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Journal of

# Scientific & Industrial Research



## IN THIS ISSUE :

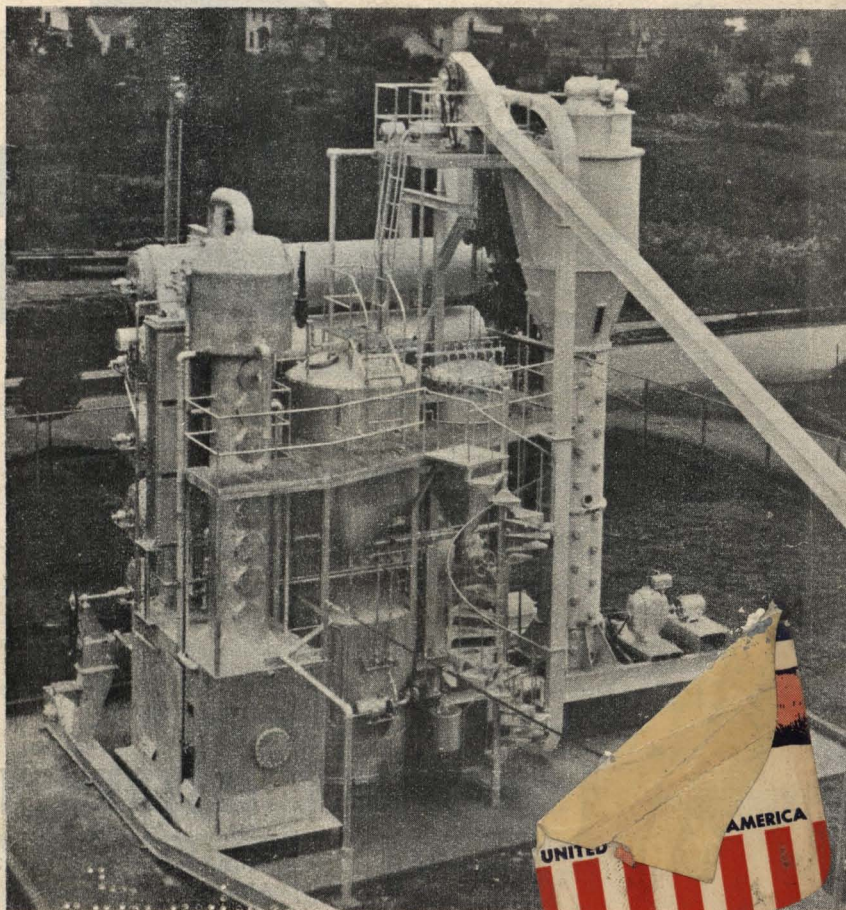
Defence Science Conference

Solvent Extraction of Vegetable Oils

Colouring Matter from Tambul Seeds

Properties of Acidic Oxycellulose

Chemistry of *Solanum torvum*



Vol. VIII

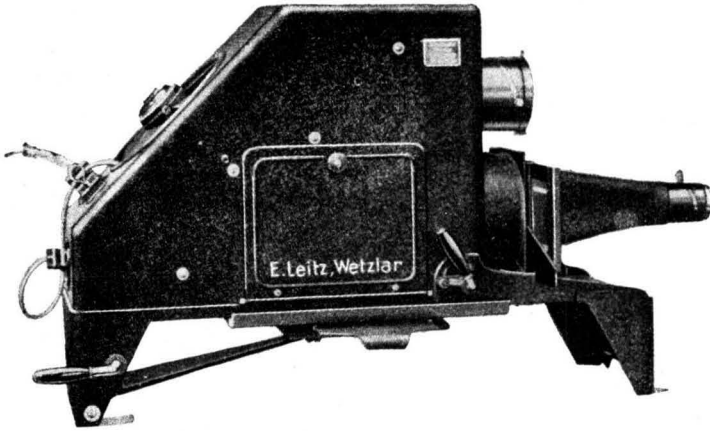
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Vol. VIII

JUNE 1949

No. 6

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## CONTENTS

Defence Science Conference	...	...	211
Third International Conference on Forestry & Timber Utilization for Asia & the Pacific	...	...	213
Characteristics of the Ionosphere over Calcutta (March 1949)	...	...	215
S. S. Baral, R. K. Mitra, D. C. Choudhury, (Miss) T. K. Pal & A. P. Mitra			
German Dyestuff Industry	...	...	217
K. Venkataraman			
A Method for the Preparation of Dioctyl Sebacate	...	...	231
J. P. Verma & Karimullah			
A Study of the Colouring Matter of <i>Tambul</i> Seeds	...	...	232
T. R. Seshadri			
Reviews	...	...	237
Developments in Vegetable Oil Extraction	...	...	241
M. N. Krishnamurti			
Non-Technical Notes			
RUBBER-LINED EQUIPMENT FOR CHEMICAL INDUSTRIES	...	...	243
Notes & News	...	...	245
Reports from States & Provinces	...	...	253
Indian Patents	...	...	254
For Contents to Section B, see page xxxiii.			
For Index to Advertisers, see page xv.			

