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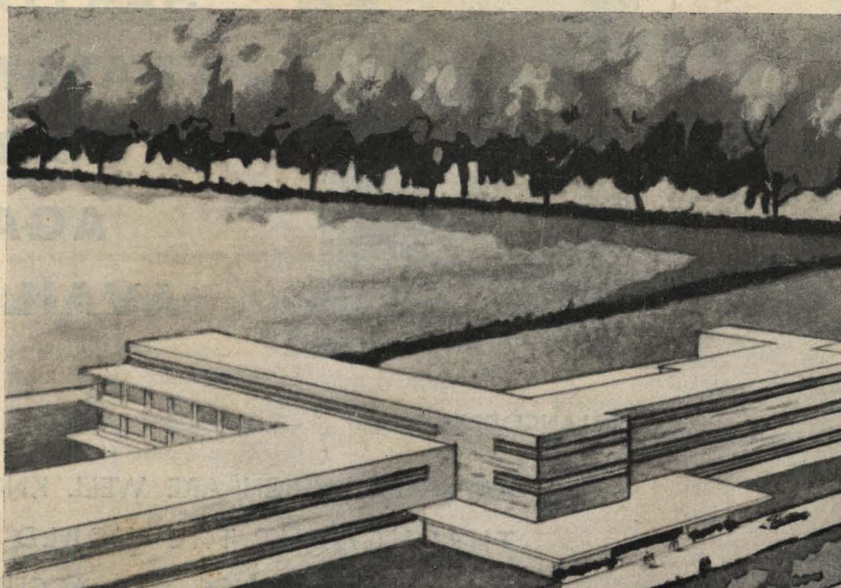

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Scientific & Industrial Research

Vol. VIII **AUGUST 1949** **No. 8**

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COVER PICTURE

A perspective view of the *Power Engineering Laboratory*, Bangalore, is shown on the cover page. This laboratory, the first of its kind in India, will be equipped with the A.C. Network Analyser (cf. page 309) and other instruments necessary for the solution of the many engineering problems connected with the nation's vast power-development projects.

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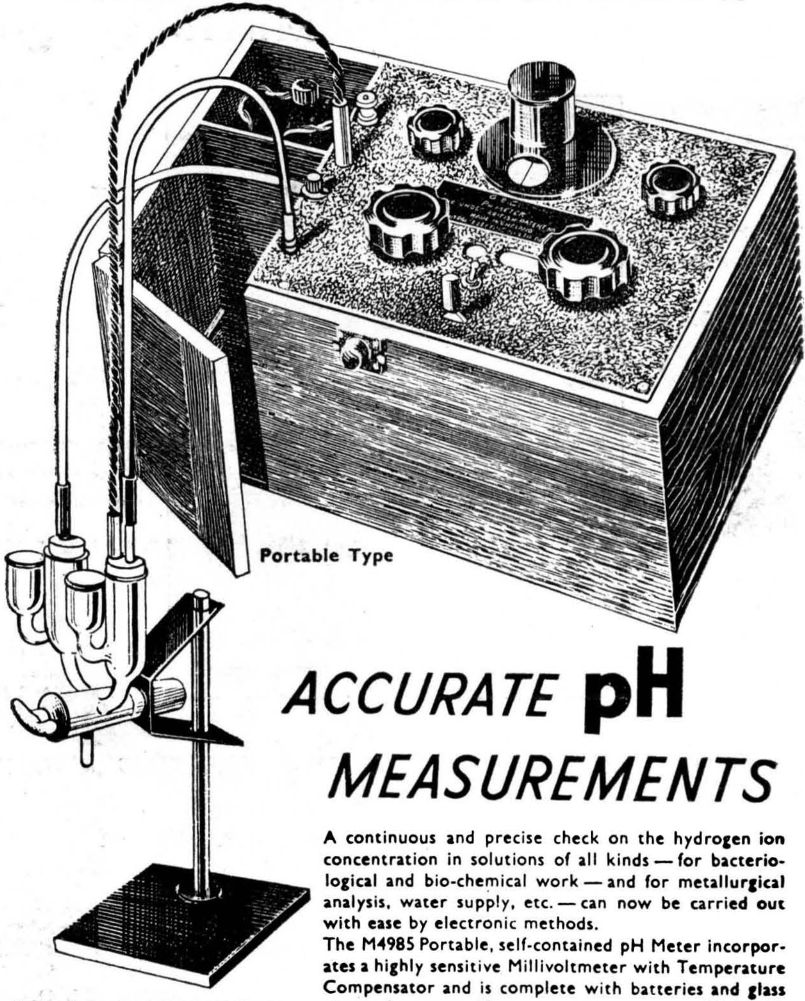

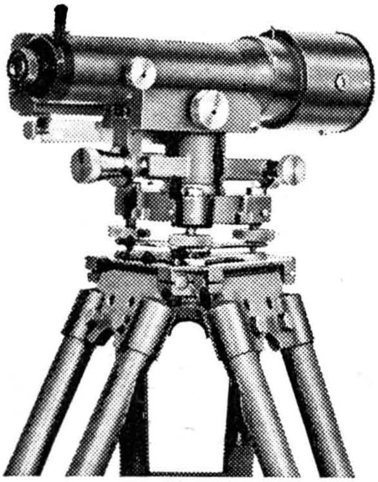

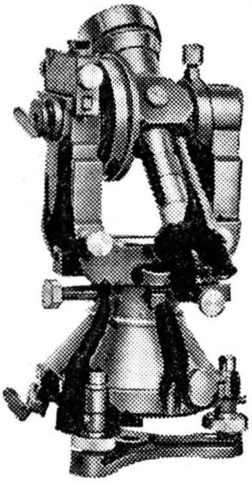

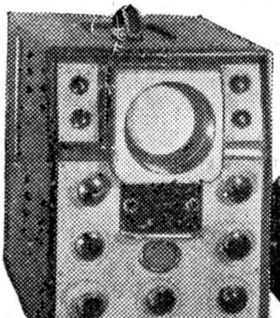

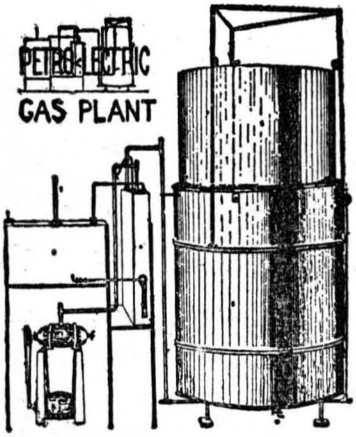
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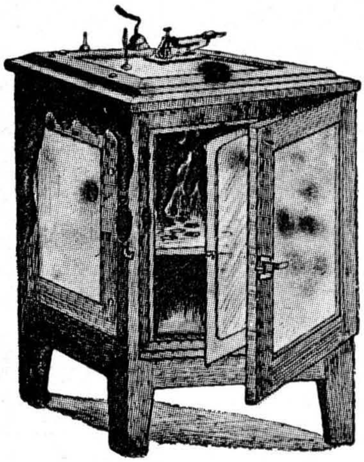

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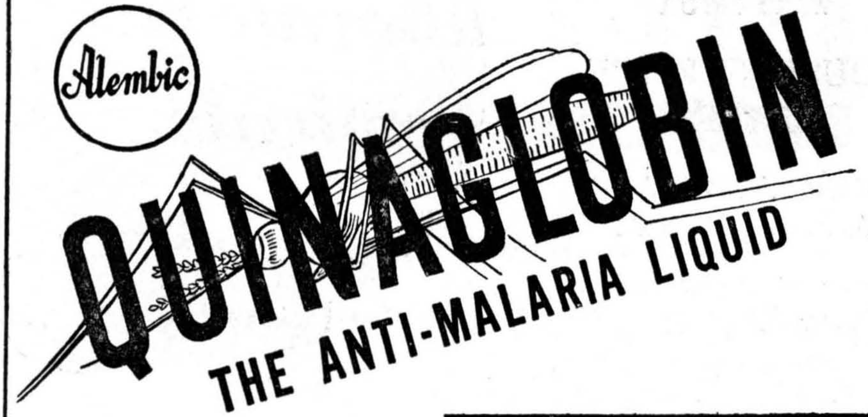

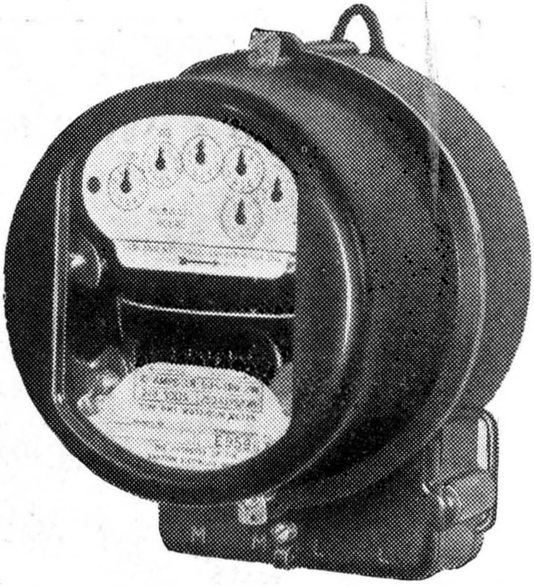

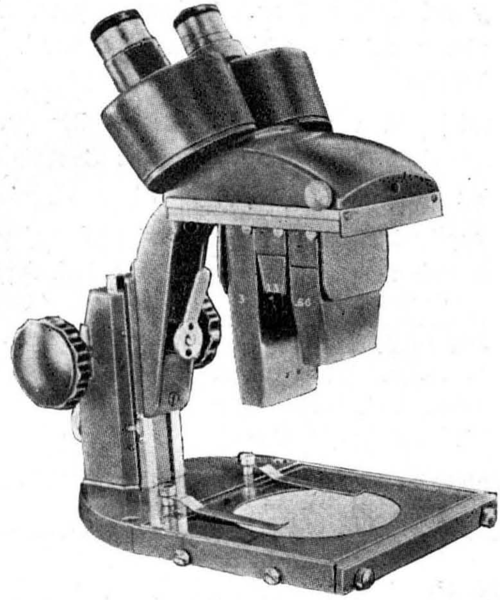

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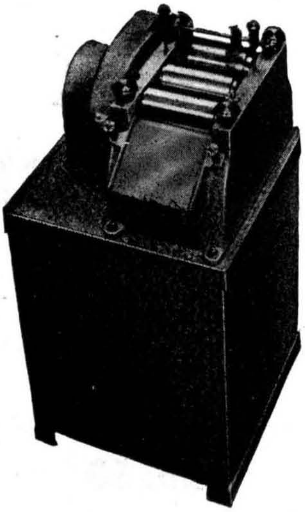

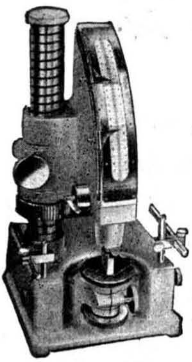

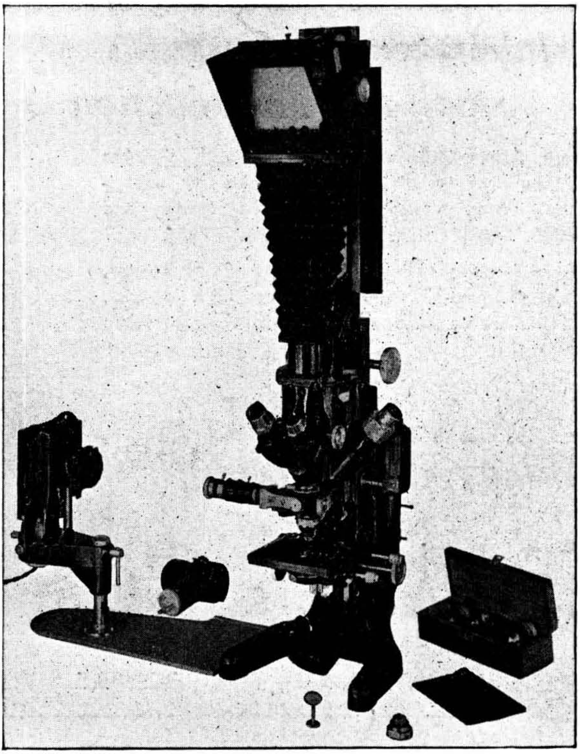

Need for Research on Malaria Chemotherapy in India*

SOME idea of the magnitude of the malaria problem in India may be obtained from the statement of Hehir who estimated in 1927 that there were about 100,00,000 cases of malaria occurring annually in India. During war-time, the incidence of the disease must have assumed more serious proportions because of the unchecked growth of the malaria-carrying mosquitoes from untreated swampy areas, their rapid transmission from place to place and the acute shortage of proper drugs to treat " carrier " cases. The total number of malaria cases in India must have, therefore, increased very significantly during the period from 1927 to to-date. The annual economic loss from malaria alone is consequently collosal, and it is believed in very authoritative quarters that if proper *curative* treatment could be elaborated for this single disease, India can be converted in a single generation to one of the most prosperous and healthy countries of the world.

Fundamental Objective of Malaria Chemotherapy

The objective of our research in this field is the control of malaria by *chemotherapy* or *chemo-prophylaxis*. There is a growing volume of opinion that the only effective way through which malaria can be *radically eradicated* is through the discovery of a drug or drugs capable of *sterilizing* the patients from the malarial infection. Preventive methods, through anti-mosquito campaigns and other methods of larvae control which have been practised widely during the last half a century, though useful, have been proved in many parts of the world to be ineffective *per se*. That we have fallen far short of our goal of *total eradication* of malaria in spite of all types of anti-malarial measures is only too evident to need further emphasis. According to Coggeshall of the Rockefeller Institute for Medical Research, New York, malaria as a world disease is probably on the increase at the present time.

This objective may be attained in 4 ways :

(i) Determination of the optimum method of using known anti-malarial drugs (e.g. quinine, mepacrine, etc.) in " suppressive " treatment ;

(ii) discovery of drugs which can act as true causal prophylactics (e.g. sulphadiazine in gallinaceum malaria in the chick) in human malarias and which can be given to man with safety over extended periods of time ;

(iii) discovery of drugs which will consistently eradicate the parasites in human infections and thus prevent relapses which commonly occur after " clinical cure " with quinine or mepacrine ; and

(iv) discovery of drugs which have anti-malarial properties qualitatively similar to quinine or mepacrine, but which possess a larger margin between the effective and toxic doses.

The first item is more or less thoroughly worked out as a result of studies made both in Great Britain and America during the war (1941-45). A good review of work already completed in this field is available from a paper by J. A. Shannon and others.* The other three ways of attaining the objective have also been intensively pursued, particularly comprehensively in America. An extensive programme of research, supported mainly by the Committee on Medical Research (CMR) of the Office of Scientific

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J. Phar.*, 1944, **81, 3730.

* From a scheme submitted before the Drugs & Pharmaceuticals Committee of the *Board of Scientific & Industrial Research* (Chairman — Dr. Jivaraj N. Mehta).

Research and Developments (OSRD), was launched as early as 1941 with a view to stamp out the dread of malaria from the fighting forces in the tropical theatres of the last global war. A detailed account of this American scheme on anti-malarial research which involved the co-operation with civilian scientists, scientists from the U.S. Army, Navy and Public Health Services, workers in industrial firms and also international co-operation with such countries as China, Great Britain, Canada, Australia, Argentina, French Guinea, Columbia, Cuba, etc., has now been published in the form of a monograph entitled *Survey of Anti-malarial Drugs* (2 volumes), edited by F. Wiselogle. This report indicates that in the search for newer anti-malarials other than the cinchonas, no significant progress can be made unless a close co-ordination can be established between the synthesis of new compounds, pharmacological and experimental therapeutic investigation of these compounds in animals, and clinical trial in human malaria of selected compounds under carefully controlled conditions.

Present Status of the Problem

The subject is so vast that any attempt made to summarize the picture in a short compass would result in more confusion than clarification of the problem. In brief, it may be stated that in spite of the very valuable researches in the field of malarial chemotherapy so far reported, an *ideal anti-malarial drug* has not yet been found and, therefore, there is room for further search. Further, most of the findings on which our present concepts on the use of such drugs as chloroquine and paludrine are based are dependent on experimental data on strains of malaria parasites of American, Australian and south Pacific origin, which may not necessarily be applicable to Indian strains of malaria ; at least this has to be confirmed experimentally in India.

How Success Can Be Achieved ?

Complete success in the elaboration of a truly effective anti-malarial would involve :

(i) An improvement in suppressive therapy (this has been achieved to a certain extent by the discovery of chloroquine and paludrine) ;

(ii) an improvement in the treatment of clinical attack (also partially achieved) ;

(iii) the development of agents which would cure *falciparum* malaria (now achieved) and *vivax* malaria at a well-tolerated dosage ; and

(iv) finally, the development of agents which would prevent the inception of these diseases (true causal prophylactic).

Target in Anti-malarial Chemotherapy

The target to be arrived at can be briefly stated as follows :

(a) The discovery of a true causal prophylactic for human malaria — a drug which would eradicate some stage of the parasite before trophozoites were produced — would constitute a great advance.

(b) The discovery of a drug which would resemble mepacrine in its anti-malarial properties but which, in addition, would cure *vivax* malaria. This would probably be of more value than (a) (e.g. sulphadiazine in chick malaria which is a prophylactic but not a curative drug).

(c) Another ideal approach would be through the use of drugs hampering the activity of the enzyme system or metabolites essential to the malarial parasites but not necessarily so to the host (e.g. activity of sulpha drugs through p-aminobenzoic acid system).

The " leads " that suggest themselves to research workers from a critical perusal of anti-malarial drug research so far conducted are given in the Appendix at the end.*

Co-ordination of Malaria Study in India

In view of the supreme importance to India of research in malarial chemotherapy and in view of what has been done in countries where the malaria problem is of far less magnitude than in our country, it is very desirable that immediate steps be taken for the initiation and active continuance of chemotherapeutic researches with special reference to malaria. As indicated in the Appendix, a number of useful " leads " are already available and this can be pursued provided an organization more or less on the lines adopted by the " Board for the Co-ordination of Malaria Studies " of the United States of America is set up in India. The set-up of the unit established in America during the war period under the auspices of the Office of Scientific Research and Development is shown in the chart.

The Board consisted of a President who was helped in his work by a Technical Executive Secretary and a Technical Aide, the latter maintaining constant liaison with the

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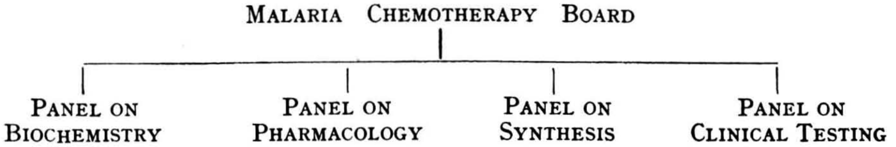
* Marshall, *Federation Proceedings*, 1946, **5**, 298.



chairmen of the 4 panels. Each panel had, in addition to a chairman, at least 3 members acknowledged to be experts in their respective fields. The chairman of each panel was responsible for initiating work but the members had an independent status and had full freedom to suggest problems and undertake work after the programme had been discussed with the chairman. Each panel was provided with a technical secretary, who undertook the review of progress of work in his field all over the world. Financial support was made available to this body from the Office of Scientific Research and Development and many private agencies also collaborated with men and money to make the effort successful.

If it is not possible to set up as elaborate an organization as planned in America, there is good reason to feel that the time is at least opportune for the setting up of a broad-based committee of experts who could probe into the problem of chemotherapy of malaria with the full knowledge of the complexity of the Indian problem and suggest solutions likely to be of value to the therapy of Indian strains of malaria. The Malaria Institute of India is already tackling the Indian malaria problem with commendable zeal and energy. It is for the chemotherapeutists of India to come forward and help the Institute with newer synthetics for their studies, or take their advice for further development in this direction.

For any success of this scheme one thing must be emphasized and that is that this kind of research involves the contribution of many sciences, and, therefore, "team work" is essential for rapid progress.* The chairman of any such committee must be a scientist of wide and comprehensive outlook with a *drive* which will make working in such a team both pleasant and profitable. The potentiality of individual thought and work upon the numerous fundamental problems of malaria must be appreciated and at the same time centralization and team work emphasized. Research in this field holds great promise for the future and there is no reason why an Indian should not be competent to contribute to this programme which has already engaged international attention. Even if no tangible results are arrived at from Indian work, the training which the workers will get from such a collaborative research undertaking will pave the way for some unknown achievement in the synthetic and chemotherapeutic field.

APPENDIX*

"Leads" from Anti-malarial Drug Research so far Conducted

(a) 4-*Amino-quinolines* — If one takes the formula (FIG. 1) for mepacrine and removes either of the outer rings, one obtains two 4-amino-quinolines, one with a methoxy group in the 6-position and one with a chlorine group in the 7-position. The first of these compounds belongs to a series which, in its anti-malarial properties, resemble quinine and mepacrine rather than the 8-amino-quinoline, pamaquine.

After examining nearly 200 compounds in this series, the American workers have screened 10 of these 4-amino-quinolines, which appear to be superior to mepacrine. Chloroquine (SN 7,618) has received the most extensive study in both

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* The principle of collaborative work between all concerned bodies on malaria research in India was approved by the Drugs & Pharmaceuticals Committee and later by the *Board of Scientific & Industrial Research* at their meeting held on 18th January 1949. The *Board* recommended that a standing committee for malaria chemotherapy be constituted to co-ordinate and direct research and other investigations in the subject carried out in India. This committee consists of 8 members excluding the chairman, the personnel being drawn as follows:

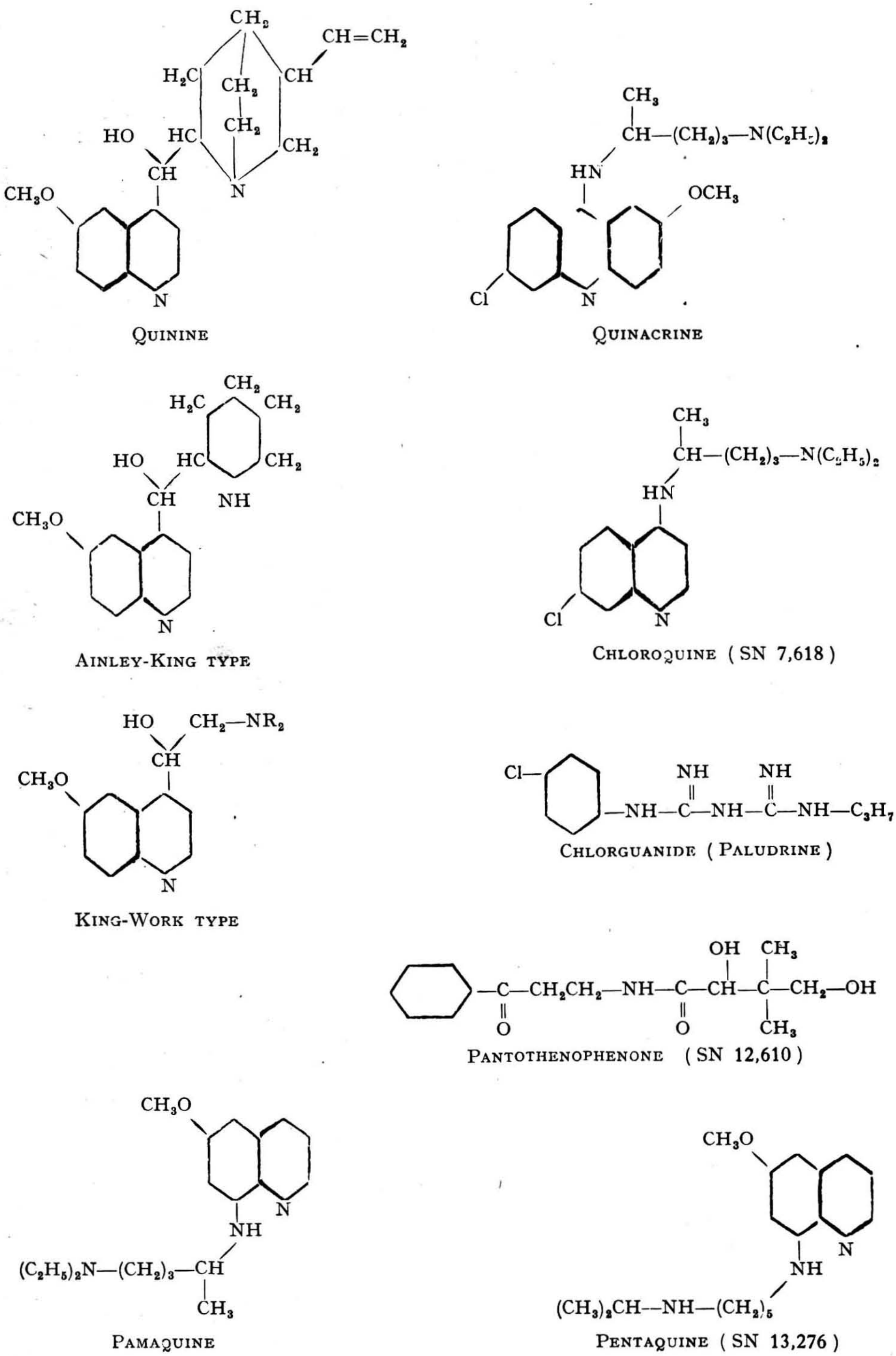
1. Chairman of the Pharmaceuticals & Drugs Committee of the *Board of Scientific & Industrial Research* or his nominee.

2. Six members (from various learned bodies, research institutions, defence science or medical organization, Malaria Institute, etc.) to be nominated by the chairman.

3. Two members to be nominated by the Scientific Advisory Board of the Indian Research Fund Association.

The Secretary of the Drugs & Pharmaceutical Committee was nominated as the Secretary of the Malaria Chemotherapy Committee.

* This statement takes note of only the highlights of chemotherapeutic research on anti-malarials and is not a comprehensive review of the vast field already covered in this field.



Quinine

Quinacrine

Ainley-King type

Chloroquine (SN 7,618)

King-Work type

Chlorguanide (Paludrine)

Pantothenophenone (SN 12,610)

Pamaquine

Pentaquine (SN 13,276)

Fig. 1 — Formulae of anti-malarial drugs.

civilian and military establishments. It is superior to mepacrine in a number of ways. Effective suppression can be obtained by administering it no more frequently than once a week in a well-tolerated dose. It will cause an abrupt termination of the clinical attack of *vivax* malaria and will cure *falciparum* malaria when administered for only one or two days. In addition, it does not stay in the skin or produce gastro-intestinal disturbances. In this field further work is urgently called for. It would, for example, be necessary to know the nature of the metabolic degradation products of 4-amino-quinoline derivatives (chloroquine) in human beings. Some knowledge about the metabolic degradation products of quinacrine has now been obtained from biochemical studies of excretory products from human urine. If similar data on chloroquine, etc., are obtained, suitable substituted alkylamino and/or more stable nuclear-substituted compounds could be synthesized in that series.

(b) *8-Amino-quinolines* — It is now practically certain that quinine and pamaquine, under certain conditions, will cure both American and South-west Pacific strains of *vivax* malaria. However, there is probably not a sufficient margin between minimal curative and maximum tolerated doses to warrant its use at this time. About 20 of the 8-amino-quinolines have been tried for their curative effects in *vivax* malaria. One compound, SN 13,276 (pentaquine), appears to be curative in a smaller dosage than pamaquine and to be about one half as toxic for man. Possibly this drug or similar drugs in this series will have a wide enough margin of safety to be used in the cure of *vivax* malaria. As the main objective would be to search for a true prophylactic, an intensive investigation in this series appears more promising as these are the derivatives which have shown gametocidal activity also.

(c) *Quinoline-methanols*—These compounds may be considered as being rather closely related to quinine. Ainley and King in 1938 reported the first compound of this series to be found active in avian-malaria. Later, King and Work prepared a series (*see* formula) and found them also to possess anti-malarial activities. About 200 compounds of the quinoline-methanol type have been prepared and in both the A-K and K-W series, we have now compounds about 40 times as active as quinine. About 5 compounds of this series have been tried in man for anti-malarial activity and comparatively high toxicity. However, due to poor absorption and toxicity, none of these compounds have appeared promising. Recent findings have shown that quinoline ring is not necessary for anti-malarial activity and it would, therefore, be of interest to introduce newer chain or nucleus to these methanol derivatives.

(d) *Pantothenic Acid & Analogues* — Trager* reported that the survival *in vitro* of *P. lophurae* was favoured by the presence of calcium pantothenate. This observation resulted in the synthesis of about 25 analogues of pantothenic acid for anti-malarial testing. A compound synthesized by Woolley† and found by him to be an antagonist for pantothenic acid in the case of bacteria, was found, when given orally, to be as active as quinine in *gallinaceum* malaria in the chick, active in *lophurae* malaria in the chick, but inactive in *lophurae* malaria in the duck. This substance, pantothenophenone, SN 12,610, was found definitely active in *vivax* malaria in the human being. This is the first instance in which an effective anti-malarial agent has been found as a result of a logical, rather than a " hit or miss " approach. However, much success has not been achieved in the parallel field of sulpha drugs therapy by working on this plan. The bottle-neck lies in our rather imperfect knowledge of the nutritional requirements of malaria parasites and their metabolic peculiarities.

(e) *Guanides, Amino Phenols, etc.* — The discovery of chlorguanides (paludrine) has switched the attention of chemotherapeutists to another new angle altogether. Though chlorguanide is " prophylactic " in *falciparum* malaria and partially so in *vivax* malaria, it is usually considered useful only as a " suppressive " drug. It may be possible, through further studies, to develop simple basic compounds such as other guanides, amino acid derivatives, amino phenols, etc., that might react with the trace elements or complex pyridine bodies like pyridoxine resulting in the elaboration of newer synthetic derivatives of value, both in prophylaxis as well as in suppression and cure of malaria. This field still remains largely unexplored.

B. MUKHERJI

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* *J. Exper. Med.*, 1943, **77**, 411.

† *Sci.*, 1944, **100**, 579.

Board of Scientific & Industrial Research Twenty-third Meeting, New Delhi

THE twenty-third meeting of the *Board of Scientific & Industrial Research* was held in New Delhi on July 11, 1949. The Hon'ble Pandit Jawaharlal Nehru presided.

On the recommendation of the Board, the Governing Body of the *Council of Scientific & Industrial Research* sanctioned 13 new schemes :

1. Synthesis of citrinin and preparation of derivatives of citrinin — DR. K. VENKATARAMAN (*Bombay*).
2. Elucidation of the constitution of essential oils and their synthesis — DR. P. C. GUHA (*Bangalore*).
3. Physico-chemical properties of hides — tanned and untanned — DR. B. N. GHOSH (*Calcutta*).
4. Scheme of research on leather — MR. B. M. DAS (*Calcutta*).
5. Studies on colloidal instability of cloud particles above and below the freezing temperature — DR. S. K. BANERJEE and DR. L. S. MATHUR (*New Delhi*).
6. Polarization of down-coming radio waves — DR. S. R. KHASTAGIR (*Banaras*).
7. Cellulose decomposition by soil fungi with special reference to its application in fermentation technology — DR. T. S. SADASIVAN (*Madras*).
8. Enzyme bates—MR. S.N. Sen (*Calcutta*).
9. Research on soft X-rays — PROF. S. N. BOSE (*Calcutta*).
10. Development of magnetic dust cores — MR. B. D. TOSHNIWAL (*Bombay*).
11. Development of an electronic type of frequency analyser — DR. N. B. BHATT (*Bangalore*).
12. Study of intensity variation of short-wave radio signals and their bearing on the ionosphere — DR. S. S. BANERJEE (*Banaras*).
13. Studies of correlation between abnormal variation of region of ionization of the ionosphere and similar variation of the night sky emission — DR. S. N. GHOSH (*Calcutta*).

The setting up of a pharmacological unit, temporarily at Calcutta, under the direction of Dr. B. Mukherji was approved. This unit will be transferred to the Central Drug Research Unit, Lucknow, as soon as its laboratories are fully equipped and start functioning.

The Central Drug Research Institute will have a full division for undertaking botanical research on indigenous drugs. This division, in addition to carrying out laboratory investigations, will undertake experimental cultivation, surveys, grading and sorting of indigenous drugs and also serve as a centre of intelligence and technical assistance to the indigenous drug industry.

The Governing Body of the Council considered the report of the Reviewing Committee which was appointed during 1947 to review the utilitarian aspects of the Council's activities. This Committee (Chairman : Mr. Ardeshir Dalal) undertook a survey of the activities of the Council and the findings are that the *Council of Scientific & Industrial Research* has done much valuable work during the short period of its existence. Some of the recommendations of the Committee with regard to increasing Council's usefulness to industry are greater liaison with industry, improvement in the publicity organization of the Council, setting up of pilot plants in the National Research Laboratories, and the establishment of a Board of Engineering Research under the auspices of the Council.

