Ind., **II**, 429.

(Rasamala, *Jutuli*)

This is a tall aromatic tree found in the forests of Assam and Bhutan. It is also found in Tonkin and thence southwards through Burma to Sumatra and W. Java.

The wood is extensively used by planters in Assam for building purposes. The heartwood has a sour odour when first exposed, but this passes off as the timber seasons.

The tree yields a resin which is called rasamala. The name rasamala has been applied not only to the product from this tree, but also to the Oriental storax from *Liquidambar orientalis* Mill. from Asia Minor and several other balsams with a similar odour.

The resin does not appear to be collected in Assam. According to Burkill (1935. *Dictionary of the Economic Products of the Malay Peninsula*, **1**, 118), " In the Levant, trees of *Liquidambar orientale* (*sic*) are exploited for liquid storax, first by stripping off the outer bark, then by scraping off the inner bark, which is packed in horse-hair bags and pressed, soaked in hot water, and pressed again, the resin being extracted by the two pressings ; or the bark is boiled and then pressed. The bark is afterwards dried and used for fumigations. In the East there is no such process, but the bark is slashed, and the scanty clear resin collected ; or fire may be applied to a hole cut in the tree and discoloured resin obtained. Damage to the tree is, in any case, necessary before the resin is got. The resin which runs from clean wounds is in the form of clear tears, which adhere but slightly to the bark. It exudes very slowly. No resin, apparently, is collected in Assam ; but there was a collecting-industry in Burma, and there is one still in Annam. It may be collected in Sumatra and Java, but scarcely comes into the market where what is sold as rasamala is generally the imported storax of *Liquidambar orientalis*.

FIG. 43 — *Altingia excelsa* Noronha

The resin of *A. gracilipes* is collected similarly in Tonkin."

The resin differs considerably in its composition, according to the locality and the manner in which it is collected. Details of the different kinds of resins obtained from this species are given by Tschirch & Stock (1935. *Die Harze,* **2**(1) : 105).

According to Hooper (1904. *Agric. Ledger* : p. 118), Manson slashed rasamala trees in Burma and obtained a white honey-like exudation from some of the wounded trees, but not from all of them. He noticed that the crust of resin near old wounds was hard and black. Manson also employed the method of obtaining resin from dipterocarps (by the application of fire ?), and recovered a dark-brown solid resin.

Schimmel & Co. (1892, April. *Rep.* : p. 56) obtained 0·17 per cent. of an essential oil by distilling the wood of Dutch East Indies origin. At ordinary temperature this oil formed a semi-solid crystalline mass having an odour resembling that of cinnamon and rhubarb. The principal constituent of the oil was a crystalline substance which melted at 54° to 55°C., and probably a ketone of which the oxime melted at 160° to 107°C. According to Gildemeister & Hoffmann, however, it is not certain whether the wood subjected to distillation by Schimmel & Co. was from *A. excelsa* or any other tree known by the name of rasamala.

Tschirch & van Itallie (1901. *Arch. Pharm., Berl.*, **239** : 541) have reported cinnamic acid, benzaldehyde, and cinnamaldehyde from the resin obtained from Java.

Hooper (*loc. cit.*) examined two kinds of balsams from Burma. The one which resembled honey in colour and consistency when fresh became white and crystalline after two years and possessed a delightful fragrance of styrene. This " soft white crystalline balsam " contained 7·65 per cent. of a volatile oil. The total cinnamic acid content in the resin was 37 per cent. The resin had the following characteristics : acid val. 24·96, ester val. 174·39, saponification val. 199·35, and iodine val. 57·3. The other sample consisted of a dark-brown solid balsam with a strong and pleasant odour, that of cinnamon predominating. It contained 38 to 41 per cent. impurities, traces of free cinnamic acid, and 9·7 per cent. combined cinnamic acid. The pure resins amounted to about 54 per cent. and the essential oil to about 6 per cent. The characteristics of the balsam after purification with alcohol were : acid val. 76·8, ester val. 53·64, saponification val. 130·44, and iodine val. 51·68.

Hooper (*loc. cit.*) confirms Tschirch & van Itallie (*loc. cit.*) that the Burmese resin, like that of Java, has a different constitution from true storax of Asia Minor. At the same time he states that the resin is rich in aromatic compounds.

The resin from Assam trees has been recently investigated at the Forest Research Institute, Dehra Dun. It is a pale-yellow odorous resin and is obtained in clear tears from clean wounds. It contains 4·4 per cent. of essential oil. The resin extracted by 95 per cent. alcohol has m.p. 90° to 95°, sp. gr.$^{20°}$ 1·046, acid val. 58·3, saponification val. 105·4, and ester val. 47·1 (Unpublished Records of the Forest Research Institute, Dehra Dun).