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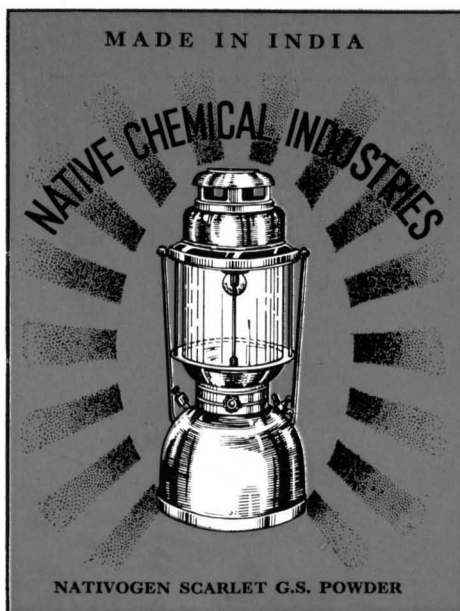
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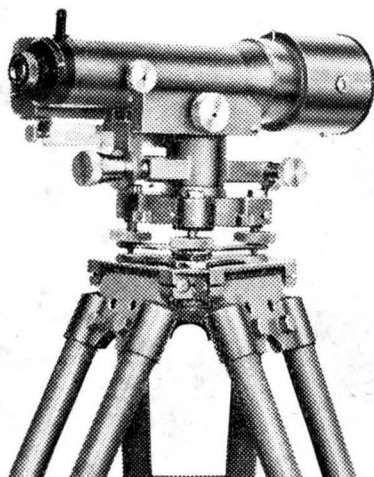
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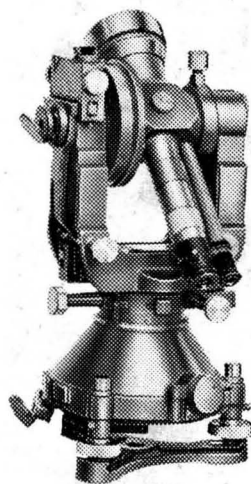
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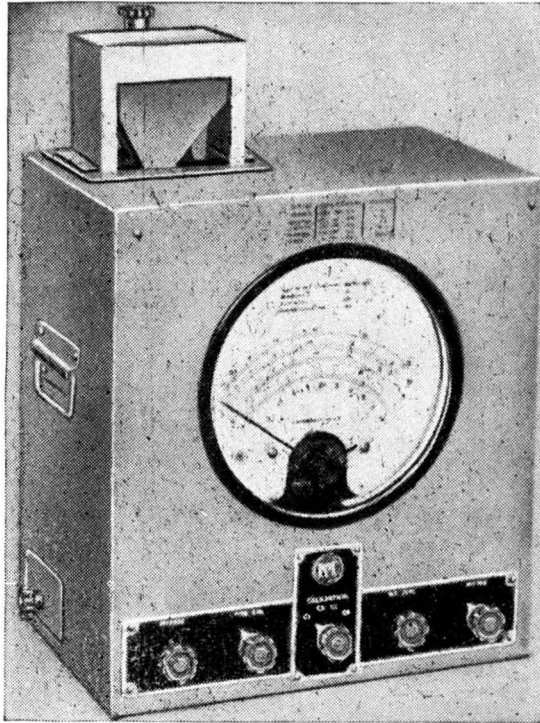
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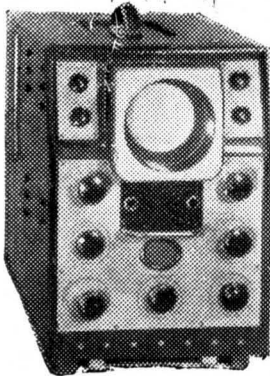
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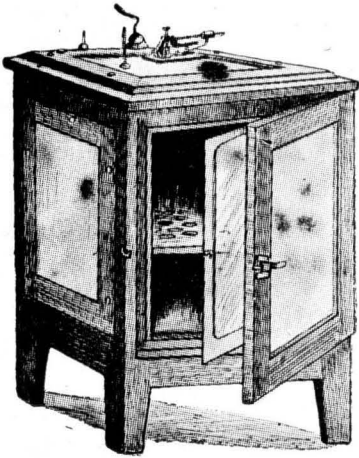
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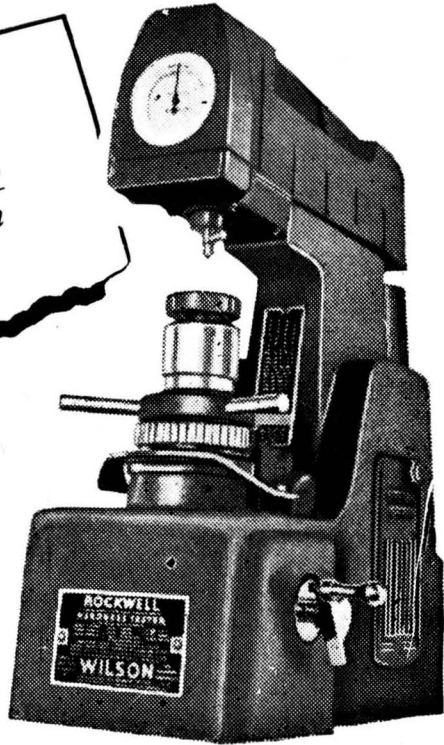
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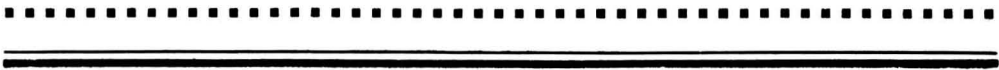
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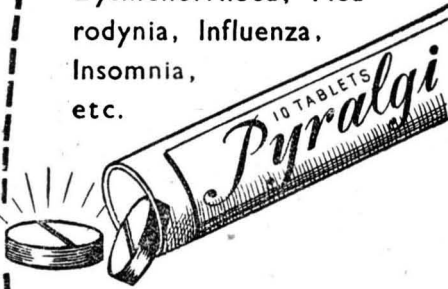