In order to facilitate a better and more complete utilization of the results of research in the country, the *Council of Scientific & Industrial Research* has recommended that the Government of India should set up a National Research Development Corporation on the lines of similar organizations in America, Britain and Canada. The primary object of the corporation will be to conduct semi-commercial trials of new processes developed in India and thereby encourage industry to adopt them in large-scale practice.

It has been recommended that the corporation should be floated with a capital of Rs. 50 lakhs and a recurring expenditure of Rs. 5 lakhs to start with, and the co-operation of all State-owned and State-controlled industries and of private concerns would be sought in the efficient working of the corporation. A committee with Mr. Ardeshir Dalal as the chairman, Dr. J. C. Ghosh, Mr. Kasturbhai Lalbhai, Mr. Sri Ram and Dr. S. S. Bhatnagar as members has been set up to work out the details.

The Governing Body approved the installation of pilot plants in all the National Research Laboratories. A programme has been outlined to render increased technical assistance to industry. An Industrial Relations Officer is being appointed to maintain liaison with all the major trade associations and chambers of commerce. Technical personnel of the Council will visit industrial establishments to render such technical aid and advice as may be necessary for the exploitation of new processes.

A Board of Engineering Research is to be established under the auspices of the *Council of Scientific & Industrial Research*. The primary function of this body will be to co-ordinate research work carried out in various engineering establishments under the Government of India.

With regard to the possibility of producing synthetic petrol from coal, the Council has proposed that pilot-plant investigations should be conducted in India itself so that all types of low-grade coals obtaining in India can be examined. Two other proposals put forward relate to the utilization of pulverized coal for firing stationary and locomotive boilers and the development of a turbine for locomotives employing pulverized coal. As an experimental measure, the Council has suggested importing a boiler in which pulverized coal can be employed. The unit is to be set up at one of the thermal stations in the Damodar Valley area.

A Fuel Economy and Combustion Section is being organized in the Fuel Research Institute, Dhanbad, to advise consumers of coal on coal conservation and economizing methods.

At the instance of the Council, the Indian Standards Institution has constituted a committee to fix standards of purity for salt manufactured in India for various purposes, and the Council has recommended that these standards be soon introduced in the salt industry. A Salt Research Station is to be established at Wadala, Bombay, where scientific methods of salt production would be worked out and demonstrated to salt producers. It is also proposed to establish another research unit at Sambhar Lake area to deal with the problems of salt manufacture from lake and pit brine. The Council has recommended to the Government of India that the Mandi salt mines and the salt deposits in the Rann of Cutch should be developed.

The proposal of the Indian Statistical Institute for the establishment of a research unit in quality control to serve the needs of industries in Bombay has been accepted by the Council. This research centre will be attached to training centre in quality control financed by industry in Bombay.

The Council approved the appointment of Professor Kriedel, Director of Research, *Bausch & Lombe*, U.S.A., as Director, Central Glass & Ceramic Research Institute, Calcutta.

Characteristics of the Ionosphere over Calcutta (May 1949)

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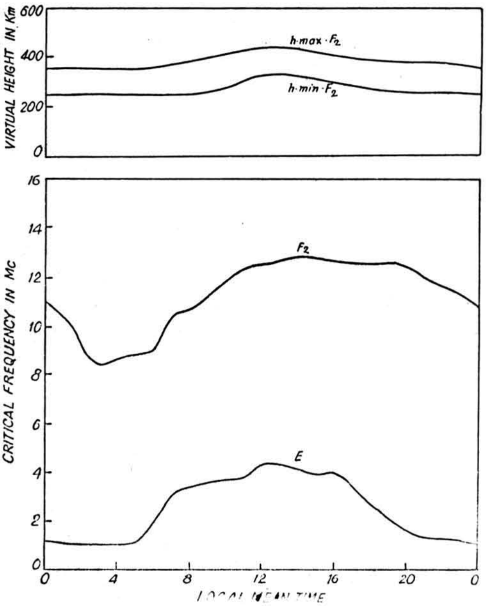
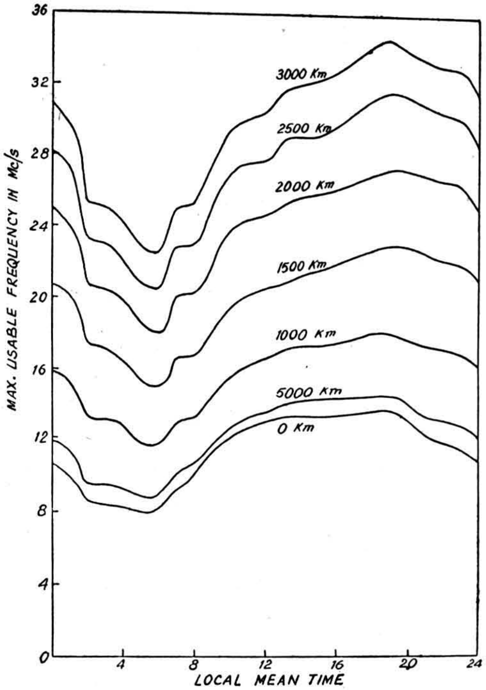
THE following are the ionospheric data observed at Calcutta for the month of May 1949. The observations were made at each hour of the day for 5 days a week.

Fig. 1 represents the mean hourly values of the virtual heights and critical frequencies of the F_2 layer and the critical frequency of the E layer in graphical form during the month of May 1949. Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmission during August 1949 by reflection at the F region over Calcutta. Table I gives the list of occasions when E region ionization was found to be abnormal and the corresponding penetration frequencies and virtual heights.

There were frequent occurrence of thunder-clouds during this month at different times



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



AT POINT OF REFLECTION.
(5 HOURS 54 MINUTES AHEAD OF G.M.T.)
FIG. 2 — PREDICTED M.U.F. VIA F_2 LAYER, AUGUST 1949.

of the day and the E region ionization was found to be abnormal on most of the occasions. The afternoon abnormality in the F region ionization observed during the previous months has almost disappeared in the month of May. During the last few days of the month, however, the ionosphere was found to be disturbed.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$^{h}E_s$ Km.
May 1949	2	16.00	4·35	135
		17.00	3·50	135
		18.00	3·30	120
	3	20.00	3·25	120
		21.00	3·00	120
		22.00	2·70	105
		23.00	2·00	105
	*5	10.00	5·00	135
		11.00	5·20	135
		12.00	5·00	135
		16.00	4·65	135
		17.00	4·20	135
	*10	18.00	6·80	135
		19.00	5·50	135
		20.00	5·35	135
		21.00	5·30	135
		22.00	5·05	135
	*12	19.00	7·05	135
		20.00	7·00	135
		21.00	6·00	135
		22.00	3·50	120
		23.00	2·90	120
	13	00.00	3·30	105
		01.00	3·00	105
		02.00	2·75	105
		05.00	3·50	105
	*16	16.00	4·75	120
		17.00	4·80	120
		18.00	4·80	120
	18	07.00	4·10	120
		08.00	4·75	120
	*19	20.00	4·25	120
		21.00	4·20	120
		22.00	4·30	120
		23.00	4·25	120
	20	*00.00	4·00	105
		07.00	7·25	120
		08.00	7·45	135
		09.00	7·70	135
		19.00	4·75	150
		*20.00	4·75	150
		21.00	4·00	135
		23.00	3·75	135
	21	00.00	3·00	120
	23	17.00	3·15	120
		18.00	3·05	120
		23.00	2·80	105
	24	00.00	2·75	90
		01.00	2·70	90
		17.00	6·00	135
		*18.00	3·65	135
		*21.00	3·25	135
	25	09.00	4·50	135
		10.00	6·00	150
		11.00	10·75	150
		12.00	11·00	150
	*26	18.00	3·55	135
		19.00	6·75	135
		20.00	5·75	135
		21.00	5·35	135
		22.00	5·20	135
	27	00.00	3·55	120
		01.00	3·05	105
		02.00	2·70	90
		03.00	2·35	90
	*30	14.00	7·45	150
		15.00	7·50	150
		16.00	7·90	150
		17.00	7·50	150
	31	22.00	3·00	105
		23.00	2·55	90

* Local thunder-storms

The A.C. Network Analyser

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THE past decade has witnessed a very rapid increase in power development and power systems in the Western Hemisphere. India is on the threshold of a vast development of her potential power resources — its best, efficient, and economic utilization by an all-India interconnection of power systems — in the creation of a " National Grid ".

In this approach to power development, numerous problems, both engineering and scientific, have to be investigated and solved. Many investigations involved in this connection would be difficult to undertake because the amount of work and time required to arrive at satisfactory quantitative conclusions would be prohibitive. This fact has been more emphasized because of the necessity for rapid technological progress over a period of time during which there would be a relative dearth of technically trained men. The engineer or the scientist, fully aware of this situation and of the need for theoretical understanding in quantitative terms, has to look to ways and means for overcoming this limitation of man-power and also of time.

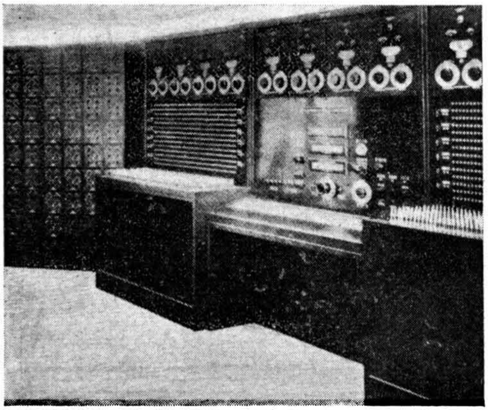

FIG. 1 — THE A.C. NETWORK ANALYSER, INCORPORATING INDUCTANCE AND CAPACITANCE IN ADDITION TO RESISTANCE IN ITS IMPEDANCE UNITS, IS APPLICABLE TO A WIDE RANGE OF DESIGN AND OPERATION PROBLEMS.

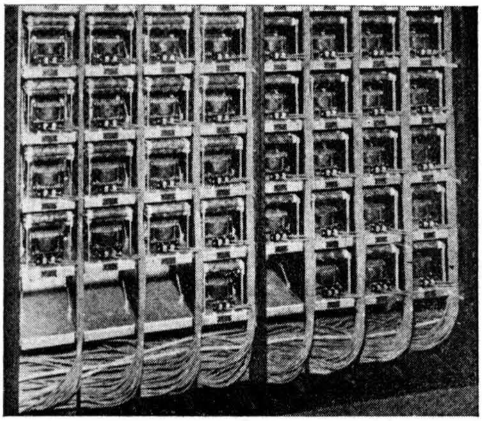

FIG. 2 — REAR VIEW OF TWO CIRCUIT-UNIT CABINETS SHOWING THE WIRING DETAILS.

For studying more readily the numerous problems associated with power system planning, design and operation, experimental methods of solution have been evolved and apparatus for such studies is built, commonly referred to as " Network Analyser " or " Network Calculator ". The use of such an analyser will be invaluable for national power planning.

A general view of this analyser board is shown in Figs. 1 and 2. It consists essentially of numerous adjustable impedance units of inductance, resistance and capacitance, such that the power systems or other electrical networks can be represented by them. Generators and impedance units can be set up to represent the desired system or systems and equivalent circuits. The system studies can be made fast, flexible and accurate. Representative circuits set up would efficiently solve certain problems which cannot otherwise be practically handled.

The types of information obtainable from the A.C. network analyser is indicated below. It is classified according to four general types of problems for which the analyser is particularly adopted, but which would suggest other uses. The list below is not final or complete but is merely indicative as a guide.

Power System Planning, Design & Operation Problems

Load Division —

- Desirable and economic location for new generation
- Effect of adding new transmission circuits
- Determination of location for new substation
- Effect of bus reactors
- Effect of increased system loadings
- Effect of changes in transformer reactance
- Effect on system of temporary loss of various transmission circuits
- Effect of temporary loss of generation or synchronous-condenser capacity
- Circuit loadings
- Bus voltages
- Reactive-power capacity needed
- Ratings of transformers and transformer windings
- Tap range of transformer required
- Benefits from load ratio control
- Benefits possible through use of phase-shifting transformers
- Effect of increased system voltages
- Effect of increased conductor sizes
- Determination of reactive-power flow; and
- Investigation of system losses and means of reducing them

Short-circuit, Three-phase & Unbalanced Faults —

- Maximum short-circuit currents for determining circuit-breaker duties
- Bus voltage during short circuits as a measure of fault severity and net effect on system
- Maximum and minimum fault currents and voltages for determining relay settings
- Determination of zero-sequence currents for inductive co-ordination problems
- Effect of mutual coupling between circuits on short-circuit currents and voltages
- Determination of grounding-transformer rating
- Effect of ground-fault neutralizer on fault currents in the transmission network
- Maximum and minimum fault currents for application of expulsion protective tubes; and
- Effect of various types of system grounding

Stability —

- Steady-state limits
- Transient limits with various types of faults;
- Switching times required for various types of faults and fault locations
- Effect of bus reactors on transient stability
- Effect of Wk^2 on transient-stability limits
- Effect of transient reactance on transient-stability limits
- Determination of increase in transient-stability limits with shorter switching and relay times
- Effect of switching and of bus arrangements on stability limits
- Effect of quick reclosure of circuit breakers
- Extent to which transient-stability limits are affected by saliency and excitation response
- Effect of damping torques on transient-stability limits
- Time required for generator to pull out of step after loss of excitation, and effect on the rest of the system; and
- Evaluation of effect of single-phase switching and reclosing on transient-stability limits

Special Circuit —

- Starting and pull-in characteristics required for large synchronous motors connected to a transmission system
- Determination of desirable methods of starting large synchronous motors or synchronous condensers
- Reduction of a complicated network to a more simple equivalent
- Determination of currents and voltages in three-phase circuits containing unbalanced impedances
- Cable-sheath currents
- Determination of fifth-harmonic voltages generated in a system by transformer magnetizing currents
- Solution of equivalent circuits representing mechanical elastic, and dynamic systems
- Solution of equivalent circuits for the hunting of electric machinery
- Determination of characteristics and performance of variable-ratio frequency-changer sets; and
- Solution of equivalent circuits representing field equations of mathematical physics

Apart from the applicability of the analyser to power system problems, some of which are briefly enumerated above, there are several significant special scientific applications which point to a wide field of the analyser's usefulness.

Among these applications may be mentioned the compressible-fluid-flow problem for either subsonic or supersonic flow; the equivalent circuit for an elastic field; equivalent circuits for elastic structures; study of the propagation of electro-magnetic waves; the Schrödinger amplitude equation for one, two and three independent variables in orthogonal curvilinear co-ordinate systems; study of certain mechanical vibration problems; hunting in certain electro-mechanical systems; the process of "scanning" the hunting network; solution of hunting problems associated with power selsyns; solution of the problems of doubly fed machines; developments of equivalent circuits for electrical machinery for hunting and unbalanced operation, etc.

The well-known analogies among mechanical, electrical, acoustic, thermal, and other systems would suggest many possibilities. If researches could be undertaken and the complete science of hydraulics be built up into equivalent electrical analogies, the possibilities of adapting the analyser to solutions of problems over the entire field of power engineering would be great.

The network analyser, which is being installed in the Power Engineering Department at the *Indian Institute of Science*, and which is expected to be ready to function by early 1950, consists of a power supply system embodying 16 generator units, each consisting of 2 power selsyns for independent phase-angle and voltage magnitude adjustments; 16 synchronous impedance units for use with the generators to represent synchronous impedances of generators in actual system; 100 line units consisting of adjustable resistors and reactors, intended for representing lines or other circuits of relatively small impedance; 50 load units also consisting of adjustable resistors and reactors which may be connected either in series or in parallel (the impedances of these units are much higher than those of the line units); 48 capacitor units to represent line capacity, synchronous condensers, or low valves of negative reactance; 32 variable ratio auto-transformer units for stepping up or down the voltage at different points in the network; 8 mutual reactor transformer units with 1 : 1 ratio used in conjunction with a line unit to represent the mutual impedance of parallel circuits.

In addition, there are jumper circuits to serve as zero-impedance ties between points in the network or as connecters for metering purposes. They are useful to represent bus-tie breakers, or the terminals of equivalent "pi" transmission lines, to tie different phase-sequence networks together during certain types of fault studies to totalize currents of two or more circuits connected to a bus, and any other purpose that requires a zero-impedance tie.

The A.C. network analyser will solve efficiently many problems which cannot be handled practically by any other means. It is hoped, the analyser will be constantly in use by the Central, Provincial and State Electricity Planning Commissions, Boards and authorities, and also by private electricity authorities and industries. Such network analysers in the U.S.A. and in the U.K. are kept in constant operation, even working on three shifts a day, for the solution of various problems in the power system development in these countries.

There is another type of analyser developed for studies, such as the power system electric transient phenomena. This is the transient network analyser and permits studies of problems involving switching with restriking, transmission-line or similar distributed-constant systems, non-linear impedances such as saturable reactors and non-linear resistors, and circuits involving commutation or discontinuities such as rectifiers and inverters. It is a unique calculating device for nemerous problems involving distributed constants, non-linearities and discontinuities which it can handle in either the transient or steady state readily and quickly. The problem need not be an electrical one primarily, but may be any problem which can be reduced to an appropriate electrical equivalent. This type of analyser is not of immediate necessity, but its installation in not too distant a future is visualized.

Practical Working of a Laboratory Equipment for Thermionic Valve Production

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Introduction

THERMIONIC valves have now wide and varied uses in many fields such as radio and line communication, various forms of industrial control, electrotherapy, television, radar etc. This has led to the development of many varieties of valves from the simple diodes and triodes to tetrodes, pentodes, hexodes, octodes and their combinations. They vary in size from a tiny acorn, barely an inch long, to the giant transmitting valves, 4′ high. They may have high vacuum inside or may be filled with vapour as in thyratrons, tungars, etc. The end of this amazing development of new types of valves is, however, not yet in sight. New types of valves with new uses are still being produced. In recent years valves like klystron, magnetron and phasitron, involving entirely new principles for work on microwaves, have been developed. This has been possible only through intensive fundamental research on the one hand and of similar developmental research on the other.

In view of the great importance of radio valves, specially because of its indispensability in appliances used by the communication and defence services, the *Board of Scientific & Industrial Research* in 1942, sanctioned a scheme of research on the laboratory production of radio valves in India. Unfortunately, on account of war conditions, the import of the necessary plant was greatly delayed. In 1945 a pilot plant built by the *Callite Tungsten Corporation*, U.S.A., was made available, and it arrived at the *University College of Science*, Calcutta in 1946. It took some time to put the plant into working condition as a number of its components had been damaged in transit. Work was, however, commenced in September 1946 in spite of the many difficulties such as non-availability of raw materials, etc. The two authors of the present note have successively been in charge of the plant, and they have worked out successfully the more important techniques in the manufacture of thermionic valves. In what follows, a description of the plant and also an account of the details of the various processes involved in the construction of a thermionic valve of type 80 full-wave rectifier is given in detail, and brief references to other processes which can be worked with the plant are also given in the Appendix.

The Pilot Plant

The pilot plant consists of the following :

(A) Main working table with all the accessories
(B) Booster unit, compound vacuum pump
(C) High frequency induction furnace, and
(D) Macleod gauge

(A) *The Main Working Table*—The various components comprising the unit (FIG. 1) are described below :

(1) Double wheel glass cutter — It consists of a cutting disc of carborundum (for soft glass) and another of steel (for hard glass) and is operated by a 110 volt A.C. motor, having 3,400 r.p.m.

(2) Five-burner adjustable cross-fires with ball joints — This comprises the following accessories with a 1/10 h.p. driving motor with reduction gear and universal head socket : (a) flare chuck, adjustable for different sizes ; (b) stem head for making tipless stem ; (c) sealing head for different bulb sizes ; (d) basing head for all types of bases ; and (e) equipment for blowing air through the tubes for making exhaust holes.

When making stems, the glass piece and lead wires are held in position by means of clamps provided and the stem head is rotated between cross-fires by means of a 1/10 h.p. motor. When it is desired to make flares, seal-in or to connect the base, the stem head is removed and is replaced respectively by flare chuck, sealing or basing head, according to the operation desired.

(3) Ribbon burner for melting long glass tubes — This is not of much importance for valve construction, but is essential for making neon signs.

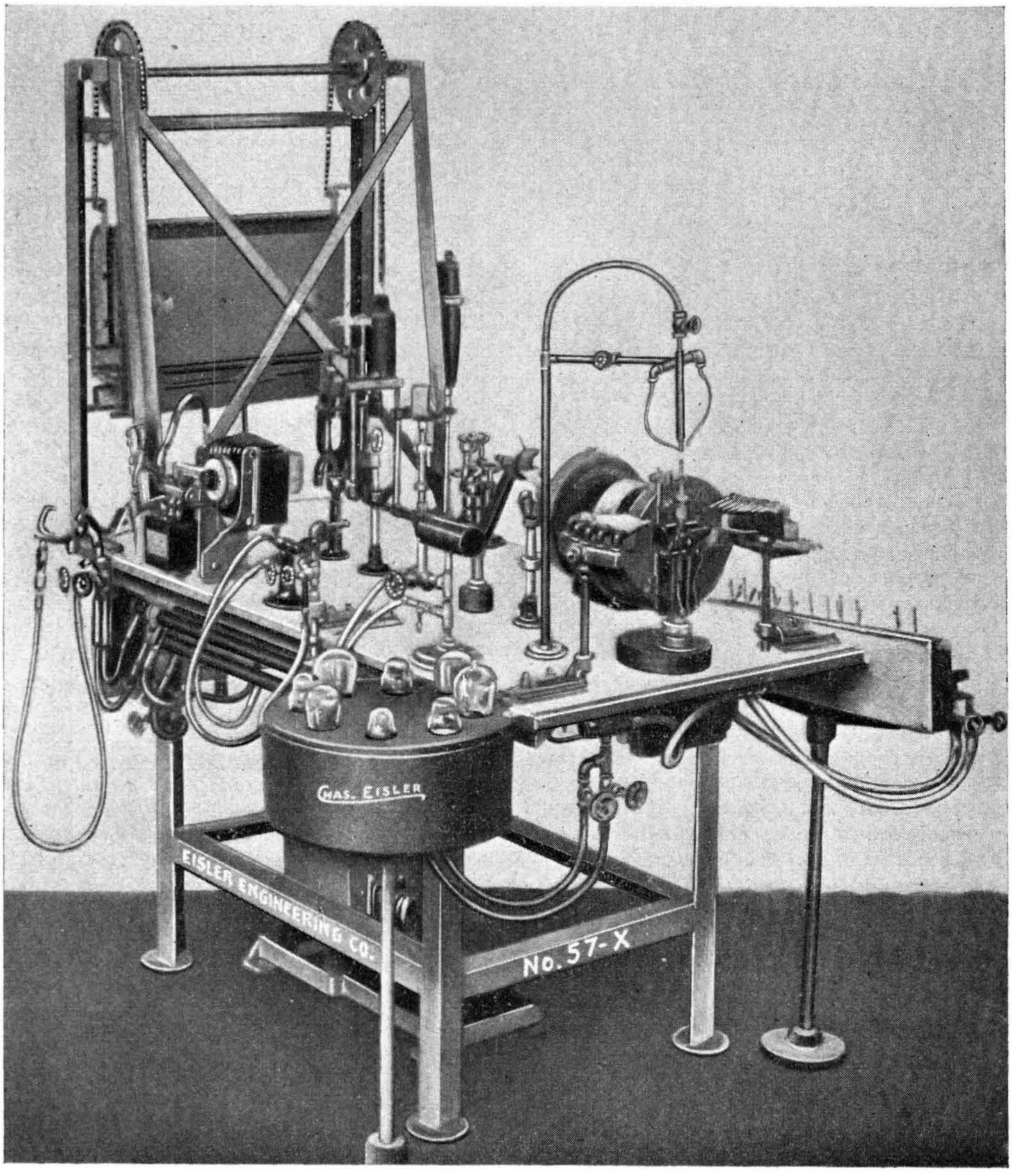


FIG. 1 — MAIN WORKING TABLE WITH ALL THE ACCESSORIES.

(4) Blast torch for glass fusion with arrangement for oxygen-air flame.

(5) Tipping torch for sealing off the valve.

(6) Needle flame burner for localized softening of glass at a point.

(7) Gas heated stem annealer — This consists of a long chamber inside which there are two parallel rows of flames. The distribution of the flames is such that the temperature is maximum at one end of the chamber and diminishes towards the other end. The stem to be annealed is introduced at the hottest end and is taken out at the coolest end. The former is called the inlet and the latter the outlet end of the chamber.

(8) Gas-heated bulb rotary annealer — Useful for annealing of the glass envelopes after they have been sealed with stem.

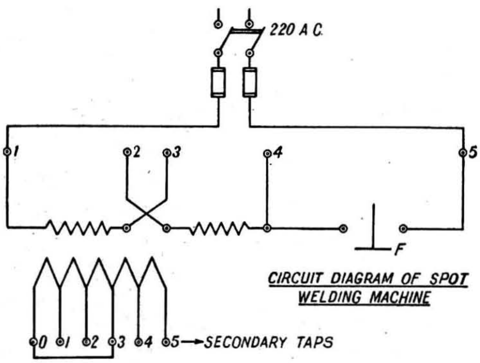


FIG. 2 — CIRCUIT DIAGRAM OF SPOT WELDING MACHINE.

(9) Three-head glass manifold with the gas-heated baking oven — The manifold is connected to the vacuum lines. The valves are sealed in the three exhaust heads and baked during the process of evacuation by the gas-heated oven. The oven can be raised or lowered by means of adjustable weights.

(10) Soldering iron with two burners — For soldering the lead-in-wires to the base socket.

(11) Spot welding machine — This consists of a single-phase power transformer with five secondary taps for varying the voltage in steps of one volt (FIG. 2). The particular voltage to be used depends on the thickness and the type of material to be welded. The welding is effected by the Joule heat generated between the metal surfaces to be welded when the current is passed in the primary of the transformer for a short time. The welder is operated by means of the foot-treadle (F) which closes the primary circuit when depressed. For finer control of welding voltage a rheostat may be included in the primary circuit. A proper weld is made by quick depression of the foot-treadle. For precision and quick working, the time of welding is to be controlled by an electronic circuit.

(12) Leak tester — This is a useful tool for locating leaks and for rough indication of pressure inside the vacuum system. It consists of a transformer, mounted in a cylindrical case provided with a small knob connected to a rheostat at one end and a metallic electrode at the other.

The tester is connected to the 110 volt A.C. line and the knob adjusted until the end of the electrode glows with a spark discharge. When held against the glass tubing of the pumping system, the spark jumps to the glass and partially covers the outer wall of the glass. A poor vacuum is indicated by a dark-purple streak or red glow while a higher vacuum shows a light-blue colour which becomes thinner and clings close to the inside of the glass wall as the pressure decreases. The glow disappears entirely at pressures below 10^{-4} mm. of mercury. A pale-red glow which gradually turns into a dark-purple streak inside the tube indicates a leak. The spark from the tester enters the tubing through the leak and becomes white in colour as it passes through the glass, the leak being indicated by a bright spot.

(B) *Vacuum Pump, Booster Unit, etc.*— This is shown in Fig. 3. A motor (M) operated on 220 volts A.C. works both the compressor for the booster (for supplying gas-air mixture to the burners) and the pump unit through a common coupling shaft. The booster unit consists of 2 rotary compressors, one (C_1) for air and the other (C_2) for gas. The outlets of the compressors are connected to separate chambers of a reservoir drum (D). The pressure within the chambers can be controlled by adjustable mechanical lever arrangement (L) for gas and by an adjustable spring valve (S) for air. The unit supplies gas at 12″ pressure on the water column — approximately equal to 7 oz. of pressure, and air at a pressure of 3 to 4 lb.— which are the conditions for best results. The compressed mixture of gas and air is circulated to various burners in the main working table.

The fore-pump (F) gives a vacuum of the order of 10^{-4} mm. of Hg. This is followed by a mercury diffusion pump by means of which a pressure of the order 10^{-6} mm. of Hg. can be obtained using a liquid oxygen freezing trap. The main vacuum line is a pyrex tube of wide bore to give the least possible resistance during pumping, and it is connected to a soft-glass manifold by a short connecting tube. Soft glass is used for the manifold to facilitate the sealing of the lead glass stem of the valve with the manifold heads.

(C) *High Frequency Induction Furnace* — The degassing of metallic parts is effected by heating in an induction furnace of spark oscillator type. The circuit diagram is shown in Fig. 4. The load to be heated is placed inside the coil (L) which is made of a hollow-copper tube. The coil together with the condenser and the load forms the tank circuit. The latter is coupled to the main spark generator circuit composed of

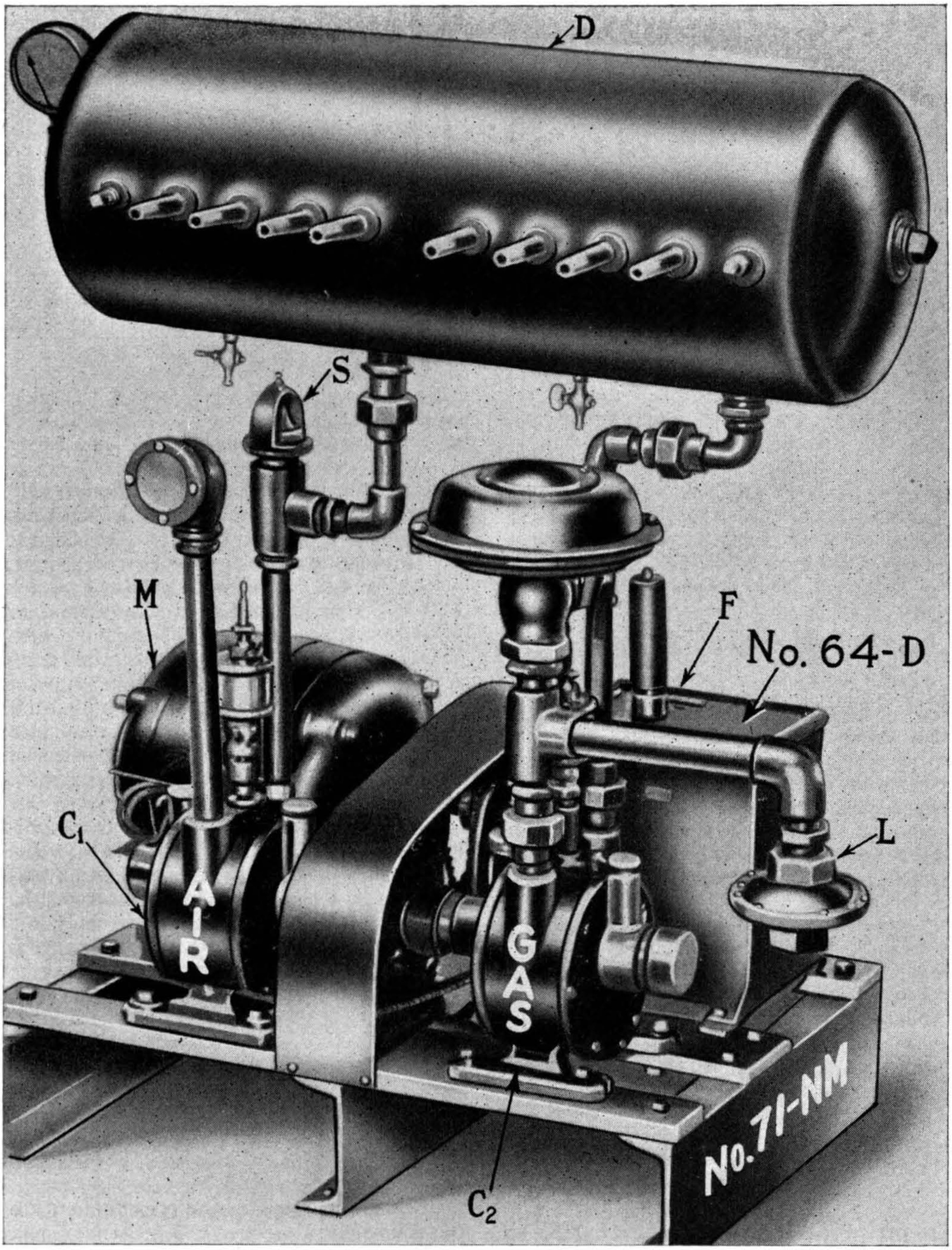


FIG. 3 — COMPRESSOR AND VACUUM PUMP UNIT — D : reservoir drum ; M : motor ; S : spring valve ; F : fore-pump ; L : mechanical lever ; C_1 : compressor for air ; C_2 : compressor for gas.