2. LIQUIDAMBAR Linn.

(Means *liquid amber*, referring to the fragrant resin which its species yield.)

This genus comprises four arboreous species which are found in the Mediterranean region, Asia, and N. America. All the species yield fragrant balsams, known in commerce as storax or styrax, but that from *L. orientalis* Mill. of Asia Minor is the most important, and called Oriental or Levant storax. This is a semi-liquid, greyish-brown, sticky, opaque substance with a pronounced aromatic odour. Next in importance is the American storax which is obtained from *L. styraciflua* Linn. (sweet gum). This balsam is a thick, clear, brownish-yellow, semi-solid or solid substance. The Chinese storax is obtained from *L. formosana* Hance.

Storax is highly prized by the perfumer on account of its fixative properties and its sweet, heavy, " Oriental " type of odour. The purified balsam is largely used in soap perfumery and cosmetics, and in perfumes of the type of hyacinth, narcissus, hawthorn, cassie, magnolia, etc. It is also used in incense, as a flavouring for tobacco, and as an important ingredient of many pharmaceutical preparations.

In Asia Minor, the outer bark of the tree is bruised and beaten in order to promote the secretion of the balsam. The outer bark is then removed and sold for fumigation purposes. The inner bark is then stripped off and boiled in water, when the balsam floats on the surface whence it is removed. The bark is also expressed subsequently (Poucher). For another and slightly different account of the collection of storax the reader is referred to an article by Jeancard (1925. *Parfum. mod.*, **18** : 73). The crude balsam, known as liquid storax of commerce, is further purified by dissolving in alcohol, filtering to remove portions of bark and other debris, and evaporating to remove the solvent ; it is then called " prepared storax ".

According to Finnemore, the Oriental balsam contains free cinnamic acid (23 per cent.), combined cinnamic acid (24 per cent.), aromatic esters (22·5 per cent.), styrene and vanillin (2 per cent.), resin (36 per cent.), water (14 per cent.), insolubles in ether (2·4 per cent.). According to Naves & Mazuyer (1947. *Natural Perfume Materials* : p. 265) a good quality balsam gives 60 to 75 per cent. of an alcoholic resinoid, 55 to 70 per cent. of a benzene resinoid, 35 to 55 per cent. of a petroleum ether resinoid, and 65 to 70 per cent. of an acetone resinoid. These resinoids are prepared with heat. Storax is known to contain a high percentage of water ; sometimes the water may reach 20 or even 30 per cent. Storax purified with alcohol has the following characteristics : acid val. 68 to 94, ester val. 105 to 154, and saponification val. 171 to 234.

The balsam yields 0·5 to 1 per cent. of a highly aromatic oil (storax oil). This oil