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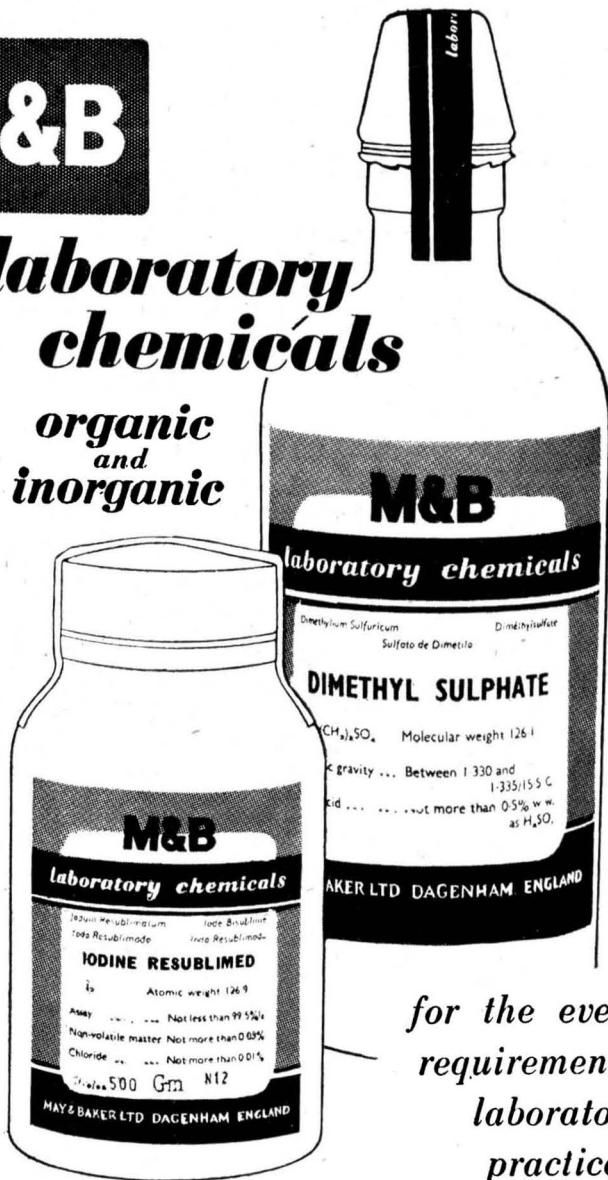
## INDEX TO ADVERTISERS

	PAGE
ALEMbic CHEMICAL WORKS Co. LTD., BARODA .. .. .	XIII
AMA LTD., BOMBAY .. .. .	IX
ASSOCIATED INSTRUMENT MANUFACTURERS (INDIA) LTD. .. .. .	XXIV
BENGAL CHEMICAL & PHARMACEUTICAL WORKS LTD., CALCUTTA .. .. .	IV
BENGAL WATERPROOF WORKS (1940) LTD., CALCUTTA .. .. .	XII
BOMBAY CHEMICALS LTD., BOMBAY .. .. .	IV
EASTERN CHEMICAL Co. (INDIA), BOMBAY .. .. .	XVIII
EASTERN ELECTRIC & ENGINEERING Co., BOMBAY .. .. .	XIV
FRANCIS KLEIN & Co. LTD., CALCUTTA .. .. .	XI
GANSONS LTD., BOMBAY .. .. .	XVII
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PHILIPS ELECTRICAL Co. (INDIA) LTD., CALCUTTA .. .. .	VIII
RAJ-DER-KAR & Co., BOMBAY .. .. .	VII
SCIENTIFIC APPARATUS & CHEMICAL WORKS LTD., AGRA .. .. .	XXV
SHREE INDUSTRIAL & COMMERCIAL SYNDICATE LTD., BOMBAY .. .. .	XVIII
THE SCIENTIFIC INSTRUMENT Co. LTD., CALCUTTA .. .. .	II
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ZILL & Co., BOMBAY .. .. .	XXVI