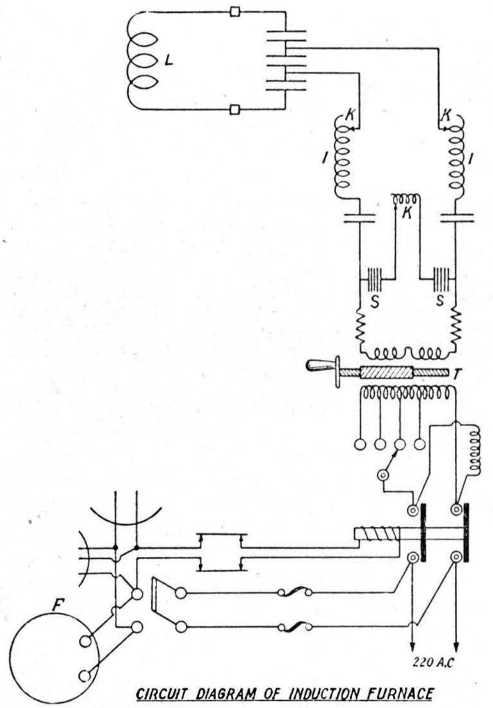
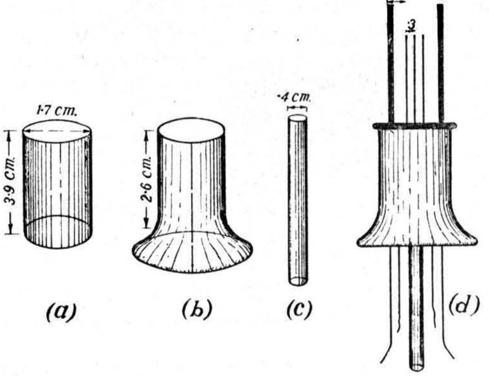


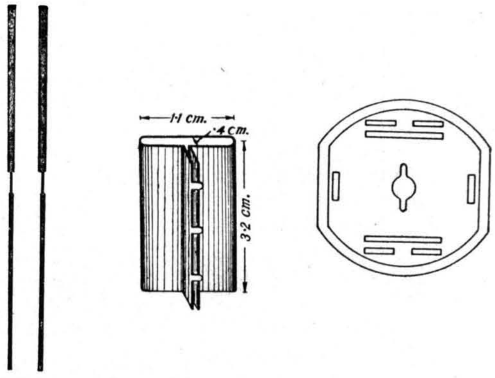
FIG. 4 — CIRCUIT DIAGRAM OF INDUCTION FURNACE — T : input transformer ; SS : spark gaps ; KK : tappings on the inductance coils ; II : inductance coils ; L : heating coil.

condenser, inductance and a set of tungsten spark gaps (SS). The tappings (K) on the coils can be adjusted for tuning. This adjustment is necessary because the heater coil has often to be changed in accordance with the size of the load. Inductance (II) in the spark circuit may be varied within a range of 5 to 18 μh. The input transformer (T) supplies power to the spark circuit. There is an adjustable magnetic shunt between the primary and the secondary coil of the transformer with the help of which the output of the furnace can be varied from 2·5 to 5·5 kVA. A thermocouple type of h.f. power metre coupled to the tank circuit serves as an indicator for the resonance condition of the two circuits. The width of the spark gap has to be adjusted to a value of 0·003″ to 0·004″ from time to time.



(a) (b) (c) (d)

FIG. 5 — STRUCTURAL PARTS OF TYPE 80 FULL-WAVE RECTIFIER — (a) Glass piece for flare ; (b) glass flare ; (c) exhaust tube ; (d) glass flare stem with lead-in-wires sealed.



(a) (b) (c)

FIG. 6 — STRUCTURAL PARTS OF TYPE 80 FULL-WAVE RECTIFIER — (a) " Dumet " lead-in-wires ; (b) anode plates of nickel ; (c) mica spacer.

(D) *Macleod Gauge*— It is used for recording pressure up to 5×10^{-5} mm. of Hg. The mercury diffusion pump, using liquid oxygen in the freezing trap, gives a pressure of the order of 10^{-6} mm. For accurate recording of higher vacuum, an ionization gauge is essential and is being constructed.

Construction of Type 80 Full-wave Rectifier

Type 80 is a directly heated, high vacuum, full-wave rectifier. It has got a maximum peak inverse voltage of 1,400 and gives a maximum D.C. output current of 125 m.a. Its filament consumes 2 amp. at 5 volts input. The constructional details of the valve are shown schematically in Figs. 5, 6, etc.

The various steps in the construction are the following :

(A) Construction of the glass stem with lead-in-wires ; annealing
(B) Construction of electrodes
(C) Cleaning and degreasing of materials ; outgassing of metallic parts ; carbonization of electrodes

(D) Construction of filaments
(E) Assembly of electrodes by spot welding; hooks and supports ; mica spacers
(F) Sealing of glass envelope
(G) Evacuation and processes during evacuation, which include : (1) degassing, (2) cathode activation, and (3) gettering and sealing
(H) Testing of finished valves

(A) *Construction of Glass Stem with Lead-in-wires*— The different stages in making of the glass flare stem are shown in Fig. 5. A short glass piece (a) is first given the flare shape (b) by means of the flare head. The exhaust tube (c) and the lead-in-wires, fixed in the lead wire spacer, are then fused to the flare (d) by means of the stem head shown in Fig. 1. The exhaust hole at the terminal of the exhaust tube is then blown by means of the equipment shown in Fig. 1 by the combined effect of heating at a point and blowing air through the exhaust tube. The working temperatures for the different operations are shown in Table I.

TABLE I

OPERATION	WORKING TEMP., SOFT GLASS °C.	WORKING TEMP., HARD GLASS °C.
Flare	800	1,000
Stem	1,000	1,250

To prevent weathering due to the electrolysis effect resulting from the deposition of moisture on the bulb, the glass must possess a high electrical resistance, must be well annealed, must have a low working temperature and maximum lead-wire spacing. In our case, soft glass of the following composition was used, since it matches the thermal expansion while sealing with the lead-in-wires. Composition of soft glass : SiO_2, 57-72% ; Na_2O, 5-8% ; K_2O, 7-12% ; PbO, 26-32% ; B_2O_3, 0·2% ; Al_2O_3, 0·2-0·8% ; Sb_2O_3, 0·5%.

Lead-in-wires (FIG. 6a) are fused to the stem by the use of proper seals. In selecting the metal-in-glass seals the guiding principle is that when cooling after heating, the metal should contract at a slightly smaller rate than the surrounding glass and that the metal should have a good thermal conductivity. Obviously, this calls for different seals for soft glass (soda and lead glass) and hard glass (Pyrex and Nonex). For soft glass, " dumet " seals, which we use, satisfy the conditions of good thermal conductivity, quick dissipation of heat, fairly cool operation and the required matching of thermal expansion with lead glass. " Dumet " seals consist of nickel-iron alloy with 48 per cent of nickel with a layer of copper which gives a better bondage with lead glass. Covering the wire with a layer of borax serves as a protection from excessive oxidation during sealing. " Dumet " seals, however, have complex expansion characteristics (radially $10{\cdot}25 \times 10^{-6}/0$°C. and longitudinally $7{\cdot}6 \times 10^{-6}/0$°C.). The effect of this can be minimized by the use of pieces of very short length. The uses of other types of metals in glass seal are given in Appendix A.

The different types of stems in use in modern valves are described in Appendix B.

Annealing is essential for removing any permanent strain set up within the material of the glass in cooling. If allowed to remain, it causes cracks when the finished product is put into service. The following two temperatures are important in connection with annealing.

Annealing Point — This is the temperature at which the viscosity is $2{\cdot}5 \times 10^{13}$ pois and 90 per cent of the strain is removed in 5 minutes from a glass sample of thickness ¼". Physically, this represents the point at which there is sufficient mobility within the glass structure to permit the movement necessary to relieve any strains set up within a definite period of time (15 min.).

Strain Point — The strain point is the temperature at which the viscosity is $4{\cdot}0 \times 10^{14}$ pois and at which 90 per cent of the strain is removed in 4 hours in a glass sample ¼" in thickness. Physically, this represents the temperature below which no permanent strain is introduced at ordinary rates of cooling and above which annealed glass cannot be heated again without introducing permanent strain and requires re-annealing to free it from strain.

The finished work is first heated above the annealing point ; the temperature is then gradually lowered following a schedule which depends on the hardness of the glass. The approximate schedule can be determined with the help of the equation given in Appendix D. In our case the approximate values of the annealing point and the strain point were 430°C. and 390°C. respectively. The stem immediately after construction is placed for a short while at the end of the annealing chamber where the temperature is slightly above 430°C. and annealed for about 5-10 minutes placing it at gradually lower temperatures in the annealing chamber. The average temperature of the chamber is 400°C. The best annealing temperature and time were determined by repeated trials. It was found that it is best to cool comparatively slowly since the glass may crack, even though no permanent strain is introduced. A well-annealed glass has 2·5 to 3·0 times the electrical resistance of an unannealed glass.

(B) *Construction of Electrodes* — Anodes of the shape and dimensions shown in Fig. 6b were made from sheet nickel with the help of an ordinary dice and press machine. The choice of nickel for such valves is determined by the following considerations.

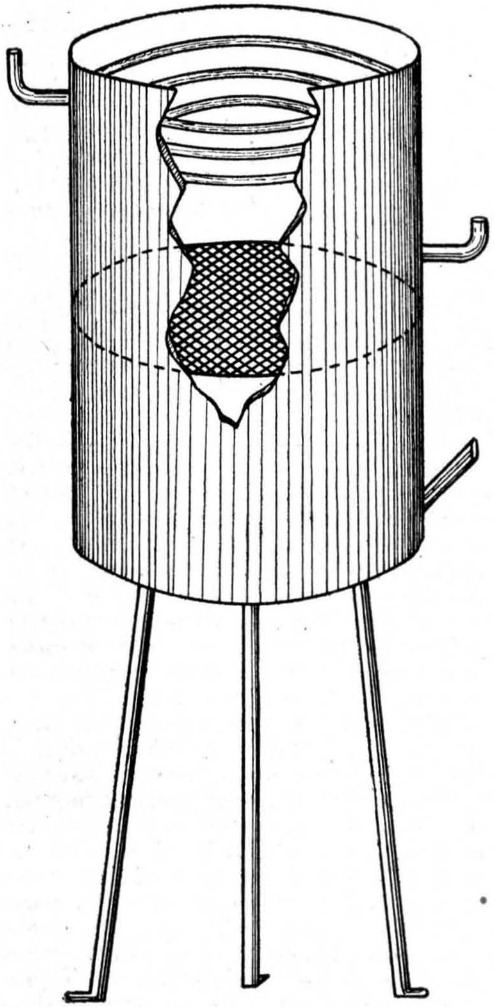


FIG. 7 — TRICHLORO-ETHYLENE DEGREASING CHAMBER.

High ductility, permitting construction of anodes of complicated shapes with less difficulty ; freedom from transformation of the crystalline structure resulting in brittleness ; low vapour pressure permitting high frequency heating in vacuum at 100°C. ; low solubility of carbon permitting carbonization to increase heat dissipation ; good strength and stiffness. Nickel, free from sulphur, is to be used since sulphur causes the grains of nickel to be separated by films of brittle sulphide.

It is to be noted that nickel is now generally replaced by nickel-plated steel although it is difficult to obtain an evenly distributed heavy plate of nickel on strip steel.

Iron and steel are used for cheapness but have the disadvantage of forming carbides during carbonization.

(C) *Cleansing & Degreasing of Materials : Outgassing of Metallic Parts & Carbonization of Electrodes* — It is essential that the glass tubes and the anode material be free from dirt, specially grease or oil. The slightest amount of grease or oil absorbed on the surface layer of the electrodes seriously hinders the attainment of high vacuum. Apart from this, subsequent heat treatment may burn the oil on the metal and spoil its surface or even affect its composition.

The glass tubes before use for flare or stem making or for bulbs for sealing into the stem, are, therefore, washed thoroughly with chromic acid, then with ordinary water and finally with distilled water and dried. Trichloro-ethylene, one of the best grease solvents known, was used for degreasing metallic electrodes. It has a low boiling point (87°C.), low specific heat, low latent heat and has the decided advantage that it has no action on common metals. Degreasing by this chemical leaves the metal in a dry, neutral condition ready for succeeding treatment.

There are many types of degreasing plants used in industry. For our purpose, a simple device was designed and constructed in the laboratory (FIG. 7). It consists essentially of a cylindrical chamber divided into two compartments by wire gauge. In the lower chamber, a small quantity of trichloroethylene is boiled by means of a gas flame. Oily metallic parts are placed on the wire gauge. Cold water is circulated through pipes at the top which condense the trichloroethylene vapour from the degreasing chamber. The metallic parts are thus exposed to a vapour bath which dissolves the oil and falls into the sink. This treatment is

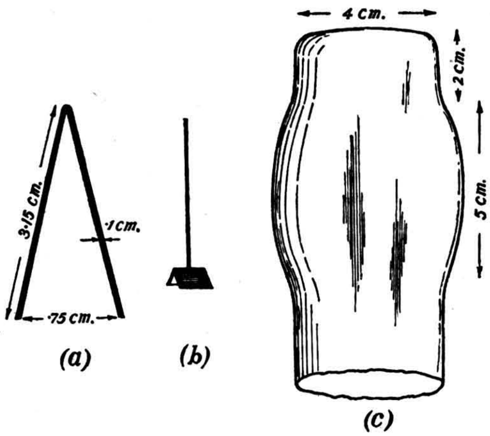


FIG. 8 — STRUCTURAL PARTS OF TYPE 80 FULL-WAVE RECTIFIER — (a) Oxide-coated filament; (b) getter tab of nickel; (c) glass envelope.

continued for half an hour by which time all traces of grease or oil are washed off. A thermometer can be introduced by the side hole for measuring the temperature.

It is also necessary to outgas the metallic parts before assembly. Unfortunately we were not able to do this for want of a proper vacuum furnace (this is under construction). The method of outgassing metallic parts before assembly is described in Appendix C.

The anode is given a coating of carbon. A carbonized anode keeps a lower temperature on account of efficient radiation. Heat dissipation is then increased and permits the use of a smaller anode area for the same dissipation thereby reducing the cost. Coating is given by spraying " aqua dag " (colloidal graphite) on the anode surface by a spraying gun. For securing a good deposit the surface must be properly cleaned.

(D) *Construction of Filaments*—The dimensions and design of the filament as used in the full-wave rectifier, type 80, is like those of standard type 80 valve. The filament is of the oxide-coated type (FIG. 8a). It consists of a strip of nickel of width 0·1 cm. to which is added 2 per cent aluminium. The strip is covered with a mixture of barium carbonate (approx. 40 per cent) and strontium carbonate (approx. 60 per cent). A length, 6·3 cm., of this strip is folded into V-shape. Carbonate coating from a length of about 2·5 mm. is removed from each end in order to facilitate spot welding of the filament with the supports. A pair of such V-shaped filaments is used in each of the valves.

(E) *Assembly & Spot Welding* — Great care is taken to avoid contamination by hand in all welding processes. For perfect welding, accurate adjustment and control of welding secondary voltages and the duration of welding is necessary. A blank trial with the metals to be used was carried out to determine the proper welding voltage and welding time. In the actual manufacturing process the welding time is automatically controlled by the use of a electronic circuit. Wires with different melting temperatures and heat conductivities weld together best when their diameters are related as follows :

$$\frac{d_1}{d_2} \doteq \frac{K_2}{K_1} \sqrt[3]{\frac{T^2m_1}{T^2m_2}}$$

where d is the diameter, Tm is the melting temperature and K is the heat conductivity.

This shows that for wires of the same material, welding will be more satisfactory when the diameters of the welded elements are equal.

This is found to be so in actual practice. The following Table II will serve as a rough illustration of the fact.

TABLE II

ELEMENTS	BEST WELDING VOLTAGE	TIME OF CONTACT
0·5×0·5 mm. nickel wire	Step No. 3	Normal*
0·75×0·75 mm. nickel wire	Step No. 4	Normal
0·5×0·75 mm nickel wire	Step No. 4	Instantaneous (with wire of small diameter below)

* The time of contact is neither too long, nor too short — 0·5 to 1·0 sec.

The anode and cathode supports are first welded in position according to design specifications. Mica insulators (FIG. 6c) cut according to the design are then fixed to the electrode for maintaining their relative spacings. The filaments are then shaped and welded to the support. Utmost care has to be taken so that the parts are not displaced or contaminated with dust or other external impurities during spot welding.

Nickel getter tabs to be used are first degassed by heating in vacuum and then a short piece of magnesium ribbon is fixed on it. In order to avoid any deposit forming on the electrode bounding system and thus short circuiting different electrodes, the getter deposit is directed towards the bottom of the bulbs by a suitable design and placing of the getter tab (FIG. 8b). To avoid ionization by electron bombardment of the gaseous molecules, which invariably remain in the residual getter, the getter is fixed to the filament supports to have the cathode potential.

Support & Hooks — Support and hooks should have sufficient ductility, thermal conductivity, high modulus of elasticity, stiffness and strength. Nickel has all these properties and is used as support wires.

Mica Spacers — Muscovite mica has good insulating properties and chemical stability up to a temperature of 500°C. and has been used by us. Above this temperature, however, muscovite mica rapidly decomposes with the liberation of water vapour — one of the most harmful gases in the valves. Phlogopite mica can be used up to a temperature of 800°C. For valves which operate at still higher temperatures, alumina, magnesia or steatite pressed from powdered material to the required shape and sintered at 1,500°-1,800°C. is used. Since mica contains 18 per cent combined water, it was baked at 200°C. before use.

(F) *Sealing of Glass Envelope* — Soda-lime glass of the composition: SiO_2, 69-73%; Na_2O, 13-16%; K_2O, 0·7%; CuO, 6·1-13%; MgO, 3·2%; B_2O_3, 1%; Al_2O_3, 1-3·5%; Sb_2O_3, 1% is used for glass bulbs (FIG. 8c) because of its cheapness and because it makes a good seal with the lead glass stem. Glass envelopes were supplied by *Bengal Electrical Lamp Works Ltd.*, Calcutta, and blown in our laboratory according to design and specification. Bulbs must be properly cleaned to avoid any stain formation for the following reasons :

(a) Glass heated to 400°C. evolves a small amount of hydrochloric acid which reacts in vacuum with the carbonate or the oxide of the cathode to give barium or strontium chloride.

(b) These chlorides, subsequently being heated, evaporate and condense on the grid and the anode and form stains.

(c) Under electron bombardment, the stain decomposes producing chlorine atoms or chlorine ions which poison the cathode emission to a pronounced degree. All traces of hydrochloric acid should, therefore, be removed before sealing.

The following cleaning schedule was found satisfactory :

The glass envelope is washed with chromic acid, then with distilled water and finally rinsed with caustic potash solution. It is then washed with distilled water, dried and sealed by the sealing head referred to in Fig. 1. The working temperature during sealing-in varies from 1,050°C. to 1,300°C. During the sealing-in of the stem with the glass envelope, care is taken not to dislocate any part. After sealing, it is annealed for 10-15 min. in the bulb-annealing chamber (annealing temperature nearly 500°C.). The complete structure of the valve before being evacuated is shown in Fig. 9.

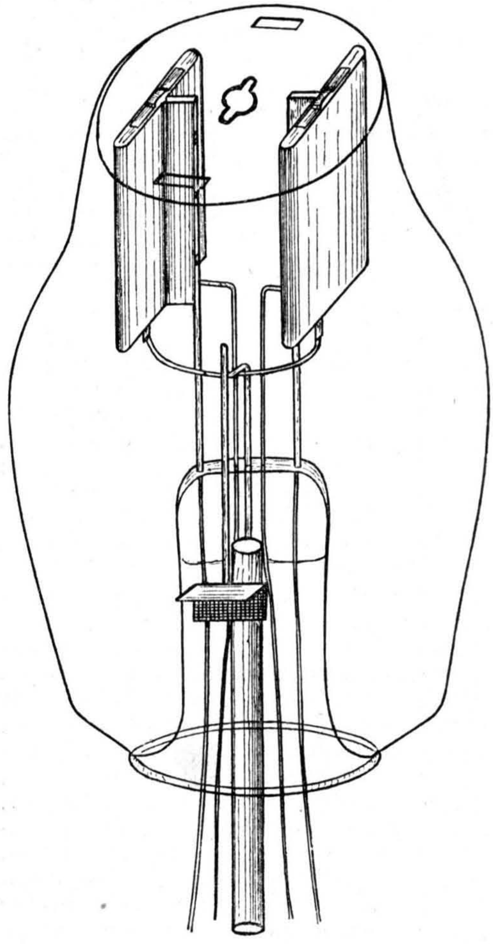


FIG. 9 — A TYPE 80 VALVE WITH ALL PARTS ASSEMBLED BEFORE EVACUATION.

(G) *Evacuation & Other Processes During Evacuation* — Rubber tubing connections are avoided as far as practicable in the vacuum line as they are often leaky and give out sulphur vapour in high vacuum which is harmful to radio tubes.

Water vapour to some extent is removed by using silica gel in a reservoir in the pumping line.

Leaks in the whole system are traced from time to time by the help of the spark coil described before.

The slightest trace of grease creeping in the pumping line is sufficient to destroy the vacuum. Use of grease in the stop cocks is avoided as far as possible and "apeizon" grease is used for high vacuum pump line. A valve has to be subjected to the following processes during evacuation :

(1) *Degassing* — There is always the danger that after setting up there remains some contamination of the surface. On glass walls, this is mainly water vapour. On metallic parts it is usually oil and grease. All parts are, therefore, carefully cleansed before assembly. In order to attain the highest vacuum it is necessary to heat the radio tubes on the pump.

First, the system is baked for half an hour at nearly 450°C. in a gas-heated oven to remove the vapour and gases adsorbed on the glass envelope and electrode parts at 10^{-6} mm. pressure. For lead glass, the temperature should be 400°C., and for pyrex glass, 600°-650°C.

The nickel plates are then heated by induction heating coil to bright red heat 3 or 4 times for 10-15 sec. at intervals of 2 min. to remove the last trace of gas and heating is stopped only when there is no bluish-violet discharge of nickel vapour due to high frequency heating. The gas extracted from nickel is mainly carbon monoxide, carbon dioxide, hydrogen, small quantities of nitrogen and water vapour.

An idea of radio frequency power N_T necessary to raise a metallic electrode to a specified temperature $T°$ (abs.) may be obtained from the following formulae :

$$N_T = 5{\cdot}75\times10^{-12}\,T^4.a \text{ watts}$$

$$N_T = 2{\cdot}80\times10^{-4}A\sqrt{f\mu\rho_t}\,(\,nI\,)^2 \text{ watts}$$

where a is surface area of the metallic electrode ; A, constant depending on the height of the cylinder to be heated ; its diameter ; and diameter of the eddy current heater coil (the thickness of the coil is negligible if below 0·3 mm.) ; f, frequency in cycles/sec. ; μ, magnetic permeability ; ρ_t, specific resistance of the material heated in ohms/cm. ; n, number of turns in the coil ; and I, r.m.s. current in the coil.

Precaution was taken to see that (a) the temperature of heating was not above the point where the vapour pressure of the metal is greater than 10^{-7} mm. of Hg. as otherwise volatilization of the metal will begin ; (b) the pumping speed was made sufficient to remove as quickly as possible the gas evolved during heating as otherwise the gas evolved may be ionized, giving rise to a discharge under the excitation of high frequency heating.

(2) *Cathode Activation* — Activation of filament means the proper formation of the surface of the cathode to give the required emission. In the case of oxide cathode, we use, barium-strontium coating is first decomposed by flashing the cathode. This is accomplished by heating the filament for a short time by a suitable filament power supply, which gives about 50 per cent input power above the normal. The carbon dioxide evolved is pumped out. The flashing time is critical and varies from 10 to 90 sec. The filament is immediately heated by 5 volts A.C., which is the normal filament operating voltage. Care is taken to see that the normal operating conditions are maintained from this time onward, because the filament is now in the active stage and any adsorption of gas due to cooling might spoil the life and activity of the filament. The critical time of flashing for the decomposition of carbonate coating for perfect emission is still under investigation.

During the decomposition of the carbonate coating, the eddy current heating of the anode and electrodes is resumed while the filament is still hot to remove any surface contamination due to carbon dioxide and free barium liberated from the filament during evacuation. The normal anode voltage is then applied. The anode current slowly rises and attains the value appropriate to the applied anode voltage in about 15 min. The building up of current is attributed to the gradual formation of an active layer of barium atoms on the surface of the filament through an electrolytic process.

(3) *Gettering Technique* — An appreciable amount of residual gas remains even after the above operations. There also remains some absorbed gases in the valve elements. These are liberated during the subsequent stage of operation, and unless these are removed, the valve will lose the vacuum. Chemically active substances called "getters" are used to absorb the residual gases. By the use of proper types of getters a vacuum of the order of 10^{-7} to 10^{-8} mm. can be easily maintained.

Gettering is carried out by any of the following processes :

(a) Gettering by dispersion, in which the vaporized metal combines rapidly with the residual gases in volume and the surplus of vapour settles on the cooler section of the bulb electrode.

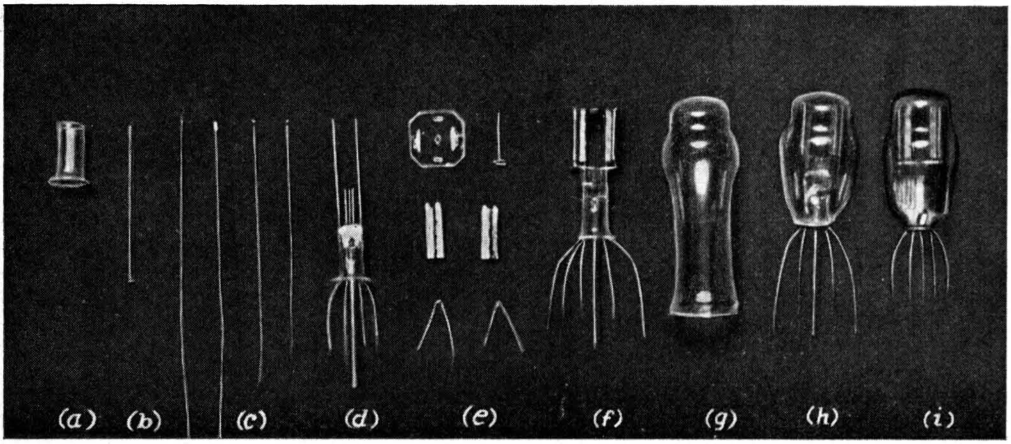


FIG. 10 — AN ACTUAL PHOTOGRAPH OF STRUCTURAL PARTS OF TYPE 80 CONSTRUCTED — (a) Glass flare; (b) exhaust tube; (c) " dumet " lead-in-wires; (d) glass-flare stem with lead-in-wires sealed; (e) mica spacer — getter tab of nickel — nickel anode plates — " V " shaped oxide-coated filaments; (f) a glass flare stem with all parts assembled ready for sealing in glass envelope; (g) glass envelope; (h) valve ready for evacuation; (i) a finished valve with barium gettering.

(b) Gettering by contact, in which the getter deposited on the bulb surface combines slowly with the gases which come into contact with it due to temperature agitation.

(c) Gettering by ionization, during which a rapid chemical action takes place between the getter deposits and ionized gases.

No other getter being available locally or from foreign countries, we are now using magnesium getter which is fairly satisfactory. It getters by ionization. Contact action is slow, being limited by the coating of oxide layer formed. The getter is flashed at a temperature of 500°-600°C. It should be noted that magnesium getter loses its activity if the tube is left unused for a long time.

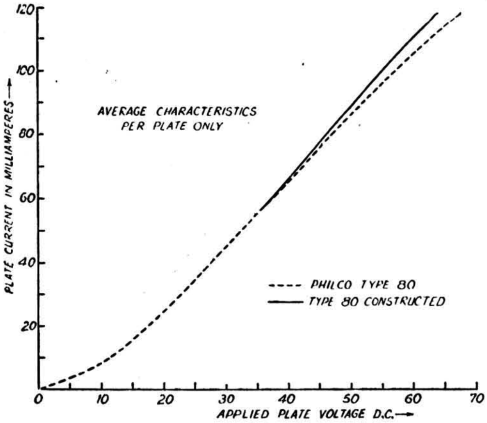


FIG. 11 — EMISSION CHARACTERISTICS OF TYPE 80 CONSTRUCTED COMPARED TO THAT OF *Philco* TYPE 80.

When the pressure is of the order of 10^{-6} mm. after the oxide formation by decomposition, the getter is flashed at a temperature of 500°-600°C. by restricting the eddy current heating to the region of getter tabs for 10-20 sec. and avoiding any undue heating of the other electrodes. Eddy current heating is stopped as soon as the getter is flashed and a good deposit is obtained.

Immediately after gettering, the tube is sealed off the pump line. It is found that a thick sealing maintains the vacuum well. A short account of the more effective getters which are actually used in the manufacture of radio tubes is given in Appendix E. An actual photograph of the structural parts leading to a finished type 80 valve is shown in Fig. 10.

(H) *Testing of Finished Valves* — (a) Emission tests: After sealing the tube off the pump, the valve is subjected to an activation schedule to complete the formation of the oxide-coated cathode. In general, a current of 100 ma. for 5-6 min. is sufficient for complete activation. A graph showing the current-voltage characteristic is prepared (FIG. 11). The graph also indicates the comparison with the characteristics of *Philco* type 80.

(b) Measurement of voltage regulation, peak diode current, diode impedances and

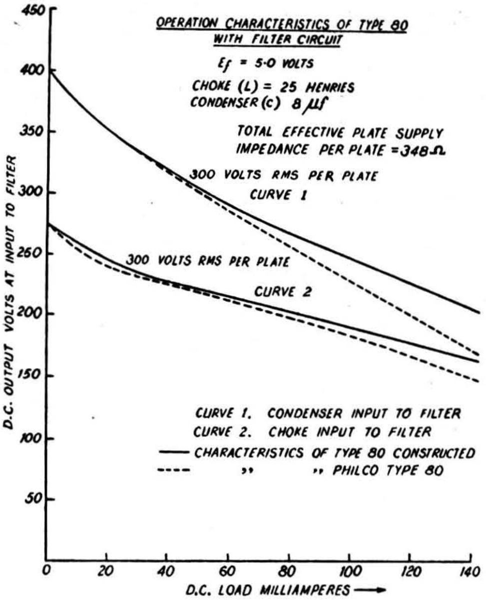


FIG. 12 — OPERATION CHARACTERISTICS OF TYPE 80 CONSTRUCTED WITH FILTER CIRCUIT COMPARED TO THAT OF *Philco* TYPE 80.

plate dissipation : Studies were made with 2 standard types of filter circuits, viz. (1) condenser-input circuit, and (2) choke-input circuit.

A typical set of curves for an input A.C. voltage 300 volts r.m.s. per plate is shown in Fig. 12. The corresponding curves for a *Philco* type 80 valve have been given for comparison. These curves provide a ready method for the measurement of voltage reuglation (ρ), peak diode emission ($\widehat{I}$), peak diode resistance ($\widehat{r}_d$), average diode resistance ($\bar{r}_d$), r.m.s. diode resistance ($Ir_d I$) and total plate dissipation (W). Calculations were made for the following value :

Max. peak voltage, $\hat{E} = 300\sqrt{2}$ volts
External resistance, $R = 348$ ohms
Capacity, $C = 8\ \mu f$
Choke inductance, $L = 25$ Henries

For condenser-input circuit :

Type 80 *constructed*	*Philco type* 80
(i) $\bar{E} = 310$ volts	(i) $\bar{E} = 300$ volts
$\bar{I}_p = 25$ ma.	$\bar{I}_p = 25$ ma.
(ii) $\bar{E} = 247{\cdot}5$ volts	(ii) $\bar{E} = 230$ volts.
$\bar{I}_p = 50$ ma.	$\bar{I}_p = 50$ ma.

For choke-input circuit :

(i) $\bar{E} = 225$ volts	(i) $\bar{E} = 220$ volts
$\bar{I}_p = 25$ ma.	$\bar{I}_p = 25$ ma.
(ii) $\bar{E} = 192{\cdot}5$ volts	(ii) $\bar{E} = 185$ volts
$\bar{I}_p = 50$ ma.	$\bar{I}_p = 50$ ma.

where $\bar{E}$ is the D.C. output voltage and $\bar{I}_p$ is the current per plate.