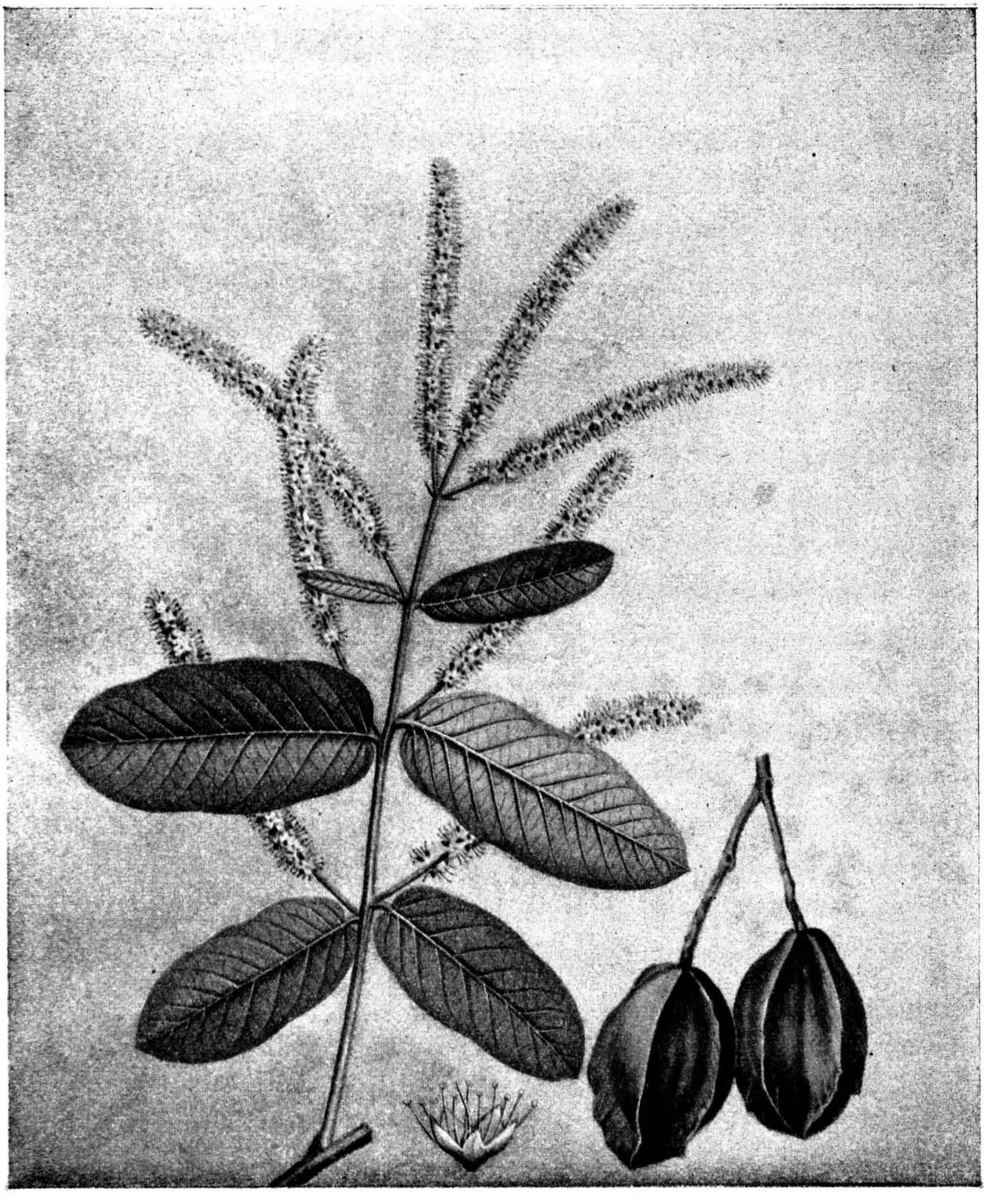


FIG. 44 — *Terminalia arjuna* Wight & Arn.

is a bright-yellow to dark-brown liquid of varying constitution, which depends upon the manner and style of distillation. It contains styrene (styrol, phenylethylene), which is the principal odorous constituent having an unpleasant, sharp, benzene-like smell, cinnamaldehyde (styrone), with the ethyl, benzyl phenylpropyl esters of cinnamic acid, and a trace of vanillin. Some cinnamyl cinnamate distils over with it. Naphthalene has also been isolated from the oil (Finnemore).