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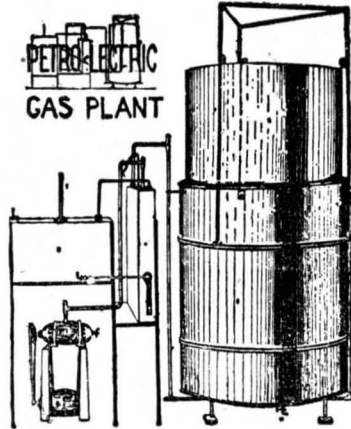
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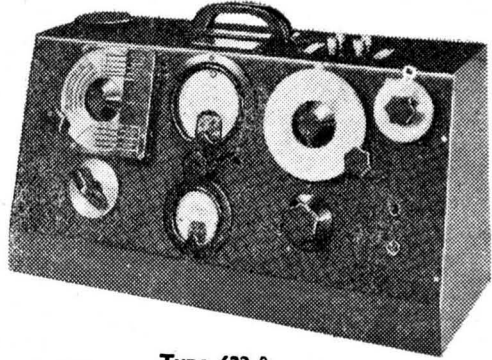
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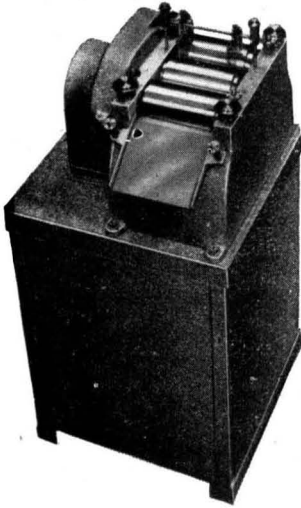
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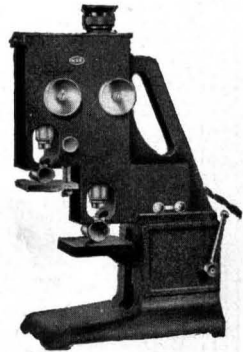
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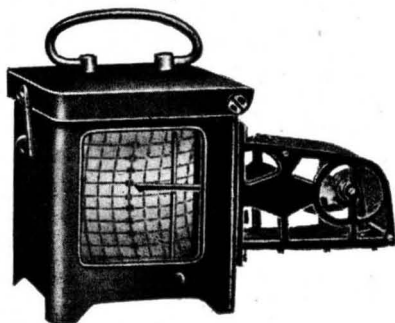
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## Defence Science Conference

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THE Defence Science Conference, organized by the Defence Ministry and inaugurated by Dr. S. S. Bhatnagar on April 20 at Delhi in the unavoidable absence of the Defence Minister, was the first of its kind to be held in India. It was intended to stimulate interest in defence science among Indian scientists, and to bring together research workers in defence establishments, universities and civilian research institutions so that problems of mutual interest may be discussed and ideas exchanged. Its success was largely due to the awareness on the part of scientists that co-operation with Government's plan for defence research would provide them opportunities not only to make their contributions to national security but also expand the field of their professional activities.

To appreciate the significance of the Conference, it would be useful to refer briefly to a few distinctive organizational features of scientific research in the Defence Department. Defence research has well-defined objectives and, therefore, lends itself to planning. It is essentially applied and developmental in character, and it caters to what Steelman\* describes as an "internal clientele". In other words, research in this field is intended primarily to meet the specific needs of the Department under whose auspices it is carried out. In this respect it differs from agricultural or medical research which caters to an "external clientele", that is, it meets the requirements, not of the sponsoring Department, but of the nation as a whole. If this feature of defence research is borne in mind, it will be clear that the agency which plans and guides research programmes should maintain intimate contact with the administration at the policy-making level, so that Service requirements may be precisely formulated and catered to.

The progress of applied or development work is dependent on knowledge revealed by basic or fundamental research, and significant results cannot be expected unless the frontiers of knowledge are deliberately and continuously widened. The Defence Department, keenly interested in the promotion of basic research, has to take steps to encourage such research activities in universities and civilian research centres where the "climate" for free inquiry and creative endeavour is favourable. In the United States of America, basic research in the Army is carried on largely outside the Department by contract, and applied and development work is the concern of the Technical Services and the Air Force. A similar distribution of research effort obtains in the United Kingdom. Thus, defence science is concerned with both basic and development research and the Defence Science Organization constitutes a bridge linking abstruse fundamental research at one end, and intensely specialized design, production, and proving work at the other. The Defence Department has a clear interest in the progress of research as a whole — in industry, the universities and in other Government Departments — and its manifest effort to enlist the understanding co-operation of the nations' scientists is necessitated both by enlightened self-interest and national security.

The need for sustained and adequate military research, and the need for increased emphasis on science in the training of officers for the Army, Navy and the Air Force have been widely recognized. Both these needs can be satisfied by securing a professional partnership between the officers of the Services and civilian scientists. Dr. Vannevar Bush, in his oft-quoted Report, *Science — the Endless Frontier*, points out how vital such partnership is for the promotion of national security, and stresses the need for a "permanent, independent, civilian-controlled

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\* *Science & Public Policy*, 1947, III, 40.

organization, having close liaison with the Army and Navy, but with funds drawn from the Congress and with the clear power to initiate military research which will supplement and strengthen that carried on directly under the control of the Army and the Navy". The value of such an organization was proved during the last war, and Dr. Vannevar Bush was called upon to direct the activities of the war-time Office of Scientific Research and Development. Civilian-controlled organizations were in operation in several Allied countries during the war, and the integration of scientific and military thought secured thereby greatly influenced the course of the war. The view expressed by Vannevar Bush has the authority of experience and achievement and has been widely accepted.