(c) Measurement of peak inverse voltage : A rough determination of the peak inverse voltage was made by gradually increasing the input A.C. voltage. Peak inverse voltage is given by the sum of the maximum peak input voltage that the tube can withstand and the corresponding D.C. output voltage. The values are recorded in Table III. The method of calculation has been briefly outlined in Appendix F.

TABLE III

QUANTITY	CONDENSER-INPUT CIRCUIT		CHOKE-INPUT CIRCUIT	
	Constructed type 80	*Philco* type 80	Constructed type 80	*Philco* type 80
ρ	0·61	0·74	0·89	0·97
$\hat{I}_p$	(i) 0·165 amp. (ii) 0·255 "	(i) 0·155 amp. (ii) 0·24 "	(i) 0·0594 amp. (ii) 0·108 "	(i) 0·059 amp. (ii) 0·107 "
$\hat{r}_d$	(i) 318·6 Ω (ii) 328·6 Ω	(i) 426·0 Ω (ii) 443·6 Ω	(i) 612 Ω (ii) 457 Ω	(i) 712 Ω (ii) 532 Ω
$\bar{r}_d$	(i) 362·0 Ω (ii) 373·4 Ω	(i) 484 Ω (ii) 504 Ω	"	"
$Ir_d I$	(i) 342·5 Ω (ii) 353·2 Ω	(i) 458 Ω (ii) 477 Ω	"	"
W	(i) 6·12 watts (ii) 3·51 "	(i) 6·38 watts (ii) 4·44 "	(i) 5·768 watts (ii) 7·785 "	(i) 5·89 watts (ii) 7·66 "
Peak inverse voltage	1,363 volts (with 620 r.m.s. per plate)	1,400	1,363	1,400

Discussion of Results

The curve accords with the general characteristics that the voltage regulation is better for a choke-input filter circuit. Compared to *Philco* type 80, the valve constructed by us shows a definitely better voltage regulation in both modes of operation and hence has a lower value of the valve impedance. Possibly, the much better regulation obtained for higher values of output current may be partly due to the presence of a trace of residual gas still left. This, if correct, should result in a lower peak inverse voltage and a diminished life of the valve. Measurement of the former quantity, however, shows that residual gas, if present, has a very negligible effect. No systematic test for life has yet been undertaken by continuous running of the tube although it should be noted that measurements were made about 4 months after the cathode had been activated.

Materials Used in Construction

It will be interesting to examine the list of various raw materials used in the construction of the valve (type 80) and to see how far these materials are available in India. The following are the chief materials employed :

(1) Soda glass bulb, (2) lead glass tube, (3) mica insulator (muscovite or ruby), (4) dumet seals, (5) copper wire, (6) nickel wire, (7) nickel sheets, (8) magnesium ribbon, (9) oxide coated filaments, (10) Kulgrid hook wire, (11) trichloro-ethylene, (12) liquid oxygen, and (13) " Aqua Dag " (colloidal graphite).

Items 1, 3 and 12 are indigenous products and are available locally. Items 5, 8, 11 and 13 are not produced in India but are readily available in the market. All the remaining items have to be imported from the U.S.A. or the U.K. These materials can, however, be produced in India. We propose to develop a suitable process for the production of oxide-coated filaments in our laboratory.

Acknowledgements

It is a pleasure to acknowledge with thanks the helps we have received. We received from *Messrs Metropolitan Vickers Electrical Co. Ltd.*, U.K., nickel sheets, wires and some special types of getters, free of cost, through the kind intervention of Sir Arthur Fleming and Dr. Kendall and from *Messrs Imperial Chemical Industries (India) Ltd.*, Calcutta, free supplies of certain chemicals for degreasing, and also certain data. *Messrs Bengal Electrical Lamp Works Ltd.* have also rendered help in many ways. The work was carried out under the guidance of Prof. S. K. Mitra, D.Sc., and we thank him for his advice and constant interest in the progress of the work.

APPENDIX A

Types of Seal in Use in Radio Tubes

There are two types of seals, viz. (1) metal-in-glass seal ; and (2) glass-in-metal seal.

(1) *Metal-in-glass Seal* — For this class of seals the metal should contract at a slightly lower rate than the surrounding glass. It is made by using " dumet " lead-in-wires (nickel-iron alloy with 48 per cent nickel and a layer of copper which gives a better bondage) with soft glass.

The alloy obtained by substitution of cobalt for nickel in nickel-iron lead-in-wires has lower expansion than soft glass and can be used in hard glass seals.

Molybdenum is found suitable for metal-in-glass seal with hard glass (SiO_2, 73% ; Na_2O, 2·4% ; K_2O, 4·2% ; CaO, 3% ; B_2O_3, 4·6% ; Al_2O_3, 3%).

(2) *Glass-in-metal Seal* — For this class of seals, the glass should contract at a lower rate than the metal after heating. Chromium-iron alloy with 27 per cent chromium has an expansion $10{\cdot}5-11\times10^{-6}/0°C$. which is greater than soft glass and is suitable for soft glass in the metal type of seals.

Tungsten can also be used for this class of seals. Its low thermal expansion matches that of " Nonex " glass (SiO_2, 75% ; Na_2O, 3-4%; K_2O, 1·9%; CaO, 0·3%; B_2O_3, 18·3%; Al_2O_3, 1%) and the adhesion of oxide to tungsten is great enough for making glass-metal seals even for the leads of power and transmitting tubes. To avoid excessive oxidation, the tungsten wire is beaded. Its high electrical conductivity is a decided advantage in seals which are to carry high frequency currents.

An alloy of cobalt-nickel-iron containing 18 per cent nickel, small amount of other impurities and molybdenum, called " Fernico ", is also used in metal tubes.

APPENDIX B

Types of Stems in Use in Radio Tubes

There are 4 types of stems : (1) glass-flare stem ; (2) metal tube stem ; (3) metal tube button stem ; and (4) all-glass button stem.

(1) The glass-flare stem consists of a glass tubing with one end formed into a flare and the opposite end pinched to form the seal containing the leads. The stem length for glass-flare stem in modern receiving valves ranges from 29 mm. to 14 mm. The total clearance usually allowed between the flare and the inside walls of the base ranges from 5 mm. to 2 mm. The clearance between the leads in flat-press stem should be increased to eliminate possible difficulty of electrolysis.

(2) In a metal tube stem, each lead is brought into the tube through an individual metal eyelet and made vacuum tight by means of a glass seal. This is not much used on account of the high cost.

(3) In a metal tube button stem, a glass button is moulded into a metal sleeve. The metal sleeve is welded to a cold, rolled-steel flange. The whole assembly is then loaded onto a mould in which the lead wires and tubing are located, with one short length of glass tubing outside and another inside. The glass is then pressed into the metal sleeve by heating.

(4) All-glass button stem is similar to the metal tube button stem except that the metal sleeve is omitted. The use of this kind of stem reduces the over-all length of the tube slightly and improves shielding.

APPENDIX C

Outgassing of Metallic Parts Before Assembly

Although degassing is done by the induction furnace heating or by electron bombardment during evacuation, previous outgassing of the metallic parts is important in attaining a high order of vacuum and in maintaining the same during the operation of the valve. The common method of outgassing consists in heating the metallic parts in a suitable vacuum furnace under a pressure of 10^{-4} mm. The metal electrode to be outgassed is placed in a molybdenum boat and inserted into the furnace. A temperature of 750°-950°C. is suitable for degassing nickel and its alloys.

Outgassing temperatures for some of the common materials are given below.

Material	*Outgassing temperature, °C.*
Tungsten	1,800
Molybdenum	950
Tantalum	1,400
Platinum	1,000
Copper and copper alloys	500
Graphite	1,500-1,800

Some metals, e.g. molybdenum, platinum, nickel and iron may be cleaned by heating in an atmosphere of hydrogen. The samples are heated at 950°C. in a silica tube furnace containing dry hydrogen at a pressure of 10^{-3} mm. of Hg. Heating in a dry hydrogen atmosphere introduces hydrogen into the metal by adsorption but owing to its high rate of diffusion, it is entirely removed by baking at 450°C. and rendered hydrogen-free.

APPENDIX D

Annealing Temperature

The initial rate of cooling, h_o, in annealing may be expressed as follows:

$$h_o = \frac{BF}{C} = \frac{\Delta N_s}{C}$$

where h_o is expressed in °C./min.; B, birefringence constant; F, stress in kg./cm.; ΔN_s, final stress in optical unit = 2·5 millimicron/mm. of glass thickness; C, constant =

$$\frac{\alpha\,\beta\,a^2}{6k\left(\frac{1}{6R}+\frac{2}{9K}\right)}$$

α, coefficient of expansion; k, thermal diffusivity, $= \frac{\text{heat condutivity}}{\text{density} \times \text{specific heat}}$; R, modulus of rigidity; K, modulus of compressibility; a, semi-thickness of glass.

After the initial cooling, the rate is h_x, where

$$h_x = \frac{h_o}{a}\left(1+2^{\frac{T_o - T_x}{20}}\right)$$

where T_x, new temperature in 0°C.; T_o, original temp. in 0°C., and h_o, initial cooling rate.

Initial cooling rate in terms of thickness for pyrex glass

$$= \frac{\Delta N_s}{3{\cdot}2a^2}$$

The whole of the glass should be maintained at a uniform temperature, higher than the annealing temperature, for a short time after which it is gradually cooled in the annealing chamber through about 100°C. at a rate not exceeding $20/t^2$ degrees per minute (where t is the wall thickness in mm.) to take it below the strain point. A cooling rate of $100/t^2$ degrees per minute should never be exceeded, the cooling being at as uniform a rate as possible. The rate of cooling afterwards can be rapid as no strain is introduced after this stage. Table A gives the temperature ranges for some common types of glass.

TABLE A

TYPE OF GLASS	ANNEALING TEMP., °C.	STRAIN TEMP., °C.	SOFTENING TEMP., °C.
Lead glass	425-435	389-398	126-630
Hard glass	647-818	450-510	697-756
Soda-lime glass	510	475	696

For lead glass, used by us, we have the following values :

$R = 0{\cdot}0040$ sq. cm./sec.

$\alpha = 87 \times 10/0°$C.

$B = 3{\cdot}2 \times 10$ due to 1 kg./sq. cm. load

$K = 0{\cdot}35 \times 10$ kg./sq. cm.

$k = 0{\cdot}24 \times 10$ kg./sq. cm.

C, as calculated according to the above formula, has the value $14{\cdot}85a^2$, where a is the semi-thickness of the glass. Therefore, the cooling rate per minute is :

$$h_o = \frac{\Delta N_s}{C} = \frac{2{\cdot}5}{14{\cdot}85 \times 0{\cdot}0025} = 67{\cdot}5°\text{C./min.}$$

According to the above formula, we have the following values for the type of lead glass used by us (TABLES B and C).

TABLE B

Temperature interval = 20°C.

GLASS THICKNESS 1 mm.		GLASS THICKNESS 1·5 mm.		GLASS THICKNESS 2·0 mm.	
Cooling rate, °C.	Time interval, sec.	Cooling rate, °C.	Time interval, sec.	Cooling rate, °C.	Time interval, min. sec.
h_0 = 67·5	17·4	30	39·6	16·8	1 12
h_{20} = 101·25	11·54	45	26·6	25·2	47·6
h_{40} = 168·75	7·20	75	16·0	42·0	28·5
h_{60} = 303·75	3·70	135	8·8	75·6	15·8
h_{80} = 573·75	2·04	255	4·7	142·8	8·4
h_{100} = 113·75	1·02	495	2·4	277·2	4·3

TABLE C

	GLASS THICKNESS 2·5 mm.			GLASS THICKNESS 5 mm.		
	Cooling rate, °C.	Time interval, min.	sec.	Cooling rate, °C.	Time interval, min.	sec.
h_0 =	10·8	1	48	2·7	7	24
h_{20} =	16·2	1	14·4	4·05	4	54
h_{40} =	27		44	6·75	2	54
h_{60} =	48·6		24·4	12·15	1	36
h_{80} =	91·8		13	22·95		52·2
h_{100} =	178·2		6·7	44·55		26·9

APPENDIX E

Types of Getters in Use in Radio Valves

The following are the types of getters in common use :

(a) *Copper-nickel-iron or Aluminium-clad Barium Getters* — A stick of barium is worked into a wire by rolling or drawing. A 0·25″ length of this assembly is taken and used as getter. Vaporization of the getter occurs at 1,000°C. when the internal pressure is great enough to eject barium vapour together with the molten metal and molten barium from the sheath. Sheaths of nickel, aluminium or iron may be used with equal advantage. This kind of getter is widely used in large glass receiving tubes. Barium gives instead of a silvery, a dark deposit when it absorbs gas. For metal tubes, iron wire having a barium core is taken and a section of this is used as getter. Barium from the interior bursts out through the inner walls permitting the getter to deposit to the section of bulbs in a plane roughly parallel with the thin edge.

(b) *Barium Azide Getter* — A nickel strip with a central hole is dipped into a concentrated solution of barium nitrite in water and then allowed to dry. During the bake out, nitrogen is released at 150°C. and the remaining barium acts as a getter. This getter gives a dark deposit. Barium azide getter is not generally used for it is explosive and azide solution is poisonous.

(c) *Barium-magnesium & Barium-aluminium Alloy* — An alloy of barium may be used as getter owing to the large difference in vapour pressure of barium and magnesium. A large amount of magnesium gets evaporated before barium disperses. The alloy is stable in air if the barium content is 25 per cent. A serious disadvantage of this getter is that gettering by barium cannot be visually observed.

A barium-aluminium alloy with 50 per cent barium can be used as getter with nickel or iron tubes. No aluminium is dispersed before barium and visual observation of gettering is possible. Barium-aluminium alloy has a flashing temperature of 700°-1,700°C. permitting the use of nickel-iron getter tabs. Magnesium may be mechanically mixed with barium-aluminium alloy or a mixture of barium-magnesium and barium-aluminium alloy may be used, for it is advantageous to use magnesium as precleaning agent.

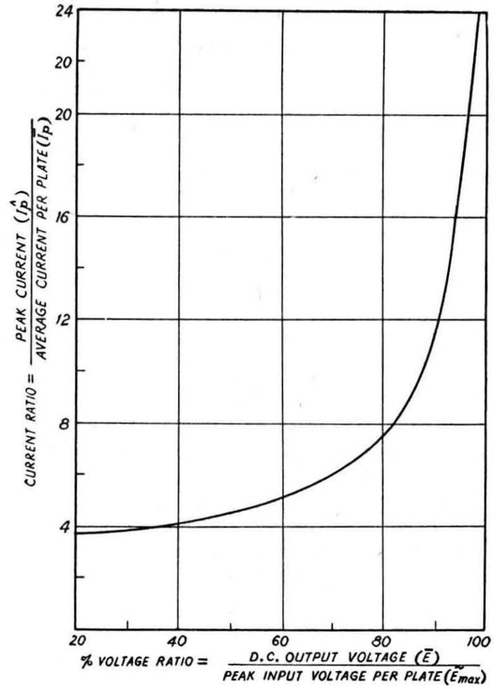


FIG. 13 — CURRENT RATIO AND VOLTAGE RATIO CHARACTERISTICS OF FULL-WAVE RECTIFIER.

(d) *Alkali Metals* — Because of their high vapour pressure, these metals are not suitable as getters in receiving tubes but are used in special tubes like caesium-vapour detector tube, photo tube, vapour lamps and gas discharge devices.

(e) *Batalum & Batalum Ribbon Getters* — These are used in metal tubes. Tantalum wire is coated with barium and strontium carbonates which are converted into oxides at 1,000°-1,100°C. and finally to metal vapour at 1,300°C. The reaction is as follows :

$$8\ BaO + 2\ Ta \longrightarrow 5\ Ba + Ba_3\ Ta_2O_8$$

(Temperature of reaction is 1,300°-1,500°C.) Batalum ribbon getters consist of a tantalum strip with the middle portion formed into a channel. A barium compound with nitrocellulose binder is filled into the channel on the concave side. The use of barium-baryllite removes the difficulty of decomposition of carbonate during exhaust.

(f) *Misch Metal* — It is a mixture of rare earth metals and contains much cearium and lanthanum and, in addition, a variable amount of sodium not exceeding 5 per cent. Misch metal is inferior to barium, since it becomes covered with a monomolecular film of oxide. It is now used in gas-filled regulators as an oxygen getter and as a means to maintain uniformity and magnitude of cathode. fall during the life.

APPENDIX F

Method of Calculation of Voltage Regulation, Diode Impedances, Plate Dissipation, etc., of Type 80 Full-wave Rectifier

By voltage regulation characteristic of a rectifier circuit is meant the relationship between the output voltage and output current. Voltage regulation ρ is defined as

$$\frac{E_{\text{no load}} - E_{\text{full load}}}{E_{\text{full load}}} = \rho \qquad (1)$$

It should be noted that ρ depends on the external resistance induced in the circuit and the type of filter circuit. Studies were made for two standard type of filter circuits :

(1) Condenser-input circuit (FIG. 14A), and
(2) choke-input circuit (FIG. 14B).

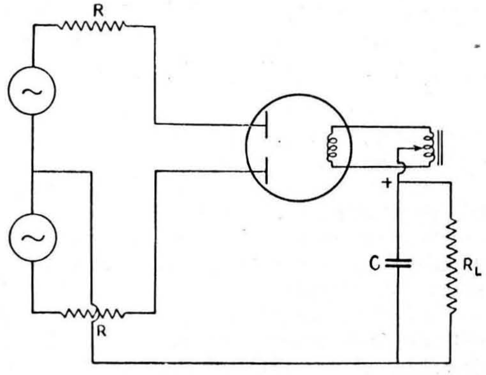


FIG. 14A — CONDENSER-INPUT RECTIFIER CIRCUIT.

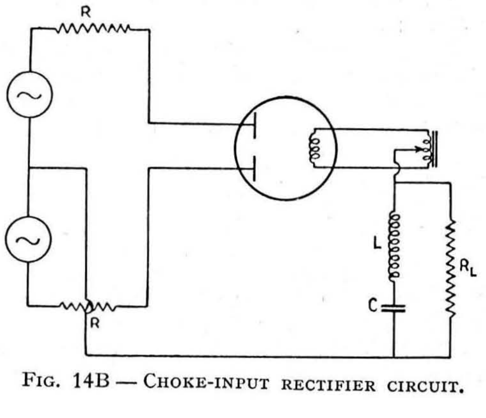


FIG. 14B — CHOKE-INPUT RECTIFIER CIRCUIT.

For measurement of ρ, $E_{\text{full load}}$ was taken at the output current of 100 ma. in eqn. (1).

For the measurement of peak diode current and valve impedances, the cases of the two filter circuits shall have to be treated differently.

Fig. 14A shows the diagram of a condenser-input filter circuit. R represents the stray resistance induced in the circuit due to the input transformer. In this case plate current flows in short pulses during the period for which the A.C. applied voltage is positive at the plate in question and in excess of the D.C. output voltage. The value of equipment diode resistance, therefore, depends on whether we are analysing for peak diode current, average diode current, etc. Thus, peak diode resistance $\hat{r}_d$ is given by

$$\hat{r}_d = \frac{\hat{ed}}{\hat{I}_p}$$

where $\hat{ed}$ is the peak diode voltage and $\hat{I}_p$ is the peak diode current.

The average diode resistance $\bar{r}_d$ is defined as $\hat{r}_d = \frac{\overline{ed}}{\bar{I}}$ where $\overline{ed}$ and $\bar{I}$ are the average diode voltage and current during the conduction period.

R.m.s. diode resistance I r_d I is defined as the resistance in which the power loss due to a current having the same value of r.m.s. diode current is equal to the plate dissipation W_p of the diode.

For all practical cases, the following relations hold within an accuracy of 5 per cent.

$$\hat{r}_d = 0{\cdot}88 r_d = 0{\cdot}93 \mid r_d \mid$$

To determine $\hat{I}$ and $\hat{r}_d$, we proceed as follows :

It has been shown by Kauzmann that if the input condenser is assumed to be very large and the current pulses to be half sinusoidal, the following relation holds :

$$\frac{\hat{I}}{\bar{I}} = \frac{\bar{\Lambda}}{2} \frac{180^\circ}{\text{Cos}^{-1} \frac{\bar{E}}{\tilde{E}}}$$

where $\bar{E}$ is the D.C. output voltage and $\tilde{E}$ the peak inverse voltage.

The relation has been shown graphically in Fig. 13. From this curve $\hat{I}$ can be determined if $\tilde{E}$, $\bar{E}$ and $\bar{I}$ are known. The value of $\hat{r}_d$ can now be determined from the relation $\hat{I}\,(R+\hat{r}_d) + \bar{E} = \tilde{E}$. Knowing $\hat{r}_d$, $\bar{r}_d$ and I r_d I can also be calculated.

Power dissipation per plate is given by

$$W_T = W_p + \tfrac{1}{2} E_f I_f$$

where $W_p = 0{\cdot}84\, \hat{I}\, \hat{r}_d I$ and $E_f I_f =$ filament input power.

Fig. 14B shows the diagram of a rectifier circuit using choke-input filter. Here again R represents the stray resistance due to the input transformer. It can be shown that provided

$$L \geq \frac{0{\cdot}91\tilde{E}}{6\sqrt{2}\,\bar{\Lambda} f \bar{I}}$$

where $L =$ choke inductance in Henries, $f =$ the frequency of the input A.C. voltage, the diode current approximately consists of rectangular pulses (except for a small first harmonic ripple at the top) and flows over an entire half cycle of the applied voltage. For such a case it is clear that

$$\hat{r}_d = \text{I}\, r_d\, \text{I} = \bar{r}_d = r_d \text{ (say)}$$

In our case for example $\tilde{E} = 300\sqrt{2}$

$f = 50$

$\bar{I} = 0{\cdot}1$ amp.

$$L \geq \frac{0{\cdot}91 \times 300}{300\,\bar{\Lambda} \times 0{\cdot}1} \geq 3H$$

The value of inductance used was 25 H and hence the relation is applicable.

Output current for a full-wave, choke-input rectifier, however, contains an appreciable portion of 1st harmonic of frequency 2f. It may be shown that the peak diode current

$$\hat{I} = \bar{I} + \frac{0{\cdot}471\bar{E}\sqrt{2}}{4\bar{\Lambda} f L}$$

No load output voltage is approximately given by

$$\frac{0{\cdot}91\tilde{E}}{\sqrt{2}}$$

Hence for an output voltage $\bar{E}$ at load current $\bar{I}$

$$\frac{0{\cdot}91\tilde{E}}{\sqrt{2}} - \bar{E} = \bar{I}\,(r_d + R)$$

The total plate dissipation is given by

$$W_T = W_p + W_f$$

where

$$W_p = 0{\cdot}5 \left(\bar{I}^2 + \frac{0{\cdot}03\bar{E}^2}{\bar{\Lambda}^4 f^2 L^2} \right) rd$$

and $W_f =$ filament input power.

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Fretting Fatigue — Literature Report No. 1

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FRETTING fatigue, also called rubbing fatigue and chafing fatigue, is a particular type of fatigue failure, which is probably related to both notch fatigue and corrosion fatigue[1,2]. The main characterisitic of these types of fatigue is a combination of two factors, (a) the repeated loads or vibrations which define the load applications as fatigue or endurance and (b) a factor which reduces the fatigue strength and endurance limit obtained in the absence of this damaging factor.

In the case of fretting fatigue, the damaging factor consists of a close contact between the part subject to vibration and another part which is either fitted on to the vibrating part or in gliding contact with it. The most important example of premature failure due to fretting fatigue is that of railway axles on to which the wheel has been press fitted on shrunk[3-16]. Fundamentally the same failure has been also observed on shafts[4,11], and on crank-pins[16,13]. Various types of joints differ from these fitted members, in that none or only a slight pressure is present during rest conditions ; however, the pressures resulting from the vibrations also cause fretting fatigue failures. The most important instance of such a joint is that between an aircraft propeller and its hub[1,19,21-25,49]. Other cases of this type are screws or bolts and nuts[23-25], keyed joints[26], couplings[27], joints between wire or wire rope and clamps[28,29], and attachments of springs[30,31]. Failures between gliding members are those of parts running in bearings[32] and of gear teeth[32]. Possible, the fatigue failures of the bearings themselves are also due to fretting fatigue. However, these will not be considered here. A large variety of fretting fatigue failures have been described by Thum and Wunderlich[33].

Most observations on fretting fatigue relate to mild steel. However, most metals and alloys appear to be susceptible to a damaging effect by fretting. It has been found to be present in aluminium alloys[1,19,22] magnesium alloys[19-22], copper alloys[1,19,22], and stainless steels[1].

The type of fatigue failures observed at contact surfaces are obviously the same for contacts either in relative rest or in relative movement. This is proved by the fact that the laws of fretting fatigue failures have been established by extensive laboratory tests[1,19,27,28,33-42] which are predominantly of the type in which failure occurs at the contact between the rotating specimen and the bearing.

The literature on fretting fatigue has been repeatedly abstracted and critically discussed[2,43-46].

The fatigue failure, under conditions where fretting is present, occurs in all instances either at the edge of the contact area between the two contacting members or close to it but within the contact surface.

The failure may occur in either one or in both contacting members.

In many instances of fretting fatigue, the production of abrasive products has been observed[40,47-51]. This process is usually called " bleeding ", the term being illustrative for the red colour of the abrasion

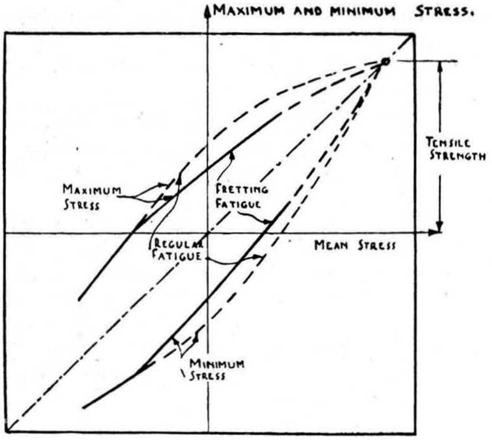

FIG. 1 — DIAGRAMMATIC REPRESENTATION OF THE RELATION BETWEEN FRETTING FATIGUE STRENGTH AND REGULAR FATIGUE STRENGTH FOR VARIOUS CONDITIONS OF AXIAL LOADING.

products of steel. The absence of such abrasion products, however, is no proof that fretting does not occur and that the fatigue strength is unaffected.

The laws of fretting fatigue, as far as they have been recognized, are generally the same as those for notch fatigue and corrosion fatigue[1]. The most important of these laws are the following:

(a) A damaging effect probably occurs only in the presence of applied tensions. On the contrary, it appears that fretting in the absence of tensions, i.e. in the presence of compressions only, would not adversely affect the fatigue strength. This is illustrated schematically in Fig. 1. However, definite information in this respect does not seem to exist at present. The graph in Fig. 1 illustrates that the fatigue strength is reduced less, the more the load condition changes from tensions only (upper right quadrant) to tension-compression, and that the damaging effect is nil in the range of compressions only (lower left quadrant).

(b) The damaging effect for a given load application such as rotating bending is highly dependent upon the material[1]. For certain groups of materials such as aluminium alloys, the fretting fatigue strength has been found, according to Fig. 2, to be equal to the regular fatigue strength (determined for polished specimens) if this value was very low. With increasing regular fatigue strength (or tensile strength, or hardness), the effect of fretting becomes increasingly larger. This results in the fact that for high strength alloys, the fatigue strength in presence of the damaging effect (under conditions of either severe fretting, notching or corrosion) is practically independent of either the regular fatigue strength or the tensile strength.

The fretting fatigue strength is also dependent, according to Fig. 2, upon the structural condition of an alloy, to a slight extent. Cast alloys are apparently more resistant than annealed which, in turn, are more resistant than either cold-worked or heat-treated alloys, on the basis of equal strength.

(c) The damaging effect increases with increasing loads. As shown in Fig. 3, it becomes generally apparent only if the load range is sufficiently low to cause failure after a considerable number of cycles,

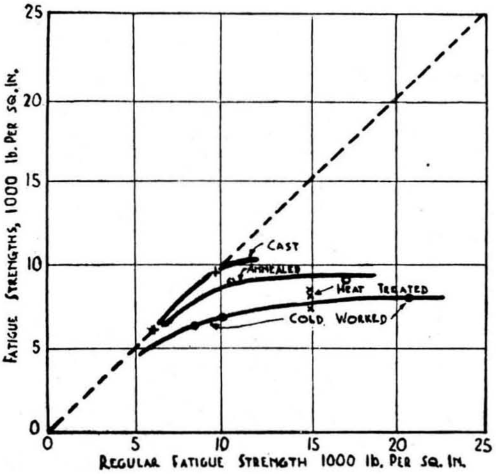

FIG. 2 — RELATION BETWEEN FRETTING FATIGUE STRENGTH AND REGULAR FATIGUE STRENGTH FOR VARIOUS ALUMINIUM ALLOYS (SACHS-STEFAN[1]).

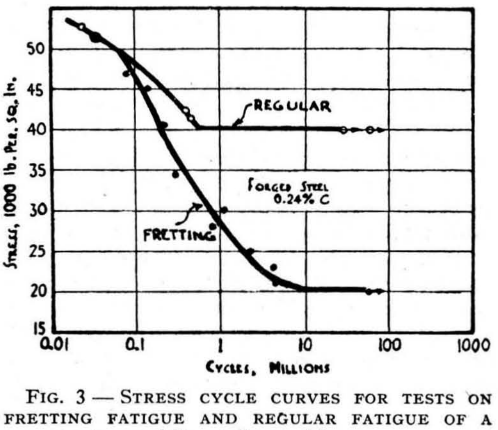

FIG. 3 — STRESS CYCLE CURVES FOR TESTS ON FRETTING FATIGUE AND REGULAR FATIGUE OF A FORGED STEEL (SACHS-STEFAN[1]).

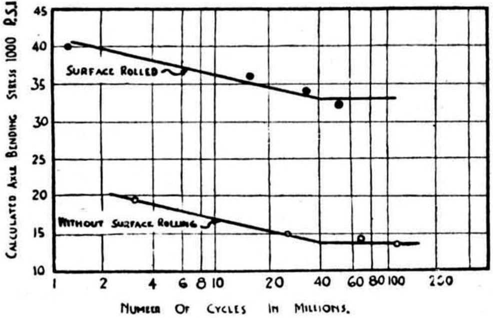

FIG. 4 — EFFECT OF SURFACE ROLLING ON THE FRETTING FATIGUE STRENGTH OF S.A.E. 1045 STEEL AXLES (BUCKWALTER-HORGER[4]).

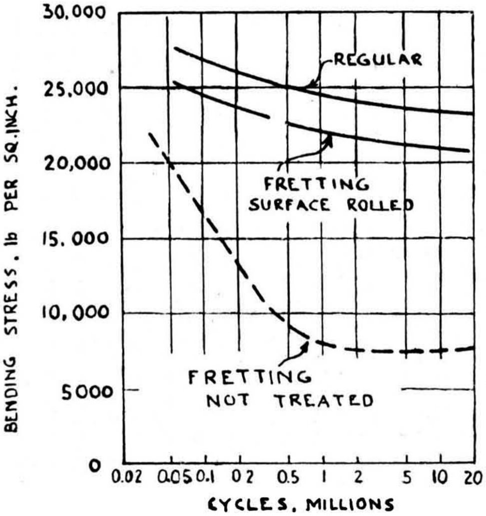

FIG. 5 — STRESS-CYCLE CURVES FOR REGULAR FATIGUE FOR FRETTING FATIGUE AND FOR FRETTING FATIGUE OF SURFACE ROLLED SPECIMENS OF A MAGNESIUM ALLOY (BOWDEN AND RIDLER[50]).

anywhere above 10,000 cycles at least. Furthermore, as shown in Fig. 3, stress-cycle curves indicate that no true fretting endurance limit exists, the value of the fatigue strength steadily decreasing with increasing number of cycles.

(d) Certain mechanical and thermal treatments eliminate the greatest portion of the damaging effects.

The mechanical treatments used to thus improve the life of parts, particularly of railroad axles, are surface rolling with one or several polished rolls,[6,9,26,52-54] and peening or shot-blasting[9,55-57]. The effects of such a treatment is illustrated in Figs. 4 and 5. The improvement resulting from such processes depends upon the processing conditions such as the roll size and rolling pressure in surface rolling[6,21], as shown in Fig.6, or the shot-size and pressure in shot-blasting[55].