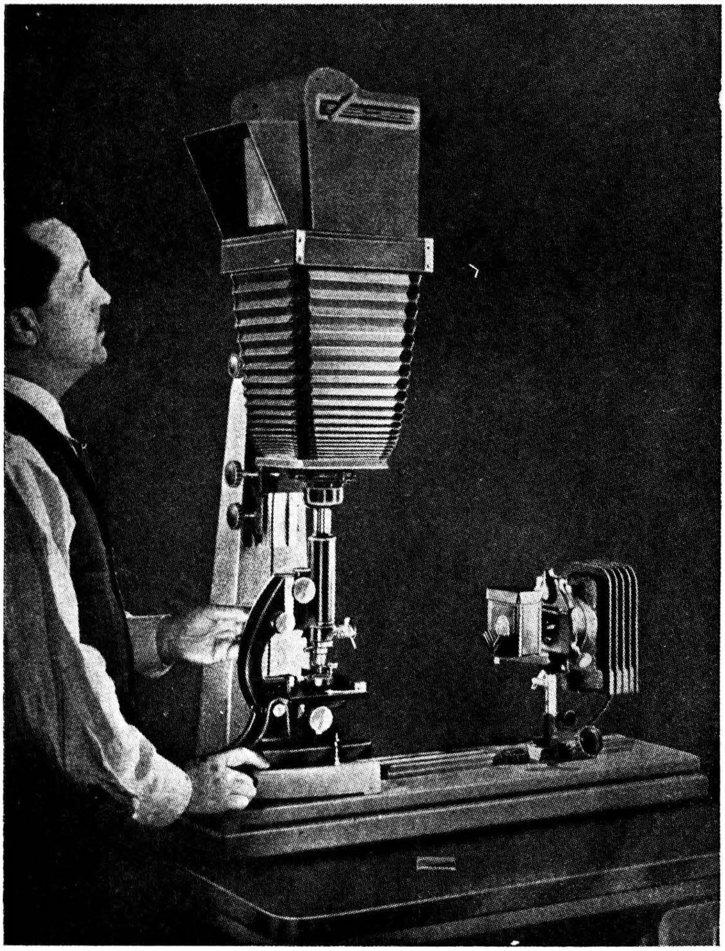
Family XLV — COMBRETACEAE

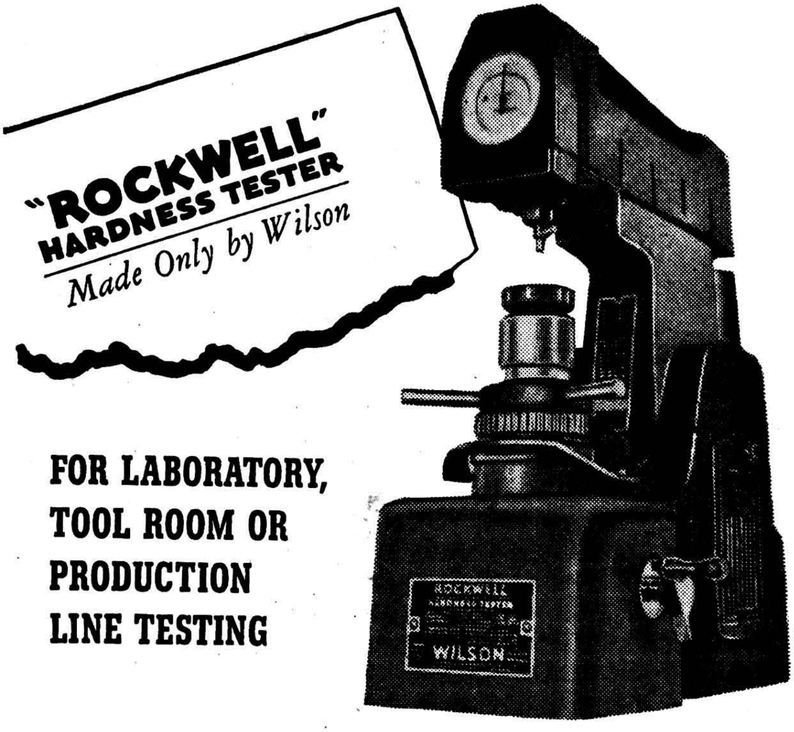
(Myrobalan Family)

This family comprises 16 genera and about 500 species of trees and shrubs, some of the latter climbing, with alternate or opposite, simple, entire leaves and perfect or rarely polygamous flowers. Its representatives are widely distributed throughout the tropical and subtropical regions of the world.

From the economic point of view this is an important family, containing as it does some of the important and widely distributed trees of India, which are valuable for their timber and other products. Several species of *Terminalia* and *Anogeissus* are especially important for their timber. The fruit and bark of many plants, especially the terminalias, have astringent properties due to the presence of large quantities of tannins ; they are often used medicinally. The fruit of *Terminalia chebula* Retz. (chebulic myrobalan, *harar*) is a valuable tanning material, and large quantities of it are exported, valued at about Rs. 4,500,000 annually. That of *T. bellirica* (Gaertn.) Roxb. *ex* Fleming (belleric myrobalan, *bahera*) enjoys considerable internal trade for use in indigenous medicine, as a purgative, etc. The gum (ghatti gum) from *Anogeissus latifolia* Wall. is commercially important, being a good substitute for gum tragacanth. *Quisqualis indica* Linn. (Rangoon creeper) is commonly cultivated in Indian gardens.

Members of this family have not been investigated from the point of view of essential oils, but a number of them bear scented flowers. The small flowers of *Terminalia alata* Heyne *ex* Roth, syn. *T. tomentosa* Wight & Arn. and *T. tomentosa* Bedd., var. *typica* C. B. Clarke (laurel, *ain*, *saj*), *T. arjuna* Wight & Arn. (*arjun*), *T. catappa* Linn. (Indian almond tree, *jangli badam*), *T. travancorensis* Wight & Arn., syn. *T. angustifolia* Roxb. (*kotta-kadakai*) are sweetly scented. The flowers of *T. chebula* Retz. and *T. bellirica* (Gaertn.) Roxb. *ex* Fleming have an offensive odour. All these are medium-sized to large trees of wide distribution in India, except *T. travancorensis* which is confined to the evergreen forests of Travancore. *Combretum nanum* Buch.-Ham. *ex* D. Don, an undershrub of the outer Himalayas, W. Bengal, and the Central Provinces, bears terminal racemes of white, fragrant flowers which are ¼ in. in diameter. The flowers of *Quisqualis indica* Linn., a large subscandent shrub which is commonly cultivated in gardens, are sweet-scented ; they are 2½ in. long and 1½ in. across, white, soon turning red, and borne in axillary and terminal spikes.





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