Defence research, in any real sense, has yet to be initiated in India. Useful experience gained during the last war is, however, available. Development work started during the emergency when communications with Europe became difficult was concerned mostly with modifications of existing equipment and weapons to render them suitable for service conditions in India. Research was initiated on a small scale, and laboratories were set up. The expansion of the Air Force and the Navy called for new facilities for designing and testing, and posed new problems for solution. The emergency organization was inadequate to meet the requirements; it was necessary to shape it towards more enduring ends. Three years ago, the Government of India invited Dr. Wansborough-Jones, Scientific Adviser to the Army Council in the United Kingdom, to advise them on the setting up of a Defence Science Organization for India. More recently, the Government sought the advice of Prof. P. M. S. Blackett and of Sir Ben Lockspeiser. Plans have been prepared and

finalized, and a Defence Science Organization is beginning to take shape. In formulating its programme so that it may serve national interests effectively, a scientific approach is a desideratum. Science is required not merely to solve problems that are seen to exist; it is even more required to discover problems and to assess the order of their importance. Planning and programming demand scientific forethought and wise counsel, and in a matter so vital as national security, the understanding co-operation and collective wisdom of military and civilian personnel are essential. The organization of defence science in India is rendered particularly difficult on account of the overall paucity of scientific workers and meagre budgetary allotments. As pointed out by the Scientific Adviser to the Defence Ministry during the Conference, there is but one scientist for every 100,000 of the population, and the total research expenditure is hardly one-fiftieth of one per cent of the national income! Even so, the prospects for scientific development are bright in view of the country's prodigious, but yet untapped, resources both in talent and wealth, and the concerted measures which the Government, fully alive to the benefits of science, are taking to augment the man-power and material requirements for scientific research. There is enthusiasm among scientists to co-operate with Government in their plans for national security, an enthusiasm which was manifested in a clear manner in the discussions and declarations during the 3-day Conference. By holding the Conference, the Defence Science Organization has not only assured itself of the co-operation of Indian scientists; it has also succeeded in encouraging those fruitful contacts outside the "official channels" which provide the surest means of raising the morale and productivity of working scientists.



## Third International Conference on Forestry & Timber Utilization for Asia & the Pacific

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**T**HE Third International Forestry and Timber Utilization Conference for Asia and the Pacific, convened by the Food and Agricultural Organization of the United Nations, was inaugurated by the Hon'ble Mr. Jairamdas Daulatram, Minister for Food and Agriculture, Government of India, on 28th March 1949 at the Cheluvamba Mansions, Mysore. Delegates from Burma, Ceylon, France, India, Malay, Netherlands, New Zealand, Pakistan, Philippines, Siam and the United Kingdom attended the Conference. Observers were present on behalf of Bhutan, Indonesia, Korea, Nepal, Portugal, *UNESCO* and *SCAP* (Japan). The Indian delegation was led by Sir T. Vijayaraghavacharya who was elected chairman of the Conference.

Mr. Norris E. Dodd, Director-General of the *FAO*, in his message, dwelt on the inter-dependence of the well-being of the land and the community, while M. Marcel Lelorp, Director of Forestry, *FAO*, explained the magnitude of the problems they had to face in Asia and the machinery needed to translate the recommendations of the Conference.

One immediate and concrete sequel to this Conference is the setting up of a regional forestry office at Bangkok with Dr. D. Y. Lin as its first Director. This office undertakes the collection and dissemination of information on all forestry matters between countries of Asia and the Pacific.

The general scheme of work for the Conference was on the same lines followed in the two earlier regional conferences to assess the forest assets and liabilities of each region, to estimate the gap between supply and demands for forest product, discuss remedies and to set up machinery to implement on a regional basis the recommendations of the Conference.

The Conference, after critical discussion, adopted resolutions on the following subjects: (1) Forest Resources; (2) Forest Policy; (3) Soil Conservation; (4) International Control in Erosion Control; (5) Forestry and Weather; (6) Control of Grazing; (7) Roving Agriculture; (8) Large-scale Afforestation

and Reforestation; (9) Domestic Needs; (10) Increased Production; (11) Balancing Supplies and Demand; (12) Distribution of Timber; (13) Standardization and Grading; (14) Forest Inventories; (15) Enumeration Methods; (16) Minor Forest Products; (17) Statistics; (18) Research; (19) Exchange of Information; (20) Dissemination of Information; (21) Training and Education; and (22) Conservation of Wild Life.