By flame hardening the contact surface, an improvement similar to that obtained by mechanical treatments can be achieved[7,9,57].

It appears that these beneficial effects are only attributable to a minor extent to a structural improvement of the material. The major portion of the improvement is explained by the introduction of compressive surface stresses by either the mechanical or the thermal treatments[11,21], which prevent tensile stresses to develop during the vibrations. The relation between the magni-

KEY	SPEC. DIA.	ROLLER SIZE & FEED			S.A.E. 1045 STEEL NORM. & DRAWN
		DIA.	CONT. RAD.	PER INCH	
○—○	0.3"	0.44"	0.09"	224	½ DIA. COLD ROLLED.
▲—▲	1"	1.46"	0.31"	60	1¾" " " " .
■—■	2"	2.92"	0.63"	60	3¼" " " " .

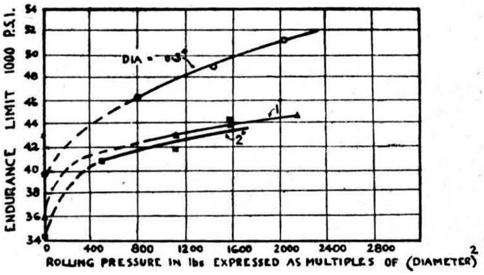

FIG. 6 — EFFECT OF ROLL PRESSURE ON THE FRETTING FATIGUE STRENGTH OF SURFACE ROLLED, S.A.E. 1045 STEEL AXLES OF VARIOUS SECTION SIZES (SACHS-STEFAN[1]).

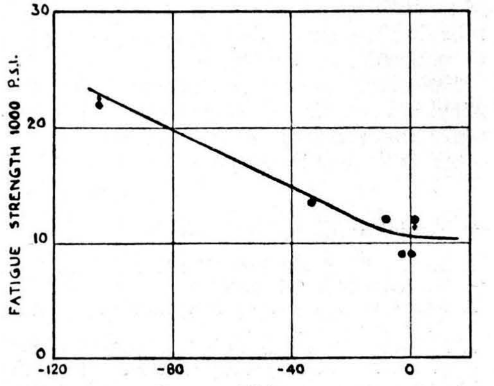

FIG. 7 — RELATION BETWEEN FRETTING FATIGUE STRENGTH AND RESIDUAL STRESSES OF 0·4 TO 0·5 PER CENT CARBON STEELS ACCORDING TO TESTS BY HORGER AND NEIFERT[11].

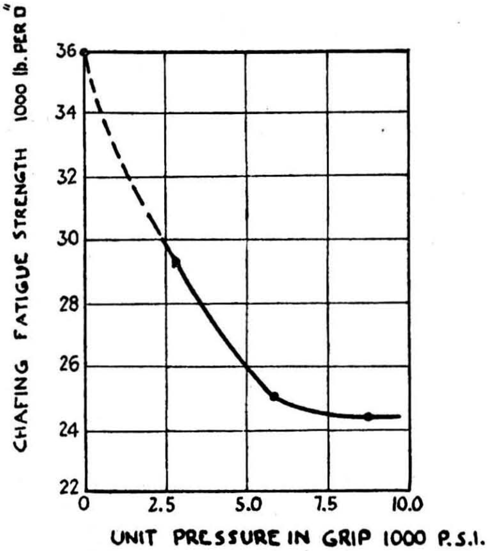

FIG. 8 — THE EFFECT OF CLAMPING PRESSURE ON THE FATIGUE STRENGTH OF CANTILEVER TYPE STEEL SPECIMENS (THUM AND WUNDERLICH[33]).

tude of such stresses and fretting fatigue strength is illustrated in Fig. 7.

Thin, soft inserts between and coatings of one of the contact surfaces also increase the fretting fatigue strength[21,37].

The analogy between fretting fatigue and notch fatigue has been also confirmed by photo-elastic studies of contacting parts[17,24]. These show that the stress distribution in a loaded press-fitted part is nearly the same as that in a single piece of the same overall shape.

This explains that the fatigue strength of press-fitted parts can be raised by stress-relieving measures similar to those applied to notched (filletted) parts.

However, the fatigue strength of press fitted parts is considerably dependent upon the pressure initially present in the press fit[37], as illustrated in Fig. 8.

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Non-Technical Notes

WATER-DETECTING COMPOSITION

WATER-DETECTING COMPOSITIONS ARE EMployed to determine the depth of water layer in storage tanks or containers of petrol or vegetable or mineral oils. Hitherto such compositions were imported, but during the war, at the instance of the Defence Department, a satisfactory composition was evolved by the *Council of Scientific & Industrial Research*.

It has now been decided to make the formula available for civilian use. Considering that millions of oil containers of various types are used in the country every year, the use of this composition should be of great help in rapidly detecting the amount of water added to vegetable and mineral oils.

The composition is used by smearing it thinly on the lower portion of an immersion rod which is then dipped to touch the bottom of the oil container. The depth of the water layer in the container is indicated by the height of the smeared portion up to which colour change has occurred.

The composition is easily prepared by mixing suitable proportions of chemicals with necessary fillers, plasticizers, gum or resins, etc. Since the composition changes colour from light blue to pink red on contact with water or exposure to moisture, a close control of humidity during manufacture is necessary to obtain a satisfactory composition. The composition is packed in metallic collapsible tubes to ensure protection from deterioration by absorption of moisture from the atmosphere.

The principal equipment and laboratory apparatus required with their approximate costs are given below:

	Rs.
Edge runner mill with motor, etc., (pan dia. 26″) 1 unit	5,000/-
Tube filling machine with kettle, 5 litre, 2 units	1,000/-
Laboratory equipment and apparatus	5,000/-
Dehumidifier and auxiliary equipment	4,000/-

All items of the plant and chemicals and other raw materials are easily available in India from imports or from indigenous sources.

Floor Space —

Mixing rooms, laboratory, etc.	
Two rooms : 16′×16′ each	512 sq. ft.
Office : one room 16′×16′	256 ,,
	768 ,,

Cost of Manufacture —

		Rs.
Hydrotropic substance	(1 lb.)	25/-
Filler	(10 lb.)	5/-
Gum	(1 lb.)	1/8/-
Plasticizer	(1½ lb.)	10/8/-
	13½ lb.	42/-
Collapsible tubes, tin clad, capacity 2 fluid oz. each, @ Rs. 175/14/- per thousand, 108 in number		19/4/-
Manufacturing cost at 40 per cent of the cost of materials		24/6/-
Cost per dozen tubes containing 2 fluid oz. each of the material		9/8/3
Or net cost of 1 lb. of material inclusive of packing		6/6/-

Demand for this material is likely to be confined to specialized trade, but the cost of manufacture is low enough to justify its manufacture being taken up.

REVIEWS

Principles of Radar, by Denis Taylor & C. H. Westcott (University Press, Cambridge), 1948, pp. 141. Price 12*s.* 6*d.*

THIS IS THE FIRST VOLUME IN THE SERIES of "Modern Radio Technique" prepared under the general editorship of Dr. J. A. Ratcliffe. The present volume will be most welcome to that large number of physicists, mathematicians and engineers who are interested in radar but whose equipment extends only to the fundamental principles of radio physics and technique. The volume would also be of interest to advanced students in providing them a general survey of the subject of radar as a whole.

A brief historical introduction is provided in the opening chapter. Following Appleton and Watson-Watt, a radar is defined as "a system for measuring at least two of the co-ordinates which define the position of an object with respect to the observer: one of these co-ordinates is the distance between them and is inferred directly from the time of travel of a radio wave travelling by the shortest path between them". (Readers interested more in the history of radar development will find a fascinating account in Rowe's *One Story of Radar.*) The importance of radar in peace and war has been widely recognized. Its contribution during the battle of Britain was most outstanding.

The second chapter deals with the generation and reception of pulse-modulated signals. It describes in a schematic way the magnetron, Fourier decomposition of a pulse-modulated signal, and the noise factor in a receiver. The performance of a radar equipment is described in chapter 3. The determination of range and azimuth are dealt with, respectively, in the next two chapters, and the determination of an elevation is described in chapter 6. Chapter 7 is concerned with two-dimensional scanning and air-interception radars. The separation of sea and ground clutter is discussed in chapter 8. There is also provided an interesting but all too brief discussion of the principles to be used for supressing response from stationary targets. Electrical characteristics of some typical radar equipment are dealt with in chapter 9 and the last chapter is concerned with I.F.F. and other secondary radar systems.

There are two appendices, one on symbols and standard formulae, and the other on the calculation of absorbing, scattering and echoing areas.

Recently there have appeared a large number of books on radar, but for any one who has little previous knowledge of the subject and who desires an elementary but serious introduction to it, the present book can be unreservedly recommended as one of the best available.

D. S. K.

Engineering with Rubber, by Walter E. Burton (McGraw-Hill Book Company Inc., New York), 1949, pp. xi+486. Price $ 6.50.

"WHEN A MAN SETS UP TO BUILD A NEW product", the author goes on to say in the preface, "he may decide that some rubber parts are needed." For the sake of one not familiar with the science of rubber and potentialities of this material, the author in collaboration with the *B. F. Goodrich Company,* Akron, Ohio, has gathered material for the present book and it deals with "industrial rubber products and their applications". The book is divided into twenty-four chapters, of which the first two deal with various types of rubber, both natural and synthetic, and their fundamental properties. From rubber, the technologist fabricates myriads of products: adhesives, V-belts, transmission and conveyer belts, hard rubber goods, mountings, sponges, latex products, moulded rubber articles, etc. Rubber can be used alone or in association with fibre, fabric or metal, and the author, in the course of next twenty chapters, goes into considerable detail with regard to the product design and performance of these. One complete chapter is devoted to the use of vinyl chloride which has a place of its own in "engineering with elastomers". Strictly speaking, it should not come under the heading rubber, for it is classified under non-vulcanizable elastomers. The book ends with a chapter on case histories where the use of rubber helped in the solution of a particular design or production problem and the author has conclusively shown that in skilled hands, rubber, like metals, is a trustworthy engineering material.

Rubber consumption statistics show that over 75 per cent of the world's production goes into the manufacture of tyres and tubes. Modern tyre is the result of painstaking research and development in the fields of textiles, chemicals and rubber, combined with a study of numerous other physical and engineering problems like rubber-metal-textile bonding, heat development on flexing, abrasion, fatigue, etc. It is surprising that no reference to this important subject is made in this book. The subject of " tyre " is big enough to be able to cover a whole volume and it is hoped that this omission will be rectified in later volumes.

There are a few inaccuracies to which attention may be drawn. It is stated (p. 9) that methyl rubber was first made around 1912 in Germany and U.S.A. While it is recognized that Germans, when cut off from all the sources of raw rubber during the first world war, developed the synthetic rubber called the methyl rubber, no such elastomer was made at that time by U.S.A. Again, it has been mentioned (p. 47) that the cements made of synthetic rubber are satisfactory for several purposes : now G.R.S. is particularly known for its " lack of tackiness " and a familiar practice is to give a coat of cement made from guayule rubber when it is desired to cement tread and carcass in the case of tyre made from synthetic rubber. The derivation of formula for centre distances in the case of pulleys worked with V-belts (p. 94) appears to be wrong. The correct derivation should be :

$$C = \frac{L - 1{\cdot}57(D+d) - \dfrac{(D-d)^2}{4\,C}}{2}$$

The formula for the horse-power capacity of transmission belting should, instead of as given in the book (p. 126), be preferably expressed as :

$$H = \frac{SWP(T_1 - T_2)}{33000},$$

where T_1 and T_2 are the tensions on the tight side and the slack side of the belt respectively.

Throughout the book, the synthetic rubbers have been referred to as American rubbers or American made rubber. For want of a satisfactory definition, the materials developed synthetically to replace natural rubber have been variously referred to as Butalastic elastomers, Butaprenes or more simply as synthetic rubbers, and to call these materials " American rubbers " would be tantamount to ignoring the pioneering and brilliant work of British, Russian and German scientists which lies behind the successful manufacture of synthetic rubbers of various types today.

These few drawbacks, however, do not take away the merits of the book. A strong case has been made out for the intelligent use of rubber and the present reviewer agrees with the author when he says that the customer who proposes to employ rubber should tell the manufacturer what the product is expected to do and not to specify the composition (p. 41). An almost infinite combination of properties is possible by an analysis and summing up of the service requirements and the technologist can evolve a satisfactory product. The technologist in turn can look up to the pure scientist for giving him elastomers capable of better performance and possessing properties hitherto unthought of.

M. R. VERMA

The Application of Ionospheric Data to Radio-Communication, by Sir Edward Appleton & W. J. G. Beynon (published by the Department of Scientific & Industrial Research, England), Special Report No. 18, 1948, pp. 44. Price 1*s*.

AS THE NAME IMPLIES, THE BROCHURE DESCRIBES methods by which ionospheric laboratory data may be utilized for deriving characteristics of long-distance radio wave propagation. It is assumed that the reader has the ground knowledge of the ionosphere propagation phenomena. The results are described in a manner in which they can be used by the radio engineer.

A parabolic distribution of ionization with height is assumed and it is shown how, from a knowledge of the height of maximum ionization and thickness of the parabolic layer, one can estimate the maximum usable frequency for long-distance radio communication.

The book contains mainly the works of the authors on the subject that appeared in *Proceedings Physical Society* (1944, 1947) and in some confidential papers having restricted circulation during the war. The principles detailed in this book form the basis of forecast of ionospheric propagation conditions which are computed monthly by the Radio Research Station, Slough.

S. S. Baral

Microwave Transmission Circuits, edited by G. L. Ragan (McGraw-Hill Book Co. Inc., New York), 1948, pp. xvii+725. Price $ 8.50.

The volume, which is No. 9 of the " Radiation Laboratory Series ", deals with the theory and practice of coaxial cables and wave-guides used in the propagation of microwaves. Seven authors who worked in the M.I.T. Radiation Laboratory have contributed to it under the heads : Elementary Line Theory ; Materials and Constructional Techniques ; Rigid Transmission Lines ; Flexible Coupling Units and Lines ; Transmission Units (between coaxials and wave-guides) ; Motional Joints, Tuners, Power Dividers and Switches, Theory of Microwave Filters, and Design of Microwave Filters.

On account of the outward appearance of these circuits they are popularly called " plumbing ". Their design is of interest not only to the microwave engineer but also to the physicist and the optical and audio engineers. This is so because at these frequencies, the cross-section of the circuits is comparable to the wavelength and diffraction and other wave phenomena become increasingly important. The components have to be machined to a much higher precision than is necessary at ordinary radio frequencies and to facilitate this the dimensions of the various components are given in inches correct to a thousandth of an inch.

Although the earlier volume, No 8, of the series, entitled *Principles of Microwave Circuits*, covers the theoretical aspects of the object, a certain amount of repetition has become inevitable in the present volume for the sake of completeness. The chapter on theory of microwave filters covers aspects applicable to low frequency waves also and it is followed by a chapter dealing comprehensively with the design of microwave filters. " The theory and techniques described in these two chapters ", it has been stated, " came too late to play a significant rôle in the war, but they should prove extremely useful in peace-time applications." The term " microwave " is as vague as ever. For example, Vol. 11 on " Microwave Measurements " defines microwaves as extending from 1 mc./sec. In Vol. 8 the word microwave does not necessarily imply a particular range of frequencies, but " a characteristic technique and aperture field ". In the present volume, the term "microwave" may be assumed to include all frequencies greater than 1,000 mc./sec., but it has been specifically stated that the circuits described operate in the range from 2,500 to 25,000 mc./sec. In conformity with other volumes of the series, *mks* units are used throughout the book, except where specifically indicated ; the text matter is well illustrated. The volume is beautifully got up, and is a monumental contribution to the rapidly growing literature on microwaves.

B. N. Singh

Research in Industry, published by the Department of Scientific Research & Board of Trade, U.K. (H.M.S. Office, London), 1948, pp. 84. Price 1*s*. 6*d*.

This booklet contains a series of articles written by accepted and well-known authorities in various fields of technology. These articles were originally published in the *Board of Trade Journal*, U.K., during 1947-48 and they have now been compiled into a single publication.

The first contribution is by Sir Edward Appleton on " How Science can help Industry ". The other 18 articles relate to the research work carried out in various research institutions in Britain in the following subjects : cotton, glass, wool, rayon, pottery, iron and steel, electronics, lace, linen, boots and shoes, paint, furniture, the electrical industry and consumer goods, plastics, machine tools and small tools, light engineering, industrial design and radar. The articles give a brief but complete account of the outstanding researches carried out at these institutions.

The subject-matter under each head gives a brief account of the progress of research in the several branches of technology and the present trends and needs. One important feature noticeable in all these articles is the valuable contribution made by industrial research associations in the country which play so important a part in the progress of the technological research.

The pamphlet contains, in addition, 2 appendices, one detailing the establishments under the *Department of Scientific Research*, U.K., and the other is a list of industrial research institutions in Britain.

Stablization of Plant Names*

V. S. RAO

Council of Scientific & Industrial Research, Delhi

ANY complaint against frequent changes in plant names is usually brushed aside by some as due to ignorance of the International Rules of Botanical Nomenclature, or a misunderstanding or a misinterpretation of those Rules, or due to a lack of experience in herbarium and taxonomic work.

The review of Kanjilal's *Flora of Assam* published in *Kew Bulletin* (1935, p. 586) illustrates the present position. The reviewer points out : " It is to be regretted that the nomenclature is not up-to-date . . . *Camellia theifera* Griff. (should be) *Thea sinensis* L. *Bombax malabaricum* DC. is more correctly *Gossampinus malabarica* (DC.) Alston . . . *Taraktogenos* has been sunk in the genus *Hydnocarpus* though there are quite adequate reasons for keeping the two distinct." It is amusing to find that in the International Rules of Botanical Nomenclature edited by Camp and others (1947), *Camellia* is given with reasons as the correct name and not *Thea* ; and *C. sinensis* appears as the correct name in Sampson's *Cultivated Crop Plants of the British Empire* (published by Kew itself in 1936, just a year after publishing *Camellia* as a wrong name), and in Rehder's *Manual of Cultivated Trees and Shrubs* (1940). Most authorities now agree that the correct name for *Bombax malabaricum* is not *Gossampinus malabarica* but *Salmalia malabarica*. And *Taraktogenos* is sunk in synonymy in Burkill's *Dictionary of Economic Products of the Malay Peninsula* (1935), Hill's *Economic Botany* (1937) and many other publications. This shows what " up-to-date " means !

As contrasted with reviews which express regret at not finding the latest names (correct according to the opinion of a reviewer, but may not be so in the view of others) in publications, there is a very recent review of the 3rd edition (1948) of Dallimore and Jackson's *Handbook of Coniferae*, by W. L. T. in *Science Progress*.* It reads : " In this edition, the old familiar specific names have been changed to comply with International Rules : thus, *Larix europea* becomes *L. decidua* and *Pseudotsuga Douglasii* is changed to *P. taxifolia*. Some will feel regret at this surrender to orthodoxy, especially because it is undesirable that the nomenclature of species of economic value should be at the mercy of arbitrary regulations, and because under the Rules the decisions of taxonomists must always remain liable to possible further revisions."

Experiences of the following type, quoted from the first issue of *Flora Malesiana* (Ser. I, Vol. 4, Pt. I, 1948, XVIII), are only too common when similar attempts are made to obtain authoritative opinion about the valid name for a plant. " A *Clausena* of unknown origin was cultivated for economic purposes at Buitenzorg. I referred it to *Clausena anisum-olens* (Blco) Merr. but the phytochemist was dissatisfied, the properties of the oil did not tally with data recorded from the same species in the Philippines. I then sent ample material with full notes to Dr. Tanaka, Dr. Swingle, and to the Kew and Paris Herbaria, for identification. The answers were all different and the phytochemist was, of course, disgusted with the practical results of taxonomy, because now he had the choice among 5 names for his plant."

The proceedings of the last International Botanical Congress, the papers of Furtado and others published some years back in *Garden's Bulletin, Straits Settlements*, and the recent symposium on plant nomenclature (which was arranged in view of the forthcoming International Botanical Congress, and published in *Amer. Journ. Bot.*, 1949) illustrate the defects in our Rules and nomenclature. The International Rules of Botanical Nomenclature are undoubtedly made to bring about stability of plant names. But if confusion prevails, apparently on an increasing scale, in spite of the Rules, and names published as correct by individual botanists are later shown to be wrong, it is time to review our " Rules " as well as our approach to the problem. The proof of the efficacy of the Rules is

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* 1949, **37**, 378.

* See also *J. Sci. Ind. Res.*, 1949, **8**, 163.

the uniformity in the publications of experts who have worked at big herbarium centres, and followed the Rules of Nomenclature in determining the correct name of a plant.

Burkill, for his *Dictionary of Economic Products of the Malay Peninsula* (1935), and Sampson, for his *Cultivated Crop Plants of the British Empire* (1936), utilized to the fullest extent the resources of the Royal Botanic Gardens, Kew, and both are avowed followers of the International Rules. But it is surprising to find that for a very large number of plants, different names are given as valid by them. A comparison of Hill's standard work on *Economic Botany* (1937) and Holland's *Overseas Plant Products* (1937) shows a similar deplorable difference. In many cases, the valid name of one is the invalid name of the other ! Both of them cannot be correct. Both these authors are also followers of the International Rules and, like Burkill and Sampson, very careful workers. Then why this difference ? Who is correct and for which name ? Or compare again the names given by Reeder in his monograph on *Grasses of New Guinea* (*Journ. Arnold Arboretum*, 1948) with those given by Bor, an acknowledged authority on Indian grasses, in Vol. V of the *Flora of Assam* (1940). Reeder has revived a number of old synonyms to validity, and reduced the valid names in Bor's work to synonymy. Or, compare some of the names given in *Official Guide to the Museums of Economic Botany*, No. 1, Kew (1930), with those in *Hand-list of Tender Dicotyledons*, Royal Botanic Gardens, Kew (1931) ; or the names in Sampson's book with those given in Hill's. These are just a few examples to indicate that, at present, stability or uniformity in plant nomenclature is a myth. It shows that something is fundamentally wrong with the approach to the nomenclatural problem. It is the realization of this utter confusion, attended with too many and too frequent changes, that prompted the International Botanical Congress to appoint a committee to draw up a list of economic plant names which is to remain in force for a period of 10 years.

I wish to make it clear that examples can obviously be given from the writings of systematic and economic botanists. It is in the nature of things and the present state of the subject that discrepancies in nomenclature occur, in spite of the best efforts of writers to find out correct names. The examples given do not reflect anything on the writers but merely indicate that there is an urgent need to stabilize plant names by some means or other. Let wrong names be changed, by all means ; but let the change be to the correct one, and not to another wrong name. Rehder's article on Names and Nomenclature in Bailey's *Standard Cyclopaedia of Horticulture* contains the following emphatic statement :

" Whenever all botanists of authority agree upon a set of changes, the horticulturists should of course adopt them ; but a cyclopaedia of this kind is not under obligation to be ' up-to-date ' in respect to recent changes that may represent only individual opinion, or which are likely to be overturned or at least not adopted by other botanists of standing. So far as possible a cyclopaedia of horticulture should represent settled practices. This work, therefore, adopts a conservative attitude in nomenclature."

Silk Production & Weaving in India*

THE monograph, *Silk Production & Weaving in India*, just published by the *Council of Scientific & Industrial Research*, is a comprehensive treatise dealing in detail all aspects of Indian silk industry as it exists today. The subject-matter is distributed over 11 parts, each part giving a brief but a complete account of the subject under its head. The monograph contains 2 important appendices, one dealing with how Japan developed her silk industry and the second refers to lines of research in sericulture, both of which are of special interest to research workers and may be read with much profit.

Part I of the monograph is in the nature of a general survey of silk industry and its requirements. Of interest to the reader and to one interested in silk industry are the short accounts under the following heads: the present state of silk industry in India, essentials of a successful silk industry and the future of silk products. The next 5 parts deal with mulberry, silk-worm, rearing of silk-worm, reeling and raw silk. Part VII deals with by-products of silk industry. All aspects of *eri*, *muga* and *tasar* silks and the lines on which research is necessary under each head are given in the next 3 parts. The last part under the title " Silk Weaving and Other Manufactures " deals with variety of subject such as raw materials in weaving, looms, types of manufacture and manufacturing processes, standardization of silk fabrics in Japan; production of standardized silk fabrics in India, etc. No aspect of silk and silk industry is omitted from this exhaustive monograph. Its value is apparent from the fact that the author has collected valuable factual data province by province and state by state to present a very clear picture of the future possibilities of the industry. The indigenous methods of silk-worm cultivation and silk production have been critically compared to the conditions obtaining in countries like Japan where silk industry has almost reached perfection. Fully illustrated with 90 useful and informative drawings, and reference tables containing exhaustive statistics on every aspect of silk industry, the publication is sure to be a valuable addition to the libraries of all technological institutions.

Of the research aspects dealt with in the monograph, the following invite attention:

Eri Silk — Castor is the staple feeding material for the *eri* silk-worm. There are many indigenous varieties of castor and selection of suitable varieties with respect to their growth, yield and food value of the leaves, effect of soil and climatic conditions and yield of seed has to be made. *Heteropanax fragrans* (*kesere*) has to be tried for its suitability as a food plant. Diseases of the worm in relation to climate and food and a study of the worm with a view to have the best cocoons are the other problems to be investigated.

Muga Silk — Investigations are to be initiated on the following: to find out if there is more than one species of *muga* worm; relative values of known food plants of the worm; experimental cultivation of *sum* and *hualu* on a plantation basis, occurrence and investigation into causes of *flacherie* and *grasserie* diseases in the worm; the worm produces creamy-white coloured silk when grown on *meazankuri* and *champa* unlike the common golden-coloured silk when fed on *sum* and *hualu*. The former fetches high prices and investigation into the fact that age of trees as well as the quality of leaves influence the type of silk produced can be taken up with profit; there is considerable scope for improvement in the reeling process of *muga* cocoons; a process for giving twist to the yarn as it is reeled has to be developed; methods for easy rearing and increasing the yield of silk have to be developed.

Tasar Silk — A systematic study of the various types of *tasar* silk-worms has to be undertaken to enable easy identification of the varieties. Methods to bring about mating of the worms under domesticated conditions have to be developed. Difficulties encountered in rearing of the worms could be minimized if steps are taken to characterize, isolate and supply seed cocoons of definite voltine characters. Cultivation of *muga* worm on a plantation basis using *asan*, *arjun* and *sal* plants has to be tried on a field scale. Reeling of *tasar* cocoons needs investigation in order to evolve suitable machinery and methods on the same lines as in the case of *muga* silk.

Research in Sericulture — There has been hardly any research on sericulture in India though its necessity has been felt and stray attempts have been made. It has not been possible to date to acclimatize foreign mulberry varieties to conditions obtaining in silk-producing areas in the country. Cocoons produced in Bengal are of a poor grade and intensive research is necessary to improve their quality. Kashmir imports univoltine French eggs; attempts to produce them locally have failed. Development of hybrid races have helped Mysore silk sericulturists to obtain better cocoons. Work on this line has to be extended to other centres.

The main lines of research on mulberry, silk-worms, diseases of silk-worms are:

Mulberry — Selection of varieties after a systematic study of indigenous and foreign varieties; study of the various varieties with special reference to chromosome number, biochemical aspects, draught resistance and genitical studies with reference to inheritance characters; study of soils in relation to the growth and health of mulberry; and physiology of mulberry plants.

Silk-worms — Acclimatization of suitable univoltine worms in India; hybridization to secure improved cocoons; genetical studies, physiology and relation of the life of the worm to climatic conditions; rearing of cocoons having high silk content and high reelability index and minimum boil-off loss; the uniformity of the cocoons is a very important factor in obtaining the above results; analysis of silk fibre from different races; physical studies of the silk fibre and development of economical methods of rearing.

Diseases of Silk-worms—The physiological diseases of silk worms, viz. *flacherie*, *grasserie*, *gattine* and *court* are not properly understood and require investigation. A useful lead can be had from the fact that these diseases are primarily due to disturbances in the metabolic or normal physiological functions of the worm by climatic factors and through deficiency or excess of certain elements in its food.

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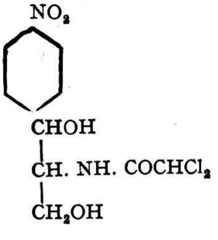
* *Silk Production & Weaving in India*, by C. C. Ghosh, C.S.I.R., Monograph, 1949. Price Rs. 8.

NOTES & NEWS

Chloromycetin Synthesis

THE STRUCTURE AND SYNTHESIS of chloromycetin have been recently described in a series of papers originating from the laboratories of *Parke, Davis & Co.* (*Nature,* 1949, **163,** 553). The empirical formula, $C_{11}H_{12}Cl_2N_2O_5$, has been assigned to it and it is shown to form a di-*O*-acetyl compound. Acid or alkaline hydrolysis produces a halogen-free base and a halogen-containing acid. The base contains both nitrogen atoms, one of which is present as a primary amine. Chloromycetin is described as (*l*)-*Ψ*-1-*para* nitrophenyl-2-dichloro acetamido propane-1 : 3-diol and has the following structure :



[(*l*)—*Ψ*—form]

The molecule contains 2 asymmetrical carbon atoms and only 1 of the isomers is identical with the natural compound and the other 3 are biologically inactive. The synthetic chloromycetin appears to possess the same rickettsiostatic and virustatic properties in experimental infections and the same usefulness in treating patients with scrub typhus.

Synthesis of *p*-Aminosalicylic Acid

A NEW SYNTHESIS FOR *p*-AMINO-salicylic acid, a drug which has potential application in the treatment of tuberculosis, has been developed at the Research Laboratories of *Hoffmann-La Roche, Inc.,* New Jersey (*Phar. Assoc., Scientific Ed.,* 1949, **9,** 38). 4-nitro-2-chlorobenzoic acid is the key intermediate. 4-nitro-2-aminotoluene is converted to 4-nitro-2-chlorotoluene by the Sandmeyer reaction. This latter compound is oxidized to 4-nitro-2-chlorobenzoic acid by an improved procedure using 80 per cent sulphuric acid and potassium dichromate. The action of carbonate-free calcium hydroxide on this chloracid yielded 4-nitrosalicylic acid and gave *p*-aminosalicylic acid with an over-all yield of about 40 per cent when reduced with stannous chloride in concentrated hydrochloric acid.

High-pressure Acetylene Chemistry

THE FIRST PILOT PLANT IN THE U.S.A. for the manufacture of acetylene derivatives, employing high temperatures and pressures, which not many years ago were considered to be impracticable and hazardous, has been put into operation by the *General Aniline Co.* (*Chem. Age,* 1949, **60,** 625). It represents the first development employment in the U.S.A., with considerable amplification, of the principles originally developed by the *I. G. Farbenindustrie* chemist, Dr. J. Walter Rappe. With this development, a new field of organic synthesis has opened up which should prove full of possibilities in the manufacture of adhesives, pharmaceuticals, paper, rubber, textiles, etc.

By making use of Rappe's discovery that acetylene could be handled under pressure and at elevated temperature if the gas streams were divided into smaller flowing streams, it was possible to increase the use of acetylene as a chemical raw material. New products from this source that now appear to be readily available and of considerable potential industrial use are vinyl ethers, butynediol, propargyl alcohol, and many others. Preliminary studies showed that from these intermediates interesting resins, adhesives and rubber-like polymers could be made.

The technique permitting the safe use of acetylene under high pressures consists of 2 methods, one involving the dilution of acetylene with an inert gas and the other wherein acetylene was reacted in small-bore equipment providing a minimum of free space for gases to collect. In its new plant in the *General Aniline Co.,* extensive instrumentation with remote controls has reduced the explosion hazard to a minimum.

Vinylation and ethylenation are 2 processes employed in the synthesis work. The vinylation products include methyl, ethyl, butyl and iso-butyl vinyl ethers. The new technique of diluting acetylene with an inert gas has been employed in order to carry out reactions with low-boiling alcohols.

Condensation of formaldehyde and acetylene was carried out using acetylene under high pressure and temperature and employing a most unusual catalyst, copper acetylide ; propargyl alcohol and butanediol were some of the products of this ethylenation. Special equipment and techniques are employed in this process as the dilution method was not suitable.

Chrome-yellow Manufacture

CONTINUOUS PROCESSES FOR THE manufacture of chrome yellow and molybdate orange and a semi-continuous process for manufacture of zinc yellow have been recently developed at *E.I. du Pont* (*C.T.J.,* 1949, **124,** 425). The production time for chrome yellow is only about 2 hr. from receipt of raw material to bagging of finished product.