The recommendations of the Conference fall into two main categories, viz. the immediate needs and the long-term requirements. While the Conference took note of the urgent issues raised by the former category and made valuable recommendations to meet them, even more valuable was its contribution to the long-term rehabilitation and development of the forest resources of the region. So much so, the first resolution of the Conference deals with "the Importance of Forest Resources", which reads as follows: "The Conference, recognizing that forests, as renewable, are essential to a country's agricultural life, industrial development, economic stability and national security;

recognizing also, that forests and forest products, both major and minor, are essential to the life of the rural and urban population; calls the attention of the governments of all countries of the region to the need for dynamic policies for the conservation, development and utilization of forest resources according to the needs and possibilities of each country;

declares that any failure by governments to accord due recognition to the great value of forest resources will have disastrous consequences for the economy of the country, both rural and industrial;

urges governments to so manage their forests as to serve the rural and urban population and thus enlist the support of the masses for the implementation of a sound policy for the protection and development of forests;

recommends that the attention of the governments of the region be drawn to the desirability of giving a high priority in their national, financial and economic plans

to the claims of forestry and the improvement of harvesting facilities."

*Forestry Commission for Asia* — The Conference considered that the conservation of natural resources and the provision of domestic needs in fuel-wood, charcoal and timber were the two outstanding problems of immediate urgency in many countries of the region. Solutions for these problems had also long-term implications. Bold schemes of protection and reforestation of the head-waters of great rivers were needed as also projects to combat soil erosion, programmes of afforestation and measures to effect flood control. Only thus could production and agriculture be made secure, water supplies be safeguarded, and living conditions be ameliorated. The effort required for these purposes has surpassed purely local or national interests. An international approach was necessary. The Conference, therefore, recommended the establishment of a Forestry and Forest Products Commission for Asia and the Pacific consisting of accredited representatives of all countries in the region, which should meet as and when necessary to co-ordinate matters of common interest to the region; to examine particular problems in the fields of forestry and forest products, both of immediate urgency and long-term significance; and make recommendations to governments through the Council of *FAO*.

It recommended, further, that in order to organize the work of this Commission, the Director-General of *FAO* set up a forestry and forest products working group in the region, composed of staff members of the Organizations Division of Forestry and Forest Products.

Of the other 23 resolutions passed by the Conference, 3 are of special importance to India.

The Resolution III deals with soil erosion which is a menace to most countries, and recommends the setting up, in each country, of a central authority for the co-ordination in both planning and execution, of land utilization policies. In this country the area

under forest is well below the safe minimum of about 25 to 30 per cent of the land area and it would be a short-sighted policy indeed if our effort at self-sufficiency in food should make inroads into our already inadequate forest areas — losing sight of the basic fact that forestry and agriculture are two different methods of utilizing the common capital of both, viz. the soil.

Resolution XVIII makes recommendations on the important subject of forest research. Attention was drawn to the fact that the existing research institutes, though well organized and well equipped, were extremely insufficient to serve the needs of the region and inadequately distributed geographically and there was lack of co-ordination of the research work carried out by different countries. India has, in the Forest Research Institute, Dehra Dun, one of the important forest research centres in this region, but it is not sufficient to cover even the needs of India. India's offer to co-operate fully by placing her available research facilities and experience at the disposal of the member nations at the Conference was very much appreciated.

The Indian delegation made positive contributions to the deliberations of the Conference on the subject of forest education which forms the subject-matter of Resolution XXI and which has a direct bearing on conditions prevailing in India at present. The facilities obtaining in India for forest education are good but grossly inadequate. The imperative need for trained foresters, forest training and research institutions and exchange of students and technicians was stressed. The type of specialist the country needs today is one with a grounding in the fundamentals of forestry and who is called upon not to manage forests but to run forest industries. The facilities for such specialized training are conspicuous by their almost total absence in India. One of the means suggested for achieving this is to provide a place for elementary forestry as a subject of liberal education in the curricula of universities.

# Characteristics of the Ionosphere over Calcutta (March 1949)

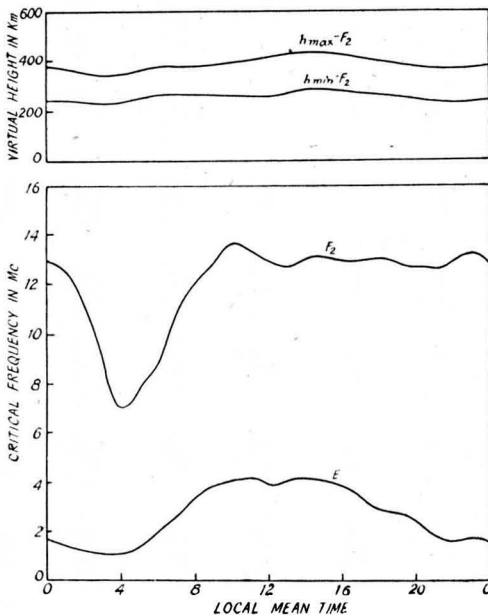
S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY,  
(Miss) T. K. PAL & A. P. MITRA

*Wireless Laboratory, University College of Science, Calcutta*

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

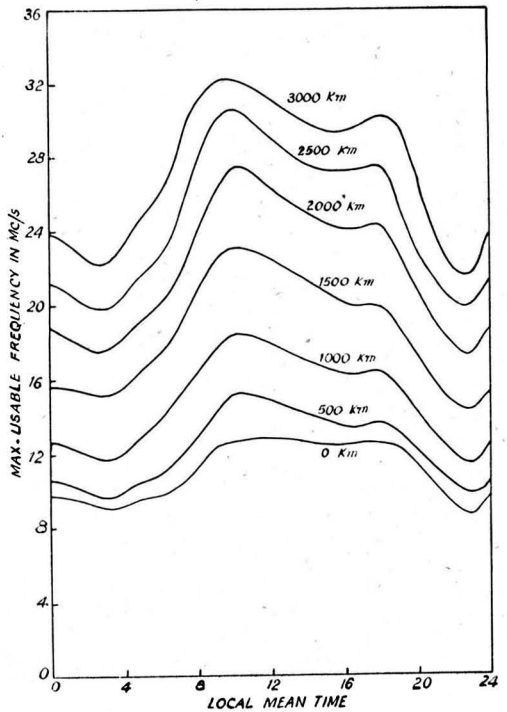
Fig. 1 represents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequency of the E layer in graphical form during the month of March 1949. The figure represents the mean of both normal and abnormal values. Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmissions during June 1949 by reflection at the F region over Calcutta, based on the normal values of the data for March 1949. Table I gives the list of occasions when E region

ionization was found to be abnormal and the corresponding penetration frequencies and heights.



(5 HOURS 54 MINUTES AHEAD OF G.M.T.)

FIG. 1 — MARCH 1949.



AT POINT OF REFLECTION.

FIG. 2 — PREDICTED M.U.F. FOR TRANSMISSION VIA  $F_2$  LAYER, JUNE 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^o E_s$ Mc.	$h E_s$ Km.
March 1949	1	01.00	2.50	95
		10.00	4.70	120
March 1949	2	09.00	4.10	120
		10.00	5.00	120
		11.00	5.70	135
		17.00	3.45	120
		18.00	3.30	120

TABLE I—contd.

MONTH & YEAR	DATE	HOUR	$f_{E_s}^{\circ}$ Mc.	$h_{E_s}$ Km.
March 1940	3	18.00	5.00	135
		19.00	4.25	120
		20.00	3.20	120
		22.00	3.00	105
		23.00	2.70	105
	4	00.00	3.30	105
		01.00	3.20	105
		02.00	3.00	95
		03.00	3.00	95
		05.00	2.85	95
		16.00	4.60	120
		17.00	4.25	120
		18.00	4.25	120
	7	19.00	3.20	120
		20.00	3.50	120
22.00		3.00	120	
23.00		2.70	120	
8	00.00	3.20	95	
	10	19.00	3.50	120
	23.00	3.15	120	
11	00.00	3.50	120	
	01.00	2.65	95	
	02.00	2.50	95	
	03.00	2.55	95	
	04.00	2.45	95	
15	22.00	4.25	120	
	23.00	4.00	105	

TABLE I—contd.

MONTH & YEAR	DATE	HOUR	$f_{E_s}^{\circ}$ Mc.	$h_{E_s}$ Km.
March 1940	16	00.00	3.00	95
		01.00	2.90	95
		03.00	2.50	95
		07.00	3.55	120
		22	22.00	2.70
		23.00	2.40	105
	24	18.00	3.20	120
		19.00	3.20	120
		20.00	2.80	105
		22.00	2.60	105
		23.00	2.50	105
	25	22.00	2.65	105
		23.00	2.45	105
	26	00.00	2.20	95
		01.00	2.00	95
28	18.00	3.15	120	
	19.00	2.85	120	
	20.00	2.50	105	
	23.00	2.85	105	
20	20.00	2.70	105	
	21.00	2.50	105	
	22.00	3.00	105	
	23.00	3.00	105	

## Patents & Designs Bill in the U.K.

THE BRITISH GOVERNMENT HAS JUST PUBLISHED the Patents and Designs Bill, recently introduced in the House of Lords. The Bill seeks to improve the patent law and thereby stimulate scientific and technical development in industry. It proposes to implement a large number of recommendations made by the expert committee, known as the Swan Committee, set up five years ago.

An important feature of the Bill is the provision of remedies for the abuse of patent rights or insufficient use of patented inventions. These provisions are directed not only against notorious types of abuse such as deliberate suppression of patented inventions but also cover cases where an invention is not being worked to the fullest possible extent, or where an export market is not being supplied or the working of another patent is hindered by the refusal of the

patentee to grant a licence on reasonable terms. Provision is also made for action by a Government Department in the public interest. There is no doubt that the Patents Enquiry Committee set up by the Government of India will carefully consider the feasibility of incorporating similar provisions in the Indian Patent Law.

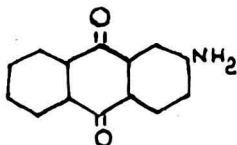
The Bill embodies numerous other proposals of reform among which, to mention only a few, may be noted the extension of the grounds of opposition to check the grant of worthless patents, direction of all appeals from the Controller's decision to the Patent Appeals Tribunal, right of an exclusive licensee to take infringement proceedings in his own name and the manufacturer's right to obtain a declaratory judgement to resolve doubts whether a particular manufacture which he proposes to undertake would infringe a specified patent.