Lead nitrate and sodium chromate solutions (35 per cent) are allowed to react in a specially designed precipitation reaction chamber, which is very small compared to continuous precipitation tanks and in which the precipitated mass remains for a few seconds only. The amount of solutions let in are controlled automatically. The lead chromate slurry is fed off to a series of vessels in which the temperatures are controlled to give the desired particle size temperatures and *p*H of the slurry is automatically controlled. The slurry is centrifuged in stainless steel Bird centrifuges (washed during centrifuging) and dried in a Proctor and Schwartz dryer assembly. The yield is 99 per cent of the theory.

A similar process is used for the manufacture of molybdate orange, but in the zinc-yellow process the actual precipitation and particle size development stages are carried out batchwise. The zinc yellow is a basic potassium zinc chromate, and the reaction is carried out in large agitated tanks. The same finishing plant is used for all the pigments and has, therefore, been designed for easy cleaning.

The new process turns out yellow pigments having higher tinting strength, better light fastness and working properties as well as better stability and uniformity of particle size than was obtainable with the old methods of production.

A New Method for Determining the Weight of Cellular Structures

SUBSTITUTION OF A CONTINUOUS spectrum for monochromatic X-rays in the methods now being used for cytochemical elementary analysis makes it possible to weight single cell structures when a sufficiently broad band of a continuous X-ray spectrum is used. The effects of the absorption jumps of the elements in the tissue can be neglected, and the total absorption is proportional to the total mass of tissue. The wavelengths of the chosen band must be selected properly.

A thin collodion foil (*c.* 0·3*U*), laid over the slit in the metal preparation holder serves as supporting membrane for the preparation. The microtome section or a smear of the tissue being examined is laid on one half of that membrane. The other half of the membrane is reserved for an absorption wedge made of thin collodion foils. The X-ray absorption of the foils should be of the same order as that of the sample. The preparation is laid against the photographic emulsion of a Lippmann film or any other very fine-grained photographic emulsion. Very good contact must be secured. A micro-radiogram is registered with long wavelength continuous X-rays (500-4,000). The voltage chosen depends on the nature of the sample. The X-ray image of the sample and the wedge is enlarged 200-500 times by photomicrography. Absorption in single cell structures can be compared with the absorption in the collodion foils, and the relative distribution of the amount of dry substance in the sample can be computed by photometric measurements on the enlarged image.

Knowing the weight per unit surface of the collodion foil and introducing the correction factor, the absolute weight per unit surface of the cell structures can be calculated. It is possible to weigh structures as small as 1μ in diameter, which is the resolving power of the Lippmann film.

This method is now being used in combination with X-ray methods for quantitative estimation of single cell elements (*Nature*, 1949, **163**, 563).

Thickness Measurement by Beta Emission

THE PRINCIPLE OF THE CONTINUOUS measurement of the thickness of sheet materials is demonstrated by a new instrument called the beta-ray thickness gauge recently announced by the *General Electric Co.* (*Chem. Age*, 1949, **60**, 557). By measuring absorption, the device indicates the mass per unit area of the material under test, but can be calibrated in terms of thickness to help operators maintain product uniformity, reduce the amount of rejected material, and save on the amount of raw material used. The new gauge is expected to find application in keeping check of the thickness of metal foils such as aluminium, copper, tin brass and steel, being rolled at high speeds. It can also be employed with plastics, textiles, rubber and other sheet materials, especially those that cannot be contacted because of their condition while in processing.

In operation, the beta-ray thickness gauge measures the deviation from a chosen setting by registering the amount of β-rays which the material under test absorbs. The source of β-rays in the gauge is 2·5 millicuries of strontium 90. Those rays, unabsorbed by the material passing through the gauging head, are gathered in an ionization chamber. An attenuated 90-cycle signal is added in phase opposition to cancel the signal from the ionization chamber. The attenuator voltage, therefore, is a measure of the ionization chamber voltage, and of the amount of material in the β-ray beam.

Operating on a power supply of 100-125 volts, 60 cycles, ±0·3 cycles, the power consumption of the gauge is about 150 watts. Its accuracy is said to be ±2 per cent or better between normal periods of calibration, while drift is not more than 1 per cent per hour after a 30 min. warming-up period. Under normal conditions it is claimed that calibration need not be made more often than once every 4 hr.

Evaluation of DDT

BIO-ASSAY PROCEDURE OF EVALUATING the potency of DDT and its preparations are intricate and the conditions of test experiments are difficult to define. Chemical methods of measuring DDT potency described recently (*Scientific Agriculture*, 1949, **29**, 53) enable accurate definition of one of the variables influencing the bio-assay methods of evaluation — the amount of insecticide to which the insects are exposed.

Three methods are described: (1) gravimetric methods ; (2) volumetric methods ; and (3) colorimetric methods. The gravimetric methods are especially useful for assay of technical grade DDT and DDT dusts, where *p,p'*-DDT crystallizes out after refluxing the sample with a 75 per cent solution of ethanol saturated with *p,p'*-DDT. The volumetric methods depend on the estimation of the labile chlorine in DDT. Of the 5 chlorine atoms in DDT molecule, one is detachable by hydrolysis in the presence of alkali. The labile chlorine atom, having become inorganic chlorine, is then measured by the Volhard titration method or electrometrically. Estimations based on total chlorine content are not specific for DDT.

The colorimetric method, using xanthydrol-pyridine-potassium hydroxide reaction, is rapid and sensitive and is adoptable for qualitative and rough quantitative field work. In a second method, the Schechter-Haller method, DDT is intensively nitrated, the isolated tetra-nitro DDT is dissolved in benzene and to which methanolic sodium methylate is added when a blue colour results showing a maximum absorption at 600μ, the intensity of which is proportional to the amount of *p,p'*-DDT. This method can also be employed for the estimation of *o,p'*-DDT which gives a violet-red colour with 2 absorption peaks at 590 and 510μ respectively. This method is specific and sensitive for *p,p'*-DDT.

Refining of Oils Containing Vitamin A

MOLECULAR DISTILLATION OF VITAMIN A rich oils, while yielding concentrates of a high vitamin A content, requires, in order to obtain satisfactory results, oils free from impurities and with low acid values. Alkali refining is shown to yield good, refined oil and the neutralized oil, far from losing anything of its original potency, has an increased potency (*Nature*, 1949, **163**, 722). In oils with high potency and high acid value, this increase can be quite substantial.

A sample of Groper liver oil with 120,000 i.u. of vitamin A per gm., 12·5 per cent free fatty acid and 3·95 per cent impurities, after neutralization with an excess of sodium carbonate, centrifuging and filtering, gives a refined oil (76·5 per cent yield) with a potency of 140,000 i.u. of vitamin A per gm. The loss due to oil stock trapped in the soap can be minimized employing the latest refining methods. Thus the alkali-refining method provides an economical and simple method of purifying oils containing vitamin A.

Treatment of Leather with Synthetic Resins

METHODS DEVELOPED FOR TREATING leather with synthetic resins are described (*J. Res.*, 1949, **42**, 63). Acrylate monomers have excellent penetration and can be polymerized *in situ* to give increased wear resistance of sole leather and water resistance to upper leather. The acrylates and methacrylates are studied because of their ready availability and relative ease of polymerization.

Three methods are employed: (1) immersion of leather in monomer and subsequent polymerization *in situ*; (2) immersion in polymer solution and followed by evaporation of solvent; and (3) immersion in partially polymerized resin followed by varying degrees of cure depending upon the desired results.

In the first method, leather specimens are immersed in a monomer containing benzoyl or lauroyl peroxide catalyst for 15 min., wiping of excess solution and heating the samples in a closed system overnight at a given temperature. The use of polymer solutions in which the molecular weight of the resin and the viscosities of the solution is low is a necessary condition in the second method. The solutions are prepared by refluxing a solution of the monomer with a lauroyl peroxide catalyst until the desired solids content is obtained. The leather specimens are immersed for half an hour, the excess wiped off and the solvent evaporated for 4 to 6 hr. The third type of resin solution that holds out promise for treating sole and shoe upper leathers is a polysulphide rubber dissolved in toluene. A proprietary preparation, Thiokol Lp-2, has been employed; using acrylate polymers as much as 50 per cent resin can be incorporated in vegetable-tanned crust leather. In the case of specially prepared solution polymers, impregnation up to 25 per cent resin has been obtained in vegetable-tanned crust, and 50 per cent in chrome retanned upper leather. Treatment with Thiokol LP-2 can be controlled to give a stiff sole or to produce a soft shoe upper leather.

Abrasion resistance of vegetable-tanned crust leather is improved about 75 per cent by *n*-butyl methacrylate polymerized *in situ*. Treatment with Thiokol LP-2 improves abrasion resistance by about 30 per cent, whereas solution-copolymers of *n*-butyl methacrylate and ethyl acrylate show no improvement. Half-hour water absorption data on vegetable-tanned crust leather indicates that treatment with *n*-butyl methacrylate, polymerized *in situ*, show 95 per cent reduction over untreated leather. Water vapour permeability of degreased chrome-retan upper leather is reduced by 60 per cent, but the reduced value of about 400 g/m^2/24 hr. is considered ample for foot comfort.

New Plasticizers

SEVERAL NEW PLASTICIZERS HAVE been described (*Chem. Age*, 1949, **60**, 548). Of these di-*iso*-octyl adipate, dibutyl " cellosolve " adipate and dioctyl adipate are the latest additions.

Di-*iso*-octyl adipate is a primary plasticizer for most resins and imparts permanent flexibility, low water extractability, extremely low temperature flexibility, good electrical properties and heat stability, excellent stability to ultra-violet light, and easy processing. It is a very efficient plasticizer and produces plastic films with low plasticizer-to-resin content with good clarity and good handle, drape and resilience. Di-*iso*-octyl adipate is particularly suitable for use with nitrocellulose and produces clear, elastic films. With ethyl cellulose it gives good quality, clear, elastic films.

Films of polystyrene plasticized with di-*iso*-octyl adipate are pliable, non-tacky and practically free from blocking. It also produces good temperature-flexible stocks with synthetic rubbers. Di-*iso*-octyl adipate has a freezing point of −65°C., a flash point of 186°C. and a fire point of 236°C. At 25°C., this plasticizer has no solubility in water, but it is completely soluble in petroleum and mineral oil.

Dibutyl " cellosolve " adipate (dibutoxyethyl adipate) has a freezing point of −30°C., a flash point of 188°C., and a fire point of 277°C. It has found wide use in the production of safety glass because it imparts excellent ultra-violet light stability and extremely low temperature flexibility to the plastic sandwich layer between the two glass plates. There are also other valuable properties of this compound which come into play when used in the combining of paper or textiles. Dioctyl adipate has a freezing point of −70°C. (clear gel) and a pour point −69°C., a flash point of 192°C. and a fire point of 299°C. and mid-boiling-point at 4 min. of 213°C. It is a very efficient plasticizer and produces good elastomers with low plasticizer-to-resin content. By comparison it has higher solvent power and it is more efficient than dioctyl phthalate in a ratio of 29 per cent to 33 per cent.

New in this field of fatty acid esters are tetrahydro-furfuryl oleate, which possesses superior internal lubricating properties, and diethylene glycol dipelargonate for low temperature performance.

The dibasic acid, azelaic acid, is now being used as a raw material for the production of plasticizers. It is also recommended for the manufacture of non-drying type alkyds, where it replaces a portion of phthalic anhydride and results in greater flexibility, improved toughness, good adhesion and good colour. The esters of azelaic acid are likely to be used to an increasing extent where high efficiency, excellent low temperature performance, negligible volatility and good oil resistance are needed for vinyl plastics.

The 2-ethyl hexyl ester of azelaic acid imparts low temperature flexibility to films which are clear, free from haze and possess excellent tear strength. Another interesting azelaic plasticizer is dioctyl azelate which is of value for plasticizing polyvinyl chloride and polyvinyl acetate-chloride cast films and calendered sheet.

Alkyl carbonates of lactates produced by acetylating lactic esters with alkyl chloroformates are newcomers to the range of lactic acid plasticizers. All these esters are compatible with ethyl cellulose and polyvinyl chloride, and judged by modulus and brittle point, these new lactates are more efficient than 2-ethylhexyl phthlate.

The esters of aconitic acid are particularly valuable for the vinyl polymers where chemical stability of highest order is required, Polymeric plasticizers based on aconitic acid possess extremely low volatility and very low extractability by water and oil.

Ranking high in phosphate plasticizers is " Santicizer 141 " produced by *Monsanto*. This is an alkyl aryl phosphate which possesses low toxicity, high compatibility with vinyl resins, low volatility, softness and drape, resistance to weathering, strength, elasticity, abrasion resistance, good low temperature flexibility. By incorporating this phosphate plasticizer in polyvinyl chloride compounds, it is possible to achieve exceptional flame-retarding qualities without sacrificing flexibility or drape. Cresyl diphenyl phosphate is another in this group with improved low temperature flexibility and superior light stability.

The glycol esters are good, all-round plasticizers, and of these triglycol dihexoate, triglycol dioctate and polyethylene glycol di-2-ethylhexoate are of immediate interest. The last-named promises to find many applications because of its unique properties. It shows the same order of non-volatility from compositions of vinyl chloride-acetate compositions as does dioctyl phthalate and, in addition, it is very efficient, since only 30·5 per cent is required to produce standard elongation.

To those working with surface coatings, moulded, extruded, and calendered plastic compositions and synthetic rubber, the polyethylene glycol di-2-ethylhexoate is of considerable value.

Nitrite rubber, a copolymer of butadiene and acrylonitrile, is another non-migratory plasticizer of special value for vinyl films to be used for food packaging. Its high molecular weight enables it to resist extractions by edible fats and greases.

Pliofilm

" PLIOFILM ", A TRANSPARENT rubber hydrochloride film, is being produced on a commercial scale by the *Good Year Tyre & Rubber Co.* (*Chem. Age*, 1949, **60**, 618). The material has many potentialities as a protective packaging material.

Crude natural rubber of good grade is milled and dissolved in benzene to obtain a cement containing 20 per cent solids. The material is cooled with refrigerated water and predetermined amounts of dry hydrochloric acid gas is let in and the rubber is allowed to continue ageing for 4-6 hr. After compounding, the pliofilm cement is filtered twice and transferred to aluminium storage tanks maintained at an elevated temperature to keep the cement from solidifying. After a third filtration, it is made into a film of 0·01″ thickness.

One of the merits claimed for pliofilm is its capacity to retain or repel moisture according to needs; it is considered to be moisture-vapour-water-proof. It has also been found to be resistant to punctures, tears and abrasions, pliable though it is. It is unaffected by atmospheric conditions or normal temperatures and is not harmed by the rigorous processes of forming packaging material into a finished package. It is also resistant to weak acids and alkalies, to grease, oils, moulds and vermin. It is non-explosive, non-inflammable, odourless and tasteless.

Pieces of pliofilm can be joined by heat sealing, the resulting weld being twice as strong as the original material.

One of the unusual characteristics claimed for it is its ability, when drawn under tension to a thin membrane, to hold in moisture and retain gases such as carbon dioxide, to slow down the metabolism of wrapped fruit. Pliofilm has been successfully used to contain and preserve frozen foods, meats, cheese, coffee, tobacco, and liquid and semi-liquid products including oils.

Bright Aluminium Alloys

AN IMPROVED METHOD OF BRIGHTENING aluminium alloy surfaces is claimed in an English patent with special reference to making the brightening more permanent and less affected by atmospheric tarnishing or other adverse action (*Chem. Age*, 1949, **60**, 690).

The surface is first thoroughly degreased (by electrolytic methods), then immersed in strong acid (*p*H below 1) solution of phosphoric and chromic acids and water for 30-60 min. at a temperature of 80°-90°C. This produces a uniform surface, whether it has been previously polished or not, and serves to descale, removing any oxide without attacking the metal itself. The surface is then subjected to anodic oxidation in an alkaline solution containing trisodium phosphate and sodium phosphate or aluminium phosphate and caustic soda. The *p*H should not be lower than 11.

The part to be brightened is made the anode; temperature is 70°-95°C., voltage 8-20 v.; current density 4-10 amp./dm.^{2}; and time required is 5-10 min. By this means a thin coating of aluminium hydroxide is deposited and is followed by rinsing with running water. The film formed, at first in a powdery condition, is dissolved in a solution of phosphoric and chromic acids, similar to that used before. Colouring with organic dyes may be imparted, and surface finally sealed with boiling water or by other means.

New Uses for Cotton

MODERN RESEARCH HAS EXTENDED the uses for cotton in many fields. In plastics, cotton is being employed as the base for plastic products. Cotton seed hulls, hitherto considered a waste product, is finding increasing application in plastic industry.

Inexpensive cotton fabric that could be made without the usual spinning and weaving processes are visualized in the near future. Cotton fibres bound together with a thin layer of plastic application forms an unspun, non-woven fabric suitable for many types of products. Among the unspun articles now being marketed are towels, napkins, draperies, etc. These articles, though inexpensive enough to be discarded in a short while, last longer than the usual cotton fabrics and have a softer and more luxurious appearance.

Coated cotton material resembling rubber and possessing many advantages over leather is being developed and used for upholstery, luggage, and shoes. The material is stain-proof, dust-proof and waterproof. Mixed with asbestos fibre (20 per cent) the resultant fabric has excellent water absorption and polishing properties.

Combined with aluminium and glass yarns, unusual fabrics have been developed. A new protective cotton fabric is made to reflect light which can be seen by night motorists at a distance of half a mile. A variety of cotton gauge developed is capable of being absorbed in the human body within a few weeks (*USIS*).

New Cleansing Agents & Disinfectants

DEVELOPMENT OF A POWERFUL series of " multi-cleanser " soap substitutes, which kill germs instantaneously besides removing dirt, has been reported to the *American Chemical Society* recently. The new detergents can be used for surgical instruments and household utensils alike. These products, known chemically as "morpholinium alkyl sulphates", are so potent that a teaspoonful of detergent is enough to yield a gallon of suds, and at the same time to match the bactericidal strength of a pound of carbolic acid. These do not require caustic substances or metallic salts to do their work and have several advantages over soap ; they do not break down and form insoluble deposits or cause the familiar " bath-tub ring " when used with hard water. Since they are non-metallic, they do not leave the caustic, slippery feeling on the skin which follows the application of soap. These non-metallic and non-caustic products described in the report show remarkable chemical stability and compatibility with other substances encountered in household use and in certain industrial processes (*USIS*).

Softening of Hard Water by Aeration

SEMI-LARGE-SCALE EXPERIMENTS on the softening of hard waters by aeration in the presence of added precipitation chalk conducted by the *Water Pollution Research Board, D.S.I.R.*, London, have shown that it is possible to reduce the alkalinity of deep-well water from about 255 parts of calcium carbonate to about 70 parts per million when diffused air at a rate of about 10 litres of air per litre of water per hour for a period of 5 or 6 hr. is passed. The presence of finely divided chalk in suspension in a concentration of about 1,000 parts of calcium carbonate per million is required if softening is to proceed at a reasonable rate ; use of greater amounts gave no further advantage. To maintain the chalk in suspension agitation other than that provided by the diffused air has been found to be necessary. Repeated use of the same chalk in 10 successive experiments did not cause any reduction in the rate of softening obtained, although a slight increase in the size of the particles of chalk was observed.

Biological Removal of Sulphate from Water

A BIOLOGICAL METHOD FOR THE removal of sulphates present in bore-well water and render it suitable for irrigation purposes has been worked out by the *Water Pollution Research Board, D.S.I.R.*, London. Bore-well water contains total solids of the order of 3,000 p.p.m. of which the major portion is generally calcium sulphate. Sulphate-reducing bacteria are employed for the purpose, the reduction occurring under anaerobic conditions. Oxygen is completely excluded and provision for the continual removal of hydrogen sulphide is also necessary.

A rectangular reaction vessel, with a capacity of about 1 gal. and having internal baffles giving in effect a passage of about 40″ long and 2″ by 3″ in cross-section, packed with degreased steel turnings and maintained at 30°C., and filled with iron turnings was used. Oxygen-free air was supplied.

Of the various media employed, the one selected contained sodium lactate 5 gm., dipotassium hydrogen phosphate 0·5 gm., ammonium sulphate 2 gm., and water 1 litre. The medium was not sterilized but was freed of oxygen by flowing in a rapid current of carbon dioxide. The reaction vessel was filled with settled sewage and stored for several days to provide an inoculum of suitable bacteria. The sewage was then displaced by the medium at a rate of about 120 c.c. per hour, giving a period of retention of about 36 hr. The water to be treated was let in next with the nutrient salts. On the 13th day about 25 per cent of the sulphate present was removed and by the 25th day the effluent was free of sulphate. The pH fell rapidly after the 63rd day and a new batch of medium had to be introduced with the pH value adjusted to 6·5.

These experiments indicated that sulphate in concentrations approximately equivalent to that in a saturated solution of calcium sulphate could be completely removed from water at a sufficiently high rate.

Experiments designed to replace lactate by grass extracts (by steeping grass in water for a week) showed a considerable reduction in the concentration of sulphate after the 87th day of continuous running, the sulphate content being reduced from 4,000 p.p.m. to 1,000 p.p.m.

Sand-lime & Concrete Bricks

THE *Building Research Station, D.S.I.R.*, London, has published a bulletin (*National Building Studies, Bulletin No.* 4, *H.M.S. Office*, London ; price 6*d.*) which gives a brief account of the properties of sand-lime and concrete bricks, and amplifies the information given in earlier reports.

The principal materials used in making sand-lime bricks are siliceous sand, lime and water, the proportion of lime ranging from 5 to 9 per cent. Hydration of lime must be complete before the bricks are pressed and limes must be high calcium limes and reasonably pure.

Ground quicklime and sand with the requisite quantity of water are thoroughly mixed in a screw or pan mixer and compressed under high pressure in a rotary table type press and steam cured for 4-15 hr. using high pressure steam (120-195 lb./sq. in.).

For concrete bricks, Portland cement is usually employed. Most of the concrete bricks are made with a dense natural aggregate ; furnace clinker, blast-furnace slag and clay brick waste are also used. Clay brick waste or rubble should not contain more than 1 per cent of acid-soluble phosphate. The proportion of cement to aggregate in concrete bricks with dense aggregates varies from 1 : 5 to 1 : 12 by volume according to the type of aggregate, moulding process and quality of brick required. Concrete bricks harden and develop their strength without any artificial curing.

The strength of dry sand-lime and concrete bricks is from 30 to 50 per cent higher than when saturated. The compressive strength for sand-lime bricks is of the order of 100-5,000 lb./sq. in. and for concrete bricks with natural aggregates, 1,000-6,000 lb./sq. in.

Under normal conditions of exposure, good sand-lime brick lasts well ; inferior quality has poor weather resistance and should not be used for external work. With ageing they improve in strength. Exposure to salt spray leads to erosion of the brick. Good quality, 9″ sand-lime brick satisfies the 6 hr. fire resistance test in all respects.

It is preferable that both sand-lime and concrete bricks should be used in a reasonably dry condition in order to minimize drying shrinkage effects.

Prevention of Dazzle on Roads

THE *Road Research Laboratory, D.S.I.R.*, London, has investigated into the problem of head-lamp dazzle on roads and the results have been incorporated in a recent technical paper (*Road Research Technical Paper No.* 14, *H.M.S. Office*, London, price 6*d.*).

Of the two measures to deal with the problem, viz. (1) those which might be applied to existing lamps with promise of immediate improvement; and (2) use of polarized light or improved lighting systems; the former has been dealt with in the paper. The paper deals with: nature of the "single beam" and "multiple beam" systems; present state of the law relating to head-lamps; a study of drivers' behaviour and the prevalence of dazzle; action recommended in relation to existing vehicles; proposed rules for the adjustment of lamps; and education of the motorist. The two appendices deal with: instructions for adjusting head-lamps and pass-lamps, and a section entitled "Some additional conclusions to be drawn from the dazzle survey".

The survey reveals that most of the dazzle on roads is due to dipped head-lamps and pass-lamps, the low-mounted pass-lamps being especially liable to this failing. It is recommended that lamps should be set so that driving beams do not rise above the horizontal and passing beams are dipped at least 3 degrees. It is also recommended that head-lamps and pass-lamps should be mounted not higher than 3′ 6″ and preferably not lower than 2′ 6″ but never less than 2′ from the ground.

Hand-operated Threshing Machine

A HAND-OPERATED MACHINE which can thresh nearly 1 cwt. of wheat, oats, barley or rice in 1 hr. has just been produced by a Scottish firm. Of low cost, it is specially designed to help the small farmer who cannot afford a large initial outlay for mechanical aids. The machine is operated by two men while a third feeds the sheaves into a drum. The drum, which runs at 350 r.p.m., is carried on a ball-bearing shaft. It is covered with a sheet metal and is fitted with four cast steel peg-type beaters. The pegs on the beaters run in the spaces between similar pegs on a stripper bar which can be adjusted to give suitable variations in the severity of threshing. A ribbed and knobbled concave divides grain from straw.

Although primarily intended for hand operation, provision is made for fitting on 1 to 1½ h.p. motor which would increase the speed of the drum to 600 r.p.m. (*BIS*).

Imperial Institute, London, Annual Report, 1948

THE TWO MAIN SECTIONS OF THE Annual Report of the *Institute* for the year under review reflect the growing interest of the State, as well as of commerce and science in new Empire sources of minerals, abrasives, oil-seeds, plant fibres, tobacco, hides and skins, essential oils and insecticides.

The Mineral Resources Department reports a 14 per cent increase in inquiries covering a wide field. Prominent among them are questions concerning world resources of coal, bauxite and gypsum. The world coal shortage intensified the need in many regions to find suitable qualities of fuel before selecting new industrial sites. Fresh fields of bauxite are required to meet the world demand in the near future, and the information sought about new deposits of gypsum is clearly connected with developments in building research that call for the use of plaster of Paris as the principal material.

Other inquiries concern non-metallic minerals and comparatively rare and hitherto little known raw materials that are proving of such importance to modern industry. These include caesium, gallium, indium, germanium, selenium, tellurium, lithium, cerium and tantalum. Many Government departments and quasi-government organizations have also sought complete information about diatomite — its nature, occurrence, quality, working, production, transport, treatment, uses, prices, alternatives and statistics. In recent years, this mineral, mainly drawn from dollar sources, has been used in the filtration of many important food, drink, and pharmaceutical products. For some time the *Institute* has tried to find Empire substitutes, with Kenya as the chief source, and even Skye has been actively considered.

In the past few years, the *Institute* has also emphasized to Government geological surveys, private geologists and mining concerns, the possible presence of vermiculite in their respective territories. Today, with more knowledge of the nature and usefulness of the mineral, the stage during which it might be dismissed as worthless mica is passing. Private and official reports of vermiculite found in entirely new areas include Gold Coast, Nigeria, South-west Africa and Mysore, though its economic possibilities there are not yet known. South Africa continues to be the chief Empire supply source, and new finds have been reported from northern Transvaal and Natal.

New outlets and applications for Sierra Leone micaceous haematitie have been considered as a weighting ingredient in oil drilling muds, as heavy medium for coal cleaning, granules for roofing felt, welding red coatings and for use in powder metallurgy. Examination of mineral specimens from Tanganyika indicated that one of them was a member of the Euxenite-polycrease series and was "appreciably radio active"; another was a garnet of good quality that was found suitable for abrasive work.

The other scientific section of the *Institute* is the Plant and Animal Products Department, whose staff, during the year, has prepared memoranda for consideration at numerous meetings of the Colonial Primary Products Committee. Other work of the Department includes plans for a world-wide series of analysis of the insecticides of pyrethrum flowers.

To assist the general plans for increasing world supplies of vegetable oils and fats, tests of sunflower seeds, four new and two older strains, have been made in Nigeria and Sierra Leone. In the former colony the new strains showed an oil content of 33 to 35·8 per cent and the older ones 27·6 to 28·9 per cent, though one of the latter gave a greater quantity per acre owing to a heavier plant yield. In Sierra Leone the oil content of two of the new strains was 23·6 per cent, that of two others from 27 to 30·4 per cent, while the older varieties yielded 22 to 26·5 per cent.

Tests of tobacco leaf grown in Nigeria by native farmers have shown that of 74 samples examined, 14 possessed good burning qualities and, under improved cultivation and curing, should be suitable for the continental and north African markets. Smoking trials of experimental samples from Nyasaland, where the resumed production of flue-cured leaf is under consideration, disclosed that five were promising as cigarette leaf of neutral type suitable for blending and sixth as pipe tobacco.

Research in Lac

IMPROVING OF CROP PRODUCTION on certain host plants, selection of suitable alternative hosts of *Bracon greeni*, new moulding compositions, varnishes, paints and enamels, fundamental work on hard and soft lac resins, hydrogenation of shellac and shellac components, effect of infra-red radiation on lac and lac fibres are some of the main features of the research work carried out at the *Indian Lac Research Institute* during the year 1947-48.

The possibilities of *Albizza lucida, Ficus bengalensis* and *Ougenia dalbergeoides* as *Baisakhi* brood-producing hosts have been investigated. The field trials show that *A. lucida* gave the best results, the rates of brood used to brood yield being 1 : 4·6.

Harvesting of immature lac crop has been shown to give poor yields. Use of 60 mesh wire-gauge baskets as brood containers has been found effective in eliminating the whole range of organisms infecting lac but permits free passage of lac larvae. This practice has made possible the cultivation of disease-free lac in fresh areas and prevents wastage of stick lac.

Gammaxane spray has given good results as an effective fumigant of brood lac. A mixture of ethylene dichloride (75 per cent) and carbon tetrachloride at a rate of 5 oz. per 10 cu. ft. of space has proved to be an excellent, non-inflammable fumigant.

Breeding studies of *B. greeni* on a mass scale on unnatural hosts showed that with the larva of *Trychylepidia fructicassiella*, 47·1 per cent parasitism was obtained ; in the case of *E. amabilis*, 43·8 per cent parasitism was obtained.

Optimum conditions have been established for lac-urea-formaldehyde moulding powders with regard to (a) particle size, moisture content level necessary to produce blister-free samples, and (b) temperature, pressure and time of curing.

Lac-cashewnut shell liquid-sulphur compositions developed at the *Institute* show promise in plastic and varnish fields. The cashewnut shell liquid is first heated with 10-20 per cent sulphur at 200°-250°C. and the resultant mass, on combination with equal proportion of lac, yields a soft rubbery material. This material in combination with fillers like zinc oxide and asbestos gives plastic composition which could be moulded in the hot or cold.

Lac-cashewnut shell liquid varnish compositions in combination with linseed oil have been prepared. These compositions yield films which are smooth and glossy and non-tacky after only 18 hr. of drying, and have good flexibility and adhesion. Waterproof varnishes for coating paper, bullets and dummy cartridges have been formulated.

The use of lime in place of litharge as an incorporating agent in the preparation of lac-linseed oil varnishes has been further investigated and modified compositions have been developed. The products obtained by employing potassium hydroxide and potassium linoleate as incorporating agents are completely soluble in water as well as in the usual varnish solvents. A new lac-linseed oil vehicle for paints and enamels has been prepared using linseed oil (100 parts), lac (20 parts), and litharge (3 parts) ; from these compositions satisfactory quick-drying paints have been obtained.

Physico-chemical studies on shellac were :

Ultra-violet absorption spectra of shellac solutions ; flowing birefringence of shellac solutions ; X-ray structure of shellac ; the dielectric properties of acetic ester of lac; the dielectric properties of lac-glycol ether ; insulating properties of lac-linseed oil-lime varnish, etc.

Infra-red heating has been employed for the determination of moisture in lac. This procedure reduces the time taken for moisture determination from 24 hr. to 4 hr.

A method has been developed to isolate wax from the waste varnish residues.

Indian Standard for Refractories

THE ENGINEERING DIVISION COUNCIL of the *ISI* has brought out a Draft Indian Standard Recommendation for Refractories for Railways. It has been prepared by an expert committee composed of representatives of manufacturers and consumers of refractories in India.

The importance of the subject can be gauged from the fact that the railways consume about Rs. 17 lakhs worth of fire-brick.

The purpose of these recommendations is to indicate the types of refractories most suited for different classes of work in the various departments and workshops of the Indian railways. The recommendations are expected to assist the railways in the standardization of designs and qualities, in issuing tenders and making purchases, and the manufacturers of refractories in arranging their production programmes.

These recommendations give the qualities of refractories for various classes of applications in different departments and workshops of the Indian railways. Some classes of work are common to workshops other than those of the railway. The recommendations may, therefore, be taken to cover similar requirements elsewhere.

Comments on the draft standard will be received till 11 September 1949, by the Director, Indian Standards Institution, Block 11, Old Secretariat, Delhi 2.

Announcements

Dr. M. S. Krishnan, Director, Indian Bureau of Mines, has been appointed the Director of the Geological Survey of India, in place of Dr. W. D. West. Dr. Krishnan is the first Indian to be appointed to the post.

Director, Coconut Research Scheme, Ceylon — Dr. Reginald Child has relinquished the post of Director of the Coconut Research Scheme, Ceylon, which he had held since 1932. He will be succeeded by Mr. F. C. Cooke, who was Chemist (Coconut Products) with the Department of Agriculture, Malay, from 1929 to 1938, and Canning Officer of the same department from 1946 to 1949.

Lady Tata Scholarships for 1949 — The following international and Indian scholarships have been awarded for the year 1949 :

The international awards totalling £ 3,000 for research in diseases of the blood with special reference to leucaemias are made to Dr. Edith Peterson (England), Dr. M. C. Bessis (France), Dr. J. Bichel (Denmark), Dr. Pierre Cazal (France), Dr. J. Clemmesen (Denmark), Dr. E. Kelemen (Hungary), Dr. Edoardo Storti (Italy), Dr. Charles Oberling (France) Dr. Jagdish Chandra Mehta (India), Pascou Atanasiu (France), and Gunther Schallock (Germany).

Indian scholarships of Rs. 250 per month each for one year for scientific investigations having a bearing on the alleviation of human suffering from disease are awarded to Messrs Gangagobinda Bhattacharya (Calcutta), Bimal Kumar Sur (Mysore), K. Ramamurti (Bangalore), Gauranga Roy (Calcutta), D. V. Siva Sankar (Madras), and P. R. Srinivasan (Coonoor).

Reports from States & Provinces

MADHYABHARAT UNION

Industrial Expansion

Power Projects — The Government have sanctioned 2 large schemes for power development. The first is a thermal station to be located at Indore at a capital cost of Rs. 121 lakhs. When completed this station would supply 15,000 kW. The other scheme is a hydel scheme on the Chambal river, 200 miles north of Indore, at a cost of Rs. 9·75 crores. The reservoir will have a catchment area of 8,000 sq. miles and the project is considered to be one of the cheapest in India from the point of view of dam construction. It is expected to produce 40,000 kW. ultimately.

The Union has both long-term and short-term industrial projects; the former ones to be taken up only after the power from Chambal projects becomes available. The contemplated ones include production of rayon and electro-chemicals.

The short-term projects relate to cement, power alcohol, sugar, paper, glass, ceramics and silk weaving.

Cement — At present, the Union produces only 60,000 tons of cement per annum and in view of the large power and irrigation projects, the demand may be nearly 100,000 tons. The Union is rich in excellent lime stone at Neemuch and it is planned to locate a cement factory of 100,000 tons capacity at this place. The capital investment on the industry is expected to be Rs. 1·25 crores.

Power Alcohol — There are 5 sugar factories in this region and are estimated to make available 10,000 tons of molasses for the industry. The Union's petrol requirements are in the neighbourhood of 40 lakh gal., and 5 lakh gal. of power alcohol would be needed for admixture. It is proposed to erect a distillery with a capacity of 5 lakh gal. per year.

The starting of a sugar unit of 1,000 tons crushing capacity, caustic soda and sulphuric acid units with 5 tons capacity each and a glass factory at Guna have been approved by the Government. Sanction has also been accorded for the establishment of a pottery work at Indore. The factory will use the extensive good quality fire clay deposits near Piplia and produce stoneware pipes, acid jars, etc. The China clay deposits near Indore may be utilized for making crockery.

WEST BENGAL

Silver Refinery for Calcutta

An up-to-date silver refinery is proposed to be set up in Calcutta. The plant and accessories are reported to cost Rs. 60 lakhs and the plant is expected to be ready in about 3 years.

Institute of Jute Technology

This Institute, the first of its kind in India, is being started under the auspices of the Calcutta University and will start functioning from 1950. The Institute has been founded by the *Indian Jute Mills' Association* in collaboration with Calcutta University. Capital expenditure amounting to Rs. 20 lakhs and the recurring cost of running the Institution will be borne by the *Association*.

BOMBAY

Research Institute for Silk & Rayon Industry

The *Council of Scientific & Industrial Research* has received a contribution of Rs. 15 lakhs from the *Silk & Art Silk Mills' Association Limited*, Bombay, for organizing a research institute for the industry at Bombay. The functions of the research institute will include research in weaving of silk and art silk, including rayon and dyeing and processing of silk and rayon fabrics.

The offer has been accepted in principle, subject to the approval of the Governing Body of the Council and the Standing Finance Committee of the Indian Legislature. After approval, the Government of India will make a grant to supplement the industry's contribution.

BIHAR

Engineering College for Sindri

A mechanical and electrical engineering college will be set up at Sindri, in the Jharia coalfield area, close to the Sindri fertilizer plant. The Government have sanctioned a sum of Rs. 70 lakhs for this purpose. The construction work is to begin in October this year and the college is expected to start functioning in 1950.

UNITED PROVINCES

Cement Factory

The Government will shortly start a cement factory to produce 700 tons of cement per day at Robertsongunj, 52 miles from Mirzapur. This is the first cement factory to be state-owned and is expected to meet a fifth of the province's requirement in cement. The Government have also surveyed other localities in the province with a view to set up a few more units.

ERRATUM

This Journal:

1949, Vol. **8**, No. 7, article entitled " A Cosmic Ray Radio Sonde ", page 259, L.H. col., para 1, the sentence " The results obtained will be published separately *by* Messrs A. S. Rao and G. S. Gokhale " should be *read* as " The results obtained will be published separately *with* Messrs A. S. Rao and G. S. Gokhale ".

INDIAN PATENTS

The following is a list of Patent Applications notified as accepted in the *Gazette of India*, Part II, Section 1, for June 1949.

Plastics & Plasticizers

39014. I.C.I. LTD. : Process for fastening nylon to nylon : *Fastening of nylon to nylon by an adhesive made from a solution of nylon in a coal-tar derivative.*

39165. I.C.I. LTD. : Improved moulding compositions : *Incorporating boric acid with or without alkali earth metal oxide or hydroxide in a phenol-formaldehyde moulding composition.*

Inorgauic Chemicals

39855. DAS GUPTA : A process for the removal of hydrogen sulphide from coal gas : *Solid product obtained or recovered from flue dust contained in the blast furnace gas.*

Organic Chemicals

40174. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ : Method for producing and refining ethyl alcohol : *The pH of the crude ethanol product is adjusted between 6 and 9 and thereafter it is fractionally distilled and hydrogenated.*

Miscellaneous Chemicals

39339. BAYER PRODUCTS LTD., BROWN & MELLOR : Method of combating diseases of potato tubers and preparation therefor : *Using chlorinated mononitrobenzene as active substance.*

37795. INTERNATIONAL GENERAL ELECTRIC CO. OF NEW YORK LTD. : Fluorescent materials : *Mixture of calcium phosphate with a cerium compound containing 2 to 20 per cent cerium fired at 950°C. to 1100°C.*

Food & Kindred Products

38698. DISTILLATION PRODUCTS INC. : Production of vitamin A and intermediates therefor : *Reacting corresponding unsaturated carboxylic ester with lithium aluminium hydride or lithium boron hydride.*

38785. AMERICAN CYANAMID CO. : Purification of aqueous sugar solutions : *Treating solutions with an acid and alkaline contact mass.*

Fuels & Lubricants

40398. C. D. PATENTS LTD. : Manufacture of carbonaceous moulding compositions suitable for extrusion and of moulded carbon products therefrom : *Mixing subdivided coal with a binding material and heating the mixture at an elevated temperature.*

Metal & Metal Products

40503. JOHN MILES & PARTNERS (LONDON) LTD. : Production of ferrochrome, high chromium steels, stainless steels and the like : *Smelting ore with alloying metal with carbonaceous material in blast furnace injected with an oxygen rich fluid.*

40291. CARNEGIE-ILLINOISE STEEL CORP. : Method and apparatus for the electrolytic coating of metal strip : *Comprises passing through an electrolyte two strips forming the cathode and the anode, guiding the strips in parallel, proximate relation to each other and maintaining shields between the edges of the strips.*

38762. WILLIAM JESSOP & SONS LTD. : Alloys of an austenitic character having a cobalt-nickel-chromium bases : *Nickel chromium steel having a proportion of niobium and vanadium and also containing 30 per cent to 50 per cent.*

41076. THE MOND NICKEL CO. LTD. : Alloys : *Containing 0·75 to 2·5 per cent carbon from 1 to 6 per cent tin, from 0·03 to 0·25 per cent magnesium, from 0·5 to 4 per cent silicon, and from 0·5 to 4 per cent manganese, the balance being nickel.*

Stone, Clays & Glass Products

39625. TURNER ASBESTOS CEMENT CO. LTD. : Improvements relating to the manufacture of reinforced asbestos cement or like sheets : *Coating asbestos cement on rotating cylinder, introducing predetermined lengths of reinforcement and cutting into shorter sheets.*

Textile & Textile Products

40306. BOXEL-MALETRA SOCIETE INDUSTRIELLE DE PRODUITS, CHIMIQUES : Method of producing products intended for improving the quality of textile materials : *Impregnating textile with a compound obtained by condensing a carbonic acid amide with glyoxal and again condensing the product with formaldehyde.*

40428. JOSEPH BANCROFT & SONS CO. : Printing of cellulose textile fabrics : *Printing paste comprising thermosetting resin-forming materials and a vat dyestuff in free leuco base form.*

Miscellaneous

CARDIS : Improved fibre decorticator and method of using same : *Comprising two semicircular arcs terminating in two rectangular pieces, one of the rectangular pieces having extensions at its end.*

40873. N. V. GANPULE & R. S. KULKARNI : Manufacture of primary galvanic dry cells : *Container made of plastic material.*

38612. MISTRY : An animal weight driven water pump : *Iron wheel tilting on central ball-shaped cast iron knob operates either diaphragm or plunger type pumps.*

40458. STANDARD TELEPHONES & CABLES LTD. : Pastes for printing electrical circuit elements : *Containing finely divided material dispersed in a hydrophilic liquid containing weighting agent.*

Journal of

Scientific & Industrial Research

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EDITORIAL BOARD

CONTENTS

Letters to the Editor

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.

Editorial communications and books and periodicals for review should be addressed to the Editor, JOURNAL OF SCIENTIFIC & INDUSTRIAL RESEARCH, *'P' Block, Raisina Road, New Delhi.*

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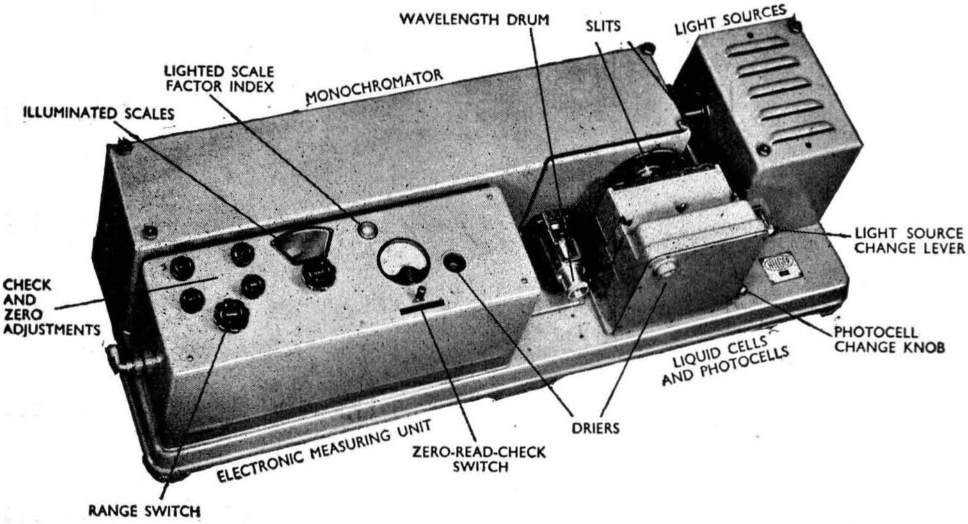

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The Analytical Uses of Dithiocarbamido-hydrazine

J. GUPTA & B. CHAKRABARTTY

National Chemical Laboratories, University Buildings, Delhi & University College of Science, Calcutta

A NUMBER of sulphur-containing organic compounds are now known to give deeply coloured complexes with bismuth. Mention may be made of thiourea[1], dimercaptothiodiazole[2], diethyl dithio-carbamate[3], and dithizone[4], based on which methods have been developed by different workers to estimate bismuth colorimetrically when the coloured complexes are soluble either in water or in solvents like chloroform or carbon tetrachloride, or by peptization in aqueous solutions if the complex is insoluble.

The colour which solutions containing bismuth develop with thiourea, a reagent which commends itself as one of the simplest and easily available among organic sulphur compounds, is largely dependent on the concentration of the reagent and other factors, and was for a long time regarded as unsuitable for colorimetry, until Mahr[1] found the limited range of working conditions in which the reaction could be useful.

The present work relates to some analytical applications of the compound dithiocarbamido-hydrazine, a simple derivative of thiourea on which no work appears to have been done, though some complex derivatives of thiourea have been examined. Some characteristic reactions of the compound with metallic cations are now noted and the possibility of utilizing them for quantitative estimation of metals and of their separation from analogous cations from which separation by the classical methods is known to be difficult, is examined.

A summary of the characteristic reactions observed with some of the commoner metals is given in Table I.

TABLE I

METALLIC ION IN SOLUTION	REACTIONS WITH THE REAGENT		
	In acid solution	In neutral solution	In ammoniacal solution
Ag^+	White heavy bulky ppt., darkens on strong heating. Insol. in acetic acid. Quantitative	White ppt.	Brown, heavy, bulky ppt. Quantitative
Bi^{+++}	Deep yellow colouration. No ppt.	...	...
Cd^{++}	No ppt.	No ppt.	Fine-grained, white ppt., dissolves in acetic acid. Quantitative
Co^{++}	No ppt.	No ppt.	Brown, voluminous, heavy, ppt., dissolves in acetic acid. Quantitative
Cu^{++}	White, granular, heavy ppt.	Black, fine-grained ppt., insol. in acetic acid. Quantitative	Black ppt.
Hg_2^{++}	Black, granular, heavy ppt., insol. in acetic acid. Quantitative	Black ppt.	...
Hg^{++}	White, granular, heavy ppt., insol. in acetic acid. Quantitative	White ppt.	...
Ni^{++}	No ppt.	No ppt.	Chocolate brown, voluminous, heavy ppt., dissolves in acetic acid. Quantitative
Pb^{++}	No ppt.	Yellow, granular, heavy ppt., dissolves in acetic acid. Quantitative	...
Zn^{++}	No ppt.	No ppt.	White, granular ppt., dissolves in acetic acid. Quantitative

The similarity of the reagent with thiourea and the thiocyanates is evident from the observations recorded in Table I. The black compound of copper obtained from acetic acid solution is strongly paramagnetic and is presumably the cupric derivative. The white compound obtained from strongly acidic solutions is, on the other hand, diamagnetic, and is thus a cuprous compound. Both precipitations are quantitative, but the white granular cuprous compound settles and filters easily and is thus more useful from the analytical point of view. Estimation by direct weighing of the precipitates was not possible, because neither of them bears a constant composition. They were decomposed by nitric acid and the copper titrated iodometrically. The cadmium complex, soluble in acids but insoluble in ammonia, also settles and filters easily, showing no signs of oxidation, but had to be decomposed and weighed as $CdSO_4$. A complete separation of copper from cadmium was effected by a single precipitation of the cuprous derivative from strongly acid solutions, and precipitating the cadmium complex from the filtrate by rendering it ammoniacal in presence of a sufficient quantity of the reagent.

Bismuth gives an yellow colour with the reagent stable below an acid concentration of 0·03N, turning turbid on long keeping. The depths of colour with varying concentrations of bismuth were examined and found to be proportional, and independent of the amount of reagent present in excess. The range from 40 mg. to 0·4 mg. Bi per 25 c.c. has been followed by three sets of standards.

Experimental

The reagent was prepared by the method of Freund and Wischewiansky[5] in the form of white needle-shaped crystals from hydrazine sulphate (10 gm.) and ammonium sulphocyanide (15 gm.). The crystals were recrystallized from hot water (m.p., 208°-210°C., yield 5-6 gm.). Sulphur was determined by peroxide fusion (found : S, 43·23 per cent ; $C_2H_6N_4S_2$ requires : S, 42·66 per cent).

Cupric Dithiocarbamido-hydrazine — This was prepared in the form of a fine, black powder from a solution of copper sulphate (1 mol.) and dithiocarbamido-hydrazine (1 mol.), the latter being added to a solution of copper sulphate containing 2 c.c. of glacial acetic acid per 100 c.c. of solution. Precipitation was carried out from warm solution with constant stirring using a slight excess of a 2 per cent solution of the reagent. The product was thoroughly washed with warm water containing a few drops of dilute acetic acid ; dried in air and finally in a vacuum desiccator. Copper was estimated iodometrically after decomposition by concentrated nitric acid. Sulphur was estimated by peroxide fusion (found : Cu, 27·30 ; S, 28·55 ; Cu : S : : 1·00 : 2·07; $C_2H_4N_4S_2Cu.H_2O$ requires : Cu, 27·70 ; S, 28·00 per cent).

The substance was paramagnetic. The gram-molecular susceptibility, found with a magnetic balance of the Guoy type, was found to be 1·41 Bohr's magneton, without diamagnetic correction for the constituents (l=8·8 cm., w=0·2708g, Δw=5·97 mg., $H_{\max}$ =10·4 × 10^3 gauss).

Cuprous Dithiocarbamido-hydrazine — This was obtained as a white precipitate by adding a 2 per cent solution of the reagent to a warm solution of copper sulphate in 2N hydrochloric acid containing 1 gm. of sodium sulphite for every gram of copper. The precipitate was filtered through a Buchner funnel and washed with warm water containing a few drops of dilute hydrochloric acid. The free acid was finally removed by washing with absolute alcohol.

The product dried in a vacuum desiccator contains an appreciable quantity of free sulphur which could be isolated by extraction with carbon disulphide.

The substance was examined in a Curie balance and found to be diamagnetic.

Cadmium Dithiocarbamido-hydrazine — Cadmium is precipitated quantitatively as a white fine-grained powder from ammoniacal solutions by the reagent. The reagent was first added to an acidic solution of the cadmium salt which was subsequently made ammoniacal by dropwise addition of strong ammonia with constant stirring. The product after washing with water and drying in a vacuum desiccator to constant weight was analysed. Cadmium was estimated as sulphate by decomposition with concentrated nitric and sulphuric acids, and sulphur was estimated by peroxide fusion (found : Cd, 40·85 per cent, S, 25·4 per cent ; Cd : S : : 1·0 : 2·17). The compound was found unsuitable for estimation of cadmium by direct weighing.

Estimation of Copper — The black cupric compound was precipitated from a warm solution acidified with 2 c.c. glacial acetic acid per 100 c.c. of the liquid with a freshly prepared 2 per cent solution of the reagent. The precipitate, after settling for sometime on the water-bath was filtered and washed with warm water containing a few drops of dilute acetic acid. It was then decomposed with concentrated nitric acid and estimated iodometrically. The results are given in Table II.

TABLE II

Copper taken gm.	Copper found gm.	Error
0·1401	0·1403	+0·0002
0·0933	0·0930	−0·0003
0·0200	0·0202	+0·0002
0·0700	0·0696	−0·0004

The white cuprous compound is a heavy granular precipitate and more suitable for analytical work. It was obtained by precipitating a hot solution of copper containing one-fifth of its volume of concentrated hydrochloric acid and 1 gm. of sodium sulphite. The liquid filters rapidly and the precipitate, after thorough washing with hot acidulated water, was decomposed with concentrated nitric acid and the copper estimated iodometrically. On long standing, the precipitate has a tendency to turn grey. The results are given in Table III.

TABLE III

Copper taken gm.	Copper found gm.	Error
0·1120	0·1116	−0·0004
0·0467	0·0466	−0·0001
0·1401	0·1402	+0·0001
0·2102	0·2096	−0·0006
0·0560	0·0562	+0·0002

Separation of Copper from Iron — Copper was precipitated from 2N hydrochloric acid solution as previously described. The filtrate was oxidized with bromine water and iron precipitated as hydroxide with ammonia and ignited to oxide. The results are given in Table IV.

TABLE IV

Copper taken gm.	Iron taken gm.	Copper found gm.	Error	Iron found	Error
0·1455	0·0930	0·1452	−0·0004	0·0931	+0·0001
0·2182	0·0279	0·2176	−0·0006	0·0280	+0·0001
0·2910	0·0557	0·2905	−0·0005	0·0554	−0·0003
0·1455	0·1440	0·1457	+0·0002	0·1436	−0·0004
0·0873	0·3901	0·0875	+0·0002	0·3900	−0·0001
0·0582	0·5574	0·0580	−0·0002	...	...

Separation of Copper from Zinc — Copper was separated as the cuprous complex as before, and the zinc was estimated in the filtrate after treatment with bromine water as zinc ammonium phosphate. The results are given in Table V.

TABLE V

Copper taken gm.	Zinc taken gm.	Copper found gm.	Error	Zinc found gm.	Error
0·0291	0·3129	0·0289	−0·0002	0·3132	+0·0003
0·2843	0·0447	0·2840	−0·0003	0·0447	nil
0·2843	0·0223	0·2846	+0·0003	0·0222	−0·0001
0·0582	0·2682	0·0584	+0·0002	0·2673	−0·0004
0·2910	0·0536	0·2909	−0·0001	0·0534	−0·0002
0·1445	0·1341	0·1452	−0·0003	0·1338	−0·0003
0·0582	0·5360	0·0581	−0·0001	...	...

Estimation of Cadmium — A slight excess of a 2 per cent solution of the reagent was added to an acidified solution of the cadmium salt followed by dropwise addition of 5N ammonia with constant stirring. The heavy white precipitate was washed with hot water containing a few drops of ammonia. It was dried in the oven and decomposed to cadmium sulphate with concentrated nitric and sulphuric acids, and weighed as $CdSO_4$. The results are given in Table VI.

TABLE VI

Cadmium taken gm.	$CdSO_4$ obtd. gm.	Cadmium found gm.	Error
0·0804	0·1483	0·0800	−0·0004
0·0832	0·1540	0·0830	−0·0002
0·1110	0·2065	0·1114	+0·0004
0·0555	0·1024	0·0552	−0·0003
0·1665	0·3090	0·1666	+0·0001

Separation of Copper from Cadmium — The cuprous compound was precipitated from 2N hydrochloric acid solution and estimated iodometrically as before. The filtrate, after further addition of the reagent, was rendered ammoniacal to precipitate the cadmium compound which was finally decomposed and weighed as $CdSO_4$. The results are given in Table VII.

TABLE VII

Copper taken gm.	Cadmium taken gm.	Copper found gm.	Error	Cadmium found gm.	Error
0·0970	0·1110	0·0968	−0·0002	0·1107	−0·0003
0·0200	0·1110	0·0201	+0·0001	0·1106	−0·0004
0·0291	0·1665	0·0292	+0·0001	0·1666	+0·0001
0·2910	0·0277	0·2909	−0·0001	0·0275	−0·0002
0·1460	0·0277	0·1455	−0·0005	0·0274	−0·0003
0·2910	0·0555	0·2913	+0·0003	0·0551	−0·0004

Colorimetric Estimation of Bismuth — Pure bismuth solutions only have been examined in the present investigation. Three standard solutions were prepared to cover the whole range of working concentrations from purest basic bismuth nitrate freshly recrystallized from hot dilute nitric acid. Known volumes of standard solutions were evaporated in a crucible which was ignited at a low temperature and bismuth content determined as oxide. Freshly prepared aqueous solutions of the recrystallized reagent (0·5 per cent) were used, and the colour examined without delay in a Helige Duboscq colorimeter. In one set of experiments the colour was measured in a Lumetron photoelectric colorimeter EF using the blue filter. The results are given in Tables VIII, IX, X and XI.

TABLE VIII

Concentration range of bismuth, 40·35–6·725 *mg./25 c.c.; concentration of reagent used,* 0·5%; *concentration of standard,* 16·81 *mg./25 c.c.; fixed reading of " unknowns ",* 20·0 *mm.*

READINGS OF STANDARD (MEAN OF 3)	CONCENTRATION OF " UNKNOWNS " BY BEER'S LAW IN MG./25 C.C.	ACTUAL CONCENTRATION IN MG./25 C.C.
50·50	42·45	40·35
41·74	35·10	33·62
37·50	31·52	30·26
33·08	27·81	26·90
28·72	24·15	23·54
24·40	20·51	20·17
20·06	16·86	16·81
15·68	13·18	13·45
13·49	11·34	11·76
11·25	9·46	10·08
9·10	7·65	8·40
6·89	5·83	6·72

TABLE IX

Concentration range of bismuth, 6·725–1·345 *mg./25 c.c.; concentration of reagent used,* 0·1%; *concentration of standard,* 3·362 *mg./25 c.c.; fixed reading of " unknowns ",* 20·0 *mm.*

READINGS OF STANDARD (MEAN OF 3)	CONCENTRATION OF " UNKNOWNS " BY BEER'S LAW IN MG./25 C.C.	ACTUAL CONCENTRATION IN MG./25 C.C.
41·78	7·02	6·72
37·48	6·28	6·05
33·00	5·54	5·38
28·68	4·82	4·70
24·40	4·10	4·03
20·11	3·37	3·36
15·70	2·63	2·69
13·59	2·28	2·35
9·00	1·51	1·68
7·01	1·17	1·34

TABLE X

Concentration range of bismuth, 1·345–0·4035 *mg./25 c.c.; concentration of reagent used,* 0·05%; *concentration of standard,* 0·6725 *mg./25 c.c.; fixed reading of " unknowns ",* 20·0 *mm.*

READINGS OF STANDARD (MEAN OF 3)	CONCENTRATION OF " UNKNOWNS " BY BEER'S LAW IN MG./25 C.C.	ACTUAL CONCENTRATION IN MG./25 C.C.
41·70	1·40	1·34
37·29	1·25	1·21
33·01	1·11	1·07
28·70	0·96	0·94
24·41	0·82	0·80
20·00	0·67	0·67
17·80	0·59	0·60
15·69	0·52	0·53
11·39	0·38	0·40

TABLE XI — LUMETRON MEASUREMENTS

Concentration of standard bismuth solution, 0·574 *mg./c.c.; reagent used,* 10 *c.c. of* 0·5% *solution; total volume* (33 *c.c.*) *same in all cases.*

C.C. BISMUTH SOLUTION	% TRANSMISSION (LUMETRON READINGS)	O.D. × 10^2
1·0	100	0
1·5	87	6
2·0	74	13
2·5	64	19
3·0	54·5	26
3·5	46·5	33
4·0	41	39
4·5	36	44

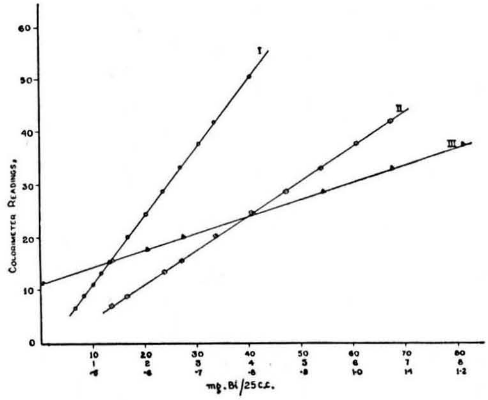

FIG. 1

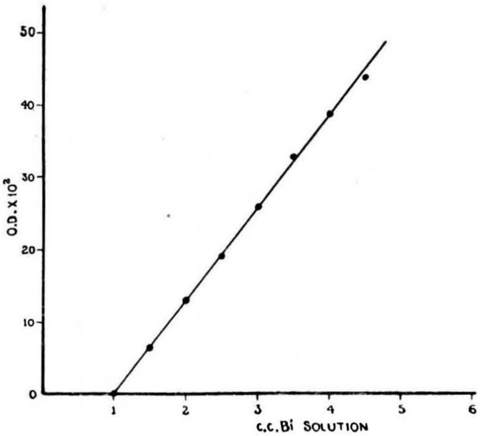

FIG. 2

The results of Tables VIII, IX and X are plotted graphically in Fig. 1, and of Table XI in Fig. 2. The variation, it will be seen, is linear.

Summary

Dithiocarbamido-hydrazine, $NH_2CSNH.NHCSNH_2$, a simple derivative of thiourea, has been found to precipitate copper and cadmium quantitatively in acid and ammoniacal solutions respectively, and to give an orange-yellow soluble complex with bismuth. Methods have been developed for the quantitative estimations of copper and cadmium, the separation of copper from cadmium, iron and zinc and for the colorimetric determination of bismuth.

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Base Exchange, Adsorption, Permeability & Related Properties of Soils

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QUANTITATIVE relationship exists between base exchange capacity (B.E.C.) and the different properties of soils, such as adsorption, exchange reaction, swelling and permeability. The other soil constants affecting these properties are the clay content and the nature of the exchangeable cation.

The following is a summary of the main results obtained by us and is published in the hope that they may be of interest to workers on similar lines elsewhere. The investigation was originally started at the instance of the Irrigation Research Section of the U.P. Government, but now discontinued. The details of experiments and results have been published in the Government Technical Memoranda, which, however, are not easily available to the public. The original problem was to find a suitable lining material for canal beds to prevent losses of water by seepage. The method ultimately recommended involved the treatment of a layer of soils one inch thick with sodium carbonate. A cheaper alternative was found in the substitution of sodium carbonate by a layer of the naturally ocurring alkaline soil or *usar*, which, in experimental canals, yielded satisfactory results. Our problem was thus the counterpart of the problem of the reclamation of alkaline soils referred to by Ramdas recently[1]. Both the problems involve the close study of the properties and behaviour of different kinds of soils from practical and theoretical points of view, on which Ramdas and collaborators have already contributed a large number of papers.*

Adsorption & Base Exchange — If a pure calcium soil is treated with a solution of sodium carbonate, the following equilibrium reaction takes place:

$$Ca(Z)+Na_2CO_3 \rightleftarrows Na_2(Z)+CaCO_3$$

This equation does not express all the significant facts connected with the reaction. There is firstly the adsorption of sodium carbonate, and secondly there is

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* The main observations of Ramdas, especially those relating to soils treated with sodium carbonate and calcium chloride are in conformity with our own. The present paper deals only with some of the mathematical relationships which we have investigated.

the base exchange. The two are related but not equivalent. As a rule the sodium carbonate adsorbed is greater than the equivalent of Ca exchanged. Hence the two aspects may be treated separately; the first may be termed general adsorption, while the second leading to the base exchange as exchange adsorption.

A series of adsorption experiments with different strengths of sodium carbonate revealed the fact that the adsorption of sodium carbonate by soils is governed by the well-known Freundlich's adsorption isotherm:

$$y = aC^{1/n}$$

where y is the quantity of sodium carbonate adsorbed, C the equilibrium concentration, a and n, constants.

A closer study of these two arbitrary constants has shown that " a " is a function of the most important soil constant, the base exchange capacity (B.E.C.). The following table illustrates the idea.

TABLE I

Soil No.	Clay %	Silt %	B.E.C. (B)	a	n	a/B²
1	52·43	39·20	13·40	0·0497	1·110	$28{\cdot}0\times10^{-5}$
2	27·44	50·25	14·80	0·0721	0·912	$32{\cdot}6\times10^{-5}$
3	16·53	23·08	10·00	0·0352	0·816	$35{\cdot}2\times10^{-5}$
4	6·00	18·50	7·90	0·0186	0·816	$29{\cdot}0\times10^{-5}$
5	27·60	58·90	20·52	0·1350	0·924	$32{\cdot}0\times10^{-5}$
6	26·60	58·90	11·60	0·0404	0·823	$30{\cdot}0\times10^{-5}$
7	24·53	44·24	16·60	0·0854	0·924	$31{\cdot}0\times10^{-5}$
8	21·85	17·36	9·88	0·0303	0·812	$31{\cdot}0\times10^{-5}$
9	26·32	21·24	7·11	0·0160	0·824	$31{\cdot}7\times10^{-5}$

From the last column of the table it is evident that " a " varies as the square of " B ", the base exchange capacity, which is illustrated graphically in Fig. 1, where the relation between " a " and " B " and that between " a " and " B^2 " have been plotted. We can now express the Freundlich equation in the form:

$$y = 31\times10^{-5}\ B^2\ C^{1/n}$$

in which we have substituted the arbitrary constant " a " by a term involving B.E.C. The value of " n " varies within narrow limits, i.e. between 0·8 and 1·1. Further work is necessary to correlate " n " with other characteristic constants of the soil.

Exchange Reaction — It is a peculiarity in surface reactions that adsorption and exchange do not follow the same laws though the two go hand in hand. The adsorption curve is a *parabola*, while the exchange curve is a *hyperbola*. Many attempts have been made to give quantitative expression to the exchange reaction, and a number of equations proposed by various workers, of which two are:

$$(1)\quad x = \frac{hC}{I + bC} \quad (\text{ Langmuir })$$

$$(2)\quad x = \frac{S\ I}{I + K} \quad (\text{ Vageler })$$

where x = base exchanged, C = equilibrium concentration, I = initial concn., h, b, K, S constants.

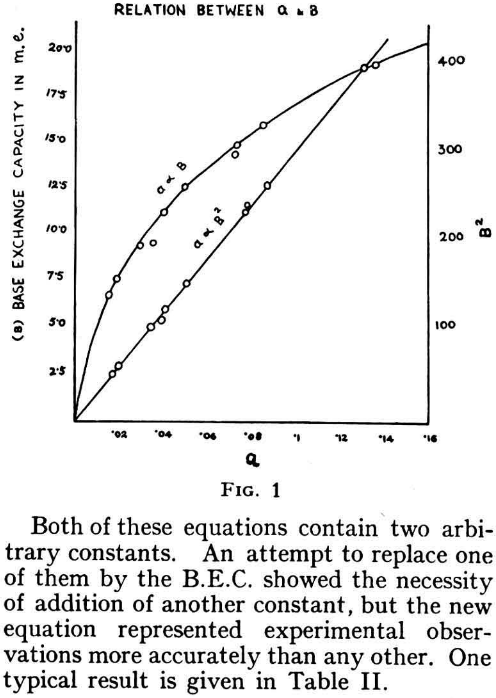

FIG. 1

Both of these equations contain two arbitrary constants. An attempt to replace one of them by the B.E.C. showed the necessity of addition of another constant, but the new equation represented experimental observations more accurately than any other. One typical result is given in Table II.

TABLE II

Initial concn. Na_2CO_3 (m.e.)	Ca exchanged observed (m.e.)	Ca exchanged calculated according to		
		Langmuir	Vageler	Nayer & Shukla
10	0·80	(0·80)	(0·80)	(0·80)
12	0·88	0·867	0·949	0·883
14	0·96	0·953	1·091	0·965
16	1·05	0·992	1·239	1·053
18	1·12	1·000	1·379	1·125
20	1·21	1·158	1·506	1·213
25	1·40	(1·400)	(1·400)	(1·400)

A comparison of the figures in the 2nd and last columns shows that agreement between observed and calculated values is most satisfactory. The merit of the new equation expressed as

$$x = \frac{p + I\ B}{q + I}$$

(where x = Ca exchanged, I = initial concn., B = B.E.C., and p, q, constants) is that it takes into account the most important property of the soil, viz. its B.E.C. Further, Langmuir's and Vageler's equations were found to be not applicable at very *low* or very *high* concentrations, while the proposed equation is applicable within wide limits. Thus it is seen that in adsorption and exchange reactions the B.E.C. plays an important rôle, and the course of each reaction can be mathematically predicted.

An Equation for the Percolation of Water in Ca-Na Soils — It is well known that sodium soils are generally impermeable to water. When a Ca soil is treated with a sodium salt, exchange takes place between Ca and Na ions, and differing amounts of sodium salt added lead to the formation of different grades of Na-Ca soils. The permeability of such soils containing two such ions can be expressed by the equation (Harris) :

$$Y = a.\ e^{-bS}$$

where Y = the rate of percolation of water, S represents the percentage saturation of the soil complex with respect to Na ion, that is,

$$S = \frac{\text{Exch. Na}}{\text{B.E.C.}} \times 100$$

and a and b, constants. This equation has been tested in a number of cases and has been found satisfactory. Our object was to find the relation, if any, existing between the arbitrary constants, a and b, and the characteristic properties of soils. The following table gives a summary of the results obtained :

TABLE III

Soil No.	Clay content (C)	B.E.C. (B)	Constant (a)	Constant (b)	a log C	b/B
12	27·44	14·80	0·136	0·0283	0·1967	$1{\cdot}9\times10^{-3}$
2	57·60	20·50	0·136	0·0400	0·1967	$2{\cdot}0\times10^{-3}$
10	26·60	16·60	0·154	0·0320	0·2290	$1{\cdot}9\times10^{-3}$
3	52·43	13·40	0·083	0·0250	0·1430	$1{\cdot}8\times10^{-3}$
13	21·85	9·88	0·178	0·0180	0·2380	$1{\cdot}8\times10^{-3}$
15	26·32	7·11	0·138	0·0130	0·1990	$1{\cdot}8\times10^{-3}$

The significance of the values in the last two columns will be easily understood. The constant " b " is directly related to the B.E.C., while the constant " a " is a function of the clay content. The small variations of values in column 6 might be due to the presence of secondary particles. To clear this point, experiments were carried out with artificial mixtures of the colloidal part of the soil and pure sand (as an inert constituent) with the following result :

TABLE IV

Soil No.	Clay content (C)	B.E.C. (B)	(a)	(b)	a log C	b/B
1	100	31·8	0·129	0·057	0·2580	$1{\cdot}8\times10^{-3}$
2	50	15·9	0·148	0·028	0·2500	$1{\cdot}8\times10^{-3}$
3	30	9·54	0·177	0·018	0·2632	$1{\cdot}9\times10^{-3}$
4	25	7·95	0·188	0·016	0·2492	$1{\cdot}8\times10^{-3}$
5	20	6·36	0·195	0·012	0·2542	$1{\cdot}9\times10^{-3}$

" a log C " is thus seen to be definitely a constant and the variation of the values in column 6 of the previous table must be attributed to the presence of secondary particles (size : 0·02-0·002 mm.).

If we now substitute the new values for a and b in Harris's equation, we obtain the expression :

$$Y = \frac{k_1}{\log C}\ e^{-k_2\ BS}$$

And since $S = \dfrac{(\text{Na})}{B} \times 100$

the final equation is

$$Y = \frac{k_1}{\log C} . e^{-k\ (\text{Na})}$$

where k_1 has the value round about 0·25 and k is almost exactly equal to 0·18, so that an approximate equation for percolation of water through Na-Ca soils is

$$Y = \frac{0{\cdot}25}{\log C}\ e^{-0{\cdot}18\ (\text{Na})}$$

where C is the clay content, and (Na) is the exchangeable sodium present in the soil complex. Thus the arbitrary constants in Harris's equation have been replaced by soil constants and numerical constants by an application of ideas underlying B.E.C.

Similar equations have been obtained in the case of soils treated with monovalent and divalent cations, which, however, need not be discussed here.

The Influence of the Size of the Exchangeable Cation — The soil permeability, as measured by the rate of percolation of water, seems also to depend on the radius of the exchangeable cation. Experiments carried out with soils *saturated* with different cations show that under comparable conditions the rate of percolation is expressed by the equation :

$$Y = p.e^{qR}$$

where R is the radius of the cation, p and q being constants. When log Y is plotted against R, a straight line is obtained for the same series of ions ; that is, the monovalent ions Li, Na, NH_4, K, Rb all lie in one straight line, while the divalent alkaline earth ions Mg, Ca, Sr, Ba lie in another[8]. This means that the rate of percolation of water through soil colloids saturated with cations is an exponential function of the ionic radius of the exchange cation.

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Studies in the Constitution of Euphoron — Part I

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IN an earlier communication[1] dealing with the characterization of the polycyclic ketone, euphoron, isolated from the dried latex of *Euphorbia tirucalli*, Karimullah and Gopalachari stated that the exact molecular formula for the ketone could not be definitely fixed at that stage of the investigations, as the molecular weight (390 : Rast) and the C, H values of the ketone agreed with the formula $C_{27}H_{42}O$, while the iodine value of the ketone and the nitrogen value of its oxime along with their C, H values were found to agree more closely with a C_{29} or C_{30} formulation. The present communication deals with the results of further studies in the constitution of euphoron. From the evidence now available it is possible to definitely fix on a C_{30} formulation for the ketone, $C_{30}H_{48}O$. The molecular weight of the ketone (422), determined on the basis of the saponification equivalent of the acetyl ester of the alcohol (euphorol) derived from it, is in good accord with this formula and excludes the alternatives suggested earlier.

The absorption spectrum of euphoron (FIG. 1) shows two bands, one in the region of 2200Å $\left(E^{1\text{ cm.}}_{1\%} = 10\right)$ and the other in the region of 2890Å $\left(E^{1\text{ cm.}}_{1\%} = 0{\cdot}785\right)$. There are, however, none in the region of 2400-2500Å which would indicate the absence of any conjugation between either of its two ethylenic linkages and the carbonyl double bond[2].

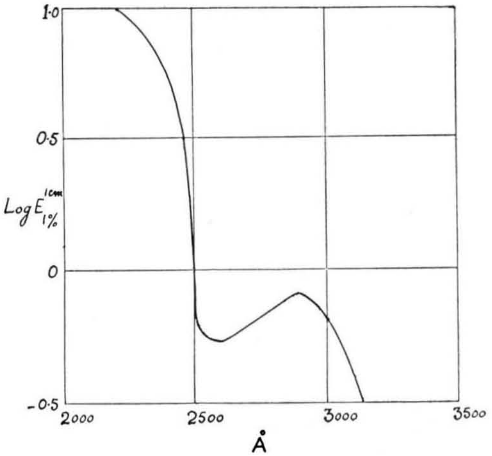

FIG. 1 — ULTRA-VIOLET ABSORPTION SPECTRUM OF EUPHORON.

As reported by the earlier authors, catalytic reduction of euphoron with platinum oxide gave dihydro-euphoron, $C_{30}H_{50}O$. Further reduction of dihydro ketone with sodium and moist ether has yielded dihydro-euphorol, $C_{30}H_{52}O$. The molecular weight of this alcohol, as derived from the saponification equivalent of its acetate, is in good accord with the formula assigned to it. Oxidation of euphoron with chromic acid in glacial acetic acid medium yielded a ketonic acid which analyses for $C_{29}H_{46}O_3$ and has been provisionally named euphoronic acid.

As noted by the earlier authors, the use of amyl alcohol and sodium in the reduction of euphoron to the corresponding alcohol euphorol was attended with considerable resinification, which lowered the yield of euphorol and necessitated a tedious repetition of fractional crystallizations for arriving at a pure product. With the use of moist ether in place of amyl alcohol for reduction, it has now been possible to obtain pure euphorol after a couple of crystallizations in a nearly theoretical yield. Euphorol, $C_{30}H_{50}O$, has two double bonds like the corresponding ketone and is similarly reduced to a dihydro derivative, $C_{30}H_{52}O$ (dihydro-euphorol), on catalytic reduction with platinum oxide. Further work on the constitution of these products is in progress.

Experimental

Oxidation of Euphoron to Euphoronic Acid— A solution of chromic acid (2·5 gm.) in 125 c.c. of glacial acetic acid was added drop by drop in the course of 4 hours into a solution of the ketone (5 gm.) in 400 c.c. of glacial acetic acid kept over a boiling water bath. After heating for a further period of 2 hours the solution was concentrated *in vacuo* and then poured into water. The precipitate formed was extracted with ether and the ethereal solution was shaken out with 10 per cent solution of sodium carbonate. The alkaline aqueous layer was neutralized with dil. hydrochloric acid and the precipitate of the crude acid thus obtained was washed, dried and crystallized from methyl alcohol, when euphoronic acid was obtained in the form of colourless needles in a poor yield (0·1 gm.), m.p. 189°-190°. It is soluble in ether, and chloroform in the cold and in methyl and ethyl alcohols in the hot. On crystallization from alcohol, the neutral portion of the oxidation products gave the unreacted ketone (2 gm.) and a viscous liquid (2·5 gm.) from its mother liquor. Found : C, 78·6 per cent ; H, 10·6 per cent ; neutralization equivalent, 446. Required for $C_{29}H_{46}O$: C, 78·7 per cent ; H, 10·4 per cent ; neutralization equivalent, 442. Required for $C_{29}H_{48}O$: C, 78·4 per cent ; H, 10·8 per cent ; neutralization equivalent, 444.

Oxime of Euphoronic Acid — 1 gm. of hydroxyl amine hydrochloride was dissolved in 2 c.c. of water and added to 100 c.c. of N/10 alcoholic potash. The solution was filtered from the potassium chloride formed and the filtrate refluxed with 0·1 gm. of euphoronic acid on a water bath. It was then diluted with water and the precipitate formed was filtered, washed free of alkali, and dried on a porous plate. On crystallization from alcohol, the oxime was obtained in the form of fine, rectangular plates soluble in ether, chloroform, and alcohol, m.p. 214°-215°.

Euphorol — A solution of euphoron (5 gm.) in 250 c.c. of ether was taken in a round-bottom flask fitted with a reflux condenser, and 100 c.c. of distilled water was added to it. When the ethereal layer had separated from the lower aqueous layer, 7·5 gm. of sodium were added in small bits in the course of 4 hours. After the completion of the reaction, the ethereal layer was washed free of alkali, dried over anhydrous sodium sulphate, and filtered. On removal of the solvent from the filtrate and crystallization of the nearly colourless crystalline residue from alcohol, pure euphorol, m.p., 108°-109°C., was obtained ; $[\alpha]_D^{35°} = +24$; found : C, 84·8 per cent ; H, 11·9 per cent ; M.W., 424 ; required for $C_{30}H_{50}O$: C, 84·4 per cent : H, 11·7 per cent ; M.W., 426.

Euphorol Acetate — A solution of euphorol in 10 c.c. of dry pyridine was warmed on a water bath and treated with 5 c.c. of acetic anhydride. After half an hour the solution was cooled and diluted with water. The precipitate formed was extracted with ether and the ethereal solution successively shaken out with dil. hydrochloric acid, dil. alkali, and water. After drying over anhydrous sodium sulphate, the solution was filtered and freed from the solvent. On crystallization from alcohol, the nearly colourless crystalline residue gave the acetate in the form of colourless needles, readily soluble in ether and chloroform in the cold and in alcohol and acetone in the hot ; m.p. 107°-108°, $[\alpha]_D^{32°} = +28{\cdot}5$. Found : C, 82·4 per cent ; H, 11·3 per cent ; saponification equivalent, 466, 467, 464. Required for $C_{32}H_{52}O_2$: C, 82·1 per cent ; H, 11·1 per cent ; saponification equivalent, 468.

Dihydro-euphorol from Euphorol — A solution of 1 gm. of euphorol was shaken with 0·1 gm. of platinum oxide (Roger-Adams) in a long-necked flask connected to a graduated hydrogen reservoir. The absorption was complete in 24 hours after which the catalyst was filtered off. On concentrating the filtrate, dihydro-euphorol crystallized out in the form of colourless needles, readily soluble in ether and chloroform in the cold and in methyl and ethyl alcohols in the hot ; m.p. 122°-123°, $[\alpha]_D^{28°} = +36{\cdot}0$.

Dihydro euphorol from Dihydro-euphoron — Dihydro-euphoron (5 gm.) dissolved in 250 c.c. of ether was reduced with sodium (7·5 gm.) following the method used for the reduction of euphoron to euphorol. The reduced product was crystallized from alcohol when it gave dihydro-euphorol in the form of colourless needles melting at 122°-123°. When mixed with the dihydro-euphorol obtained from euphorol by catalytic reduction, it showed no depression in m.p. Found : C, 84·1 per cent ; H, 12·3 per cent ; M.W., 431 ; volume of H absorbed, 4·96 lit./100 gm. Required for $C_{30}H_{52}O$: C, 84·1 per cent ; H, 12·2 per cent ; M.W., 428 ; volume of H required for one double bond, 5·25 lit./100 gm.

Dihydro-euphorol Acetate — Dihydro-euphorol, on treatment with acetic anhydride and dry pyridine, as in the preparation of euphorol acetate, gave the acetate in the form of colourless needles which were readily soluble in ether, and chloroform in the cold and alcohol in the hot, m.p. 125°-126°, $[\alpha]_D^{32°} = +29$. On saponification with N/2 alcoholic potash, it gave dihydro-euphorol, m.p. 122°-123°. Found : C, 81·5 per cent ; H, 11·5 per cent ; saponification equivalent, 474. Required for $C_{32}H_{54}O_2$: C, 81·7 per cent ; H, 11·5 per cent ; saponification equivalent, 470.

Acknowledgement

The authors take this opportunity to express their thanks to Dr. Weiler, Microanalytical Laboratory, Oxford, for the analytical data and the absorption spectrum curve incorporated in this publication.

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Some Experiments in Kier Boiling

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THE bleaching of woven cotton goods ordinarily consists of the following three operations, viz. (a) desizing or the removal of the greater part of starch added in sizing ; (b) kier boiling, resulting in the removal of waxy, fatty and other materials such as proteins, pectins, etc. ; and (c) bleaching proper or the destruction of natural colouring matter in the cotton. The skill of the bleacher lies in the efficient removal of the impurities present in the cotton, with the consequent whiteness, with the least damage to the fibres from the point of view of their textile quality. Efficient kier boiling in alkaline liquors is the foundation of successful bleaching. During the kier boiling process, the waxes, oils and fats are in part saponified and in part emulsified, mechanically held dirt is loosened and sizing materials are solubilized. These actions should proceed as nearly as possible to completion. A permanent white with a brilliance of tone is ultimately required for the cloth. On the other hand, cloth required for dyeing and printing should be, as far as possible, free from fatty and resinous matter so that it is highly and uniformly absorbent.

The present investigation was undertaken with a view to study the efficiency of a few specially designed kier-boiling treatments in removing the waxy and fatty matter from cotton cloth and yarn so as to render them suitable for the dyeing and printing processes.

Materials

Cloth — Commercial samples of twill and longcloth made locally from American cotton were used. In one set of experiments, yarn made from short staple Indian cotton was used. The twill in the grey condition contained 2·8 per cent and the longcloth 1·8 per cent of total fatty matter respectively. The yarn contained 0·6 per cent total fatty matter.

The various chemicals used in the boiling and bleaching experiments were commercial products used in the textile trade.

Colecton B — This product is marketed by the *Collective Chemical Company Ltd.*, Eagle Road, Bristol, England, as a powerful assistant in laundry washing. It is considered to possess excellent degreasing properties. We are greatly indebted to Dr. K. Venkataraman, Director, Department of Chemical Technology, for making this product available to us for these investigations.

The details of the kier-boiling experiments are given below.

Experimental Methods

The boiling was carried out in an experimental kier of 3 lb. capacity with a material liquor ratio 1 : 6. The arrangement of the kier and the external heater for liquor is shown in Fig. 1. The cloth was desized as described under each treatment and kier boiled for 6 hr. under 25 lb. steam pressure. The boiled sample was thoroughly washed and bleached with bleach liquor containing 1·5 gm. of available chlorine per litre, and adjusted approximately to a *p*H of 10·5. At the end of bleaching, the sample was washed and soured using a half per cent hydrochloric acid solution, washed again free of acid, dried and conditioned.

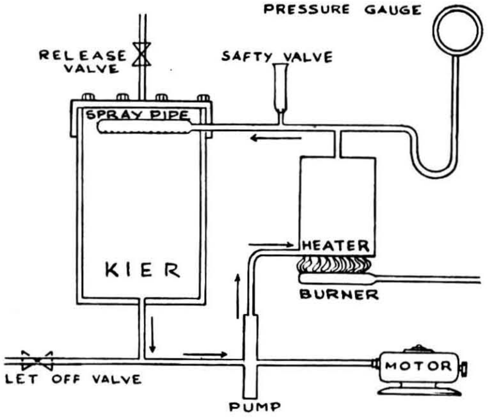

FIG. 1 — DIAGRAMMATIC REPRESENTATION OF THE EXPERIMENTAL KIER AND HEATER. The arrows indicate the direction of liquor flow.

Determination of Total Fatty Matter

The total fat content in the bleached and unbleached samples was determined by extracting a known weight in a Soxhlet apparatus for 8 hr. using a 1 : 1 mixture of dry benzene and absolute alcohol. The extract was dried to constant weight at 105°-110°C. and the residual fatty matter expressed on the weight of dry material after correcting for hygroscopic moisture.

The Kier-boiling Treatment

(1) The grey twill was desized using an enzyme preparation, washed thoroughly and kier boiled using the following 3 recipes:

All chemicals are expressed as percentages on weight of cloth.

(a) 3 per cent caustic soda, 1 per cent soda ash and 0·2 per cent Lissapol C;
(b) as in (a) except that Lissapol C was replaced by 0·2 per cent Turkey Red oil;
(c) the desized sample was first boiled using 2 per cent lime and 0·2 per cent Lissapol C, washed and again boiled using 3 per cent caustic soda, 1 per cent soda ash and 0·2 per cent Lissapol C.

(2) The twill was desized by grey chemicking with a bleaching powder solution containing 2 gm. of available chlorine for 2 hr. and washed free from chlorine. It was then kier boiled as given below:

(d) 3 per cent caustic soda;
(e) kier-boiled sample from (d) was again boiled in the open with 0·33 per cent Colecton B and 3 per cent soda ash for 2 hr.

(3) (f) The grey twill was desized as in B and boiled in the open using 0·33 per cent soda ash for 3 hr.

(4) Grey yarn was kier boiled using
(g) 3 per cent caustic soda and 0·25 per cent sodium silicate;
(h) 3 per cent caustic soda and 0·2 per cent Lissapol C;
(i) 3 per cent soda ash and 0·33 per cent Colecton B in the open for 3 hr.

(5) The grey longcloth was desized as in (2) and kier boiled as given below:
(j) 0·4 per cent Colecton B and 3 per cent soda ash, and
(k) 3 per cent caustic soda and 0·2 per cent Lissapol C.

Experimental Results

The results are summarized in Table I.

The saponifiable waxes are normally converted into soaps by the action of alkalis in the kier but the unsaponifiable fatty matter needs special methods for its removal. Of the 4 detergents employed, it is seen that in the case of open soda boil with Colecton B, the saponification of the saponifiable waxes is not complete, but the quantity of total

TABLE I

CLOTH VARIETY	BEFORE BOILING		SAMPLE NO.	RESIDUAL FAT % AFTER BOILING	DETERGENT USED
	Total fatty matter %	Unsaponifiable fatty matter %			
Grey twill	2·8	0·8	a	0·61	Lissapol C
			b	0·91	Turkey Red oil
			c	0·53	Two boils with Lissapol C
			d	0·69	nil
			e	0·40	Colecton B
			f	0·41	do
Grey yarn	0·6	0·32	g	0·36	Sodium silicate
			h	0·26	Lissapol C
			i	0·15	Colecton B
Grey longcloth	1·9	0·9	j	0·41	do
			k	0·51	Lissapol C

residual fatty matter in the cloth and the yarn has fallen to a very small value. Scholefield and Ward[1] have shown that under the experimental conditions employed by them using Lissapol A, the wax content of cloth fell to a very small value. In the present investigation, it is seen that the wax content of the cloth even after 2 alkaline boils to which Lissapol C was added, the residual wax content in the boiled cloth is quite appreciable. In view of the observations recorded by Ward and Scholefield , this appears to be surprising.

Turkey Red oil and sodium silicate appear to be the least effective of all in the emulsification of unsaponifiable fatty matter in the cloth since experiments carried out in absence of any assistant in the kier show much better scouring efficiency (d). Comparing these results with those obtained with Colecton B, it is seen that its emulsifying ability is quite satisfactory. Even by a weaker alkaline treatment of shorter duration, carried out at temperatures lower than those employed in normal kier-boiling processes, the wax content in both the cloth samples and yarn has fallen to a much smaller value.

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Preliminary Investigations on Brick Clays in India

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A SURVEY of clays used for brickmaking in various parts of India has been undertaken by the *Building Research Unit* with a view to ascertain the analytical factors that go to form a good brick. A large number of clay samples were received through the courtesy of provincial governments and states for examination and analysed. The present paper interprets a few of the results obtained.

The brick clays were lightly pounded to break the clods, air dried and sieved through a standard sieve No. 40. Samples passing through a 2 mm. (No. 10) sieve were used for mechanical analysis by the Bouyouco's hydrometer method, using sodium silicate as deflocculating agent.

The liquid and plastic limits and the plasticity index were determined according to *A.S.T.M.* D-423 and 424-39 methods[1-3] respectively. The linear and volumetric shrinkages were determined by the methods prescribed by the *U.S. Bureau of Public Roads*, and the *p*H by the Beckman glass electrode *p*H meter in 1 : 5 soil-water extracts.

The results are presented in Table I.

TABLE I — PROPERTIES OF BRICK CLAYS IN INDIA

Ser. No.	Locality	Colour	Workability	User's opinion	Mechanical composition %			Liquid limit	Plasticity index	Shrinkage %		pH
					Sand	Silt	Clay			Liner	Volumetric	
1.	Jullundur (Punjab)	Pale yellow	Medium	Good	33	48	19	28	7	4	35	7·5
2.	Mangalore (Madras, W.C.)	do	High	Medium	17	19	64	58	28	13	66	7·0
3.	Dhanbad (Bihar)	Reddish yellow	Medium	Bad	45	28	27	36	12	9	30	6·1
4.	Madras	do	do	Medium	34	32	34	39	20	13	58	7·5
5.	Ootacamund (Madras)	Black	High	Bad	24	26	50	57	23	16	95	8·1
6.	Pipariya (C.P.)	Yellow brown	Low	do	61	22	16	25	9	5	23	7·7
7.	Muzaffarpur (Bihar)	Grey	High	Medium	3	43	54	48	21	7	42	7·8
8.	Chinsura (Bengal)	Ash	do	do	15	44	41	47	23	10	59	7·8
9.	Poona (Bombay)	Red brown	Low	Bad	48	30	22	33	2	4	25	8·0
10.	Sholapur (Bombay)	Brown	do	do	40	30	30	37	13	11	60	8·2
11.	Roorkee (U.P.)	Light yellow	Medium	Good	45	25	30	32	14	6	30	8·2
12.	Delhi	Yellow	do	do	26	52	22	34	18	6	29	8·2
13.	Panchmarhi (C.P.)	Red	Low	Bad	37	28	35	35	14	11	47	8·0
14.	Coimbatore (Madras)	Brown	do	do	45	33	22	62	28	15	88	8·0
15.	Vellore (Madras)	Yellow	High	do	14	46	40	60	26	15	73	7·8
16.	Chittoor (Madras)	Golden yellow	Medium	...	26	35	39	43	17	9	49	8·0
17.	Bhandara (C.P.)	Brown black	do	Bad	50	23	27	36	15	7	32	7·6
18.	Salem (Madras)	do	High	do	14	34	52	56	29	11	87	7·9
19.	Ferozepur (Punjab)	Grey	Medium	Good	19	30	51	35	13	5	29	8·2

Discussion

The bricks used in India are usually hand-made, and for this purpose the brick clay should be soft and easy to work.

It can be seen from Table I that the clays of the Indo-Gangetic alluvium and those from the west and east coasts of peninsular India generally give good bricks.

The results of mechanical analysis provide an indication of the plasticity of the clays. The sample from Pipariya has a high sand percentage and low plasticity. Muzaffarpur clay has only 3 per cent sand but still gives a poor brick. The calcarious nature of the clay gives the brick a creamy appearance. Samples 9 and 17 show high sand contents and bricks prepared from them are of poor quality.

The liquid limit of a clay indicates its capillary capacity and influences the plasticity index which in turn determines the cohesive property. With increasing plasticity the cohesive characteristics increase, while the permeabilities decrease[4]. The liquid limit is influenced by the clay fraction. In the case of Coimbatore and Vellore samples, the high values for liquid limit suggest that either "active" clay minerals or unusually fine particles are present in the clay fraction; a combination of the two factors[5] may also contribute to the high value. The value of liquid limit for suitable brick clays lies between 35 and 45.

The index of plasticity is greatly influenced by the percentage of sand present in the clay; the higher the sand fraction the lower is the index of plasticity. This will be clear from the figures for Poona and Pipariya clays. Materials whose plasticity index lies between 10 and 25 are suitable for brick-making, although for making bricks by hand a value above 25 is considered desirable. The plasticity index for Jullundur clay is 7, and yet the bricks made from it are good. This low value is due to the high silt and low clay content. It has been observed that materials containing equal percentages of clay and silt are easily workable, and are suitable for brick-making.

Volumetric shrinkage is influenced by the silt content of materials. This observation has been corroborated by the work done on soil stabilization, as applied to low-cost housing, carried out in the *Building Research Unit*[6].

Linear shrinkage is related to the liquid limit of the plastic mass. The higher the liquid limit the higher the linear shrinkage. The range of percentage linear shrinkage considered satisfactory for brick-making is to 5 to 8. Volumetric shrinkage is also related to the liquid limit. The shrinkage is considered high when the value exceeds 60 per cent, medium when it is between 30 and 60, and low when the value is below 30 per cent. Materials which show high values are unsuitable for brick-making — as bricks made from them develop cracks under service conditions. Clays with medium shrinkage values are favoured for brick-making.

The properties of good brick clays are summarized in Table II.

TABLE II

PLACE	MECHNICAL ANALYSIS, %			LIQUID LIMIT	PLASTICITY INDEX	VOLUMETRIC SHRINKAGE %
	Sand	Silt	Clay			
Jullundur	33	48	19	28	7	35
Ferozepur	19	30	51	35	13	29
Roorkee	45	25	30	32	14	30
Muzaffarpur	3	43	54	48	21	42
Chinsura	15	44	41	47	23	59
Delhi	26	52	24	34	18	29
Mangalore	17	19	64	58	28	66
Madras	34	32	34	39	20	59

The author's thanks are due to Dr. Gurdas Ram, Officer-in-Charge, *Building Research Unit*, Roorkee, for his interest in the work and for many helpful suggestions.

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Letters to the Editor

SOLUBILITY AND HEAT OF SOLUTION OF RIBOFLAVIN (VITAMIN B_2) IN WATER

SENGUPTA AND GUPTA[1] HAVE DETERMINED the solubility of riboflavin at different temperatures, plotted the solubility-temperature curve and extrapolated the solubility curve down to 0°C.

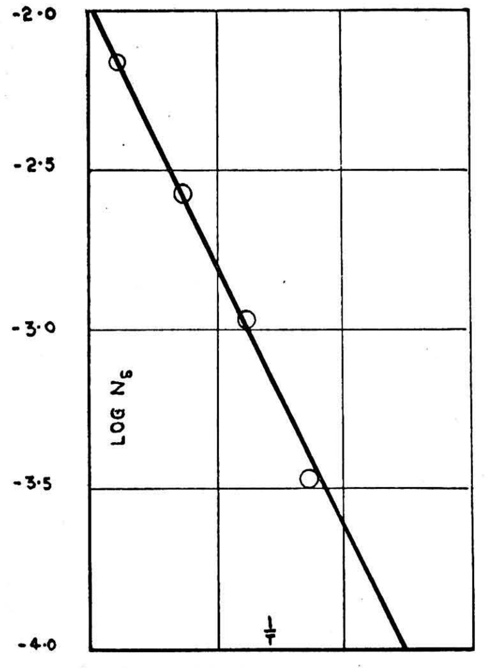

FIG. 1

Such direct extrapolation is, however, rendered unreliable by the high curvature of the solubility curve. A better procedure in such cases is to employ the following equation :

$$\frac{d\ln N_s}{dT} = \frac{L_s}{RT^2} \text{ or } \log_{10} N_s = -\frac{1}{2{\cdot}303}\frac{L_s}{RT} + \text{const.}$$

where N_s=molar fraction of solute in the saturated solution ;
L_s=differential heat of solution at saturation (assumed to be constant over the range of temperature) ;
R=gas constant ; and
T=absolute temperature.

Fig. 1 gives the straight line graph obtained by plotting $\log_{10}N_s$ versus $\frac{1}{T}$ which, on extrapolation, gives for solubility of riboflavin at 0°C. the value of 2·9 mg. per 100 c.c. as against 8 mg. per 100 c.c. as shown by the graph of Sengupta and Gupta. From the slope of the curve in Fig. 1, the differential heat of solution of riboflavin at saturation comes to be 9,200 cal. per mole.

A. KALYANASUNDARAM

Indian Institute of Sugar Technology
Kanpur
June 2, 1949

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Printed by H. JACQUEMOTTE, S.J., at the Catholic Press, Ranchi, India
Issued by the Council of Scientific & Industrial Research, India. Editor : B. N. SASTRI, M.Sc.