# German Dyestuff Industry\*

K. VENKATARAMAN

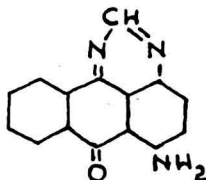
(Continued from May issue)

**APPENDIX 67**  
2-AMINOANTHRAQUINONE



In an autoclave (9 cu. m.) are introduced 2-chloroanthraquinone (1,400 kg.), ammonia 25 per cent (4,400 kg.), water (1,100 kg.) and arsenious acid 80 per cent (64 kg.). The autoclave is heated to 207°C. and a pressure of 45 atm. It is maintained at this temperature for 24 hr. At the end of the reaction the outlet valve is opened and ammonia absorbed in an absorbing vessel in the course of 8 hr. The gas is first passed through a Spitz cylinder, then 2 worm coolers and finally into an absorber in which water (500 l.) is placed. After the ammonia gas has been let off from the autoclave, it is filled with water (2 cu. m.), and the contents filtered. The filter cake is washed with warm water until there is no ammonium chloride in the filtrate. The filter cake is then dried when the crude amine (1,260 kg.) of purity 88 per cent is obtained. The yield is 87 per cent of theory.

**APPENDIX 68**  
4-AMINO-1:9-ANTHRAPYRIMIDINE

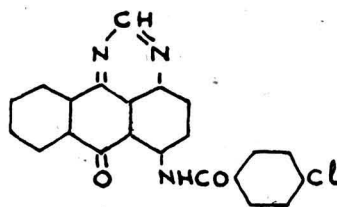


1:4-Diaminoanthraquinone, sublimed (0.5 kg.), ammonia 30 per cent (1.0 kg.),

formaldehyde 30 per cent (1.0 kg.) and Ludigol (0.375 kg.) are heated in an iron autoclave for 15 hr. at 88°-92°C. (pressure about 2 atm.). After cooling, the product is filtered, washed neutral with water and dried. Yield, 0.51 kg. of crude product.

The crude product is purified by dissolving in sulphuric acid, 96 per cent (5.0 kg.). The sulphate is then precipitated with the addition of water (2.5 kg.) in a dropwise manner at 100°C. After cooling, small needles of crystalline sulphate are filtered off and washed with sulphuric acid 58 per cent (2.0 kg.). The sulphate is pasted with hot water and decomposed with ammonia, filtered and washed neutral with hot water. Yield 0.42 kg. of pyrimidine = 84 per cent of 1:4-diaminoanthraquinone used or 81 per cent of theory.

**APPENDIX 69**  
INDANTHRENE YELLOW 7GK POWDER  
(CRUDE)



The above dyestuff is prepared by benzoylation of Pyrimidine AFS with *p*-chlorobenzoyl chloride in nitrobenzene in presence of acid-binding agents like pyridine.

In an enamelled vessel provided with an agitator and an oil bath are introduced Pyrimidine AFS (60 kg.), nitrobenzene (300 kg.), pyridine (30 kg.) and *p*-chlorobenzoyl chloride (66 kg.). The contents are heated to 125°-130°C. and maintained at this temperature for 3 hr. The

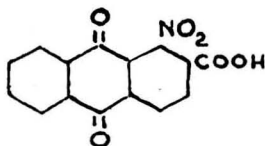
\* The documents mentioned in the report are available for consultation at Technical Information and Document Unit, German Division, Board of Trade, 40 Cadogan Square, London, S.W. 1.

benzoylation sets in at about 120°C. and finishes as a rule in 90 min. The contents of the vessel are maintained for a further 2 hr. at 125°-130°C. The vessel is allowed to cool without stirring and filtered. The filter cake is washed with nitrobenzene (200 kg.) till the filtrate is yellowish. The filter-pressed material is then freed from nitrobenzene and dried. From 60 kg. Pyrimidine, 90 kg. Indanthrene Yellow 7GK Powder (crude) are obtained, the yield being 91.2 per cent of theory.

For obtaining the dyestuff in paste form, it is dissolved in sulphuric acid, 96 per cent, and reprecipitated.

#### APPENDIX 72

##### 1-NITROANTHRAQUINONE-2-CARBOXYLIC ACID



In an acid-resistant 22 cu. m. vessel with lead stirrer is introduced a mixture of sulphuric acid 96 per cent (2,200 kg.) and sulphuric acid 58°Bé (10,800 kg.). To this is added 1-nitro-2-methylanthraquinone calculated at 100 per cent (640 kg.) in 2 lots. A warm solution of sodium dichromate (1,280 kg.) in water (640 kg.) at 50°C. is run in during 6 hr. The temperature is now raised in 6 hr. to 65°C. and the mixture heated at this temperature till no more of the carboxylic acid is formed (24 hr.). At the end of this period, 10-15 per cent of unconverted nitromethylantraquinone is present. Further oxidation serves no useful purpose as it takes a disproportionately long time. The reaction mixture is diluted with ice and water, cooled to 25°-30°C. and filter pressed. The filter cake is washed neutral to Congo paper with cold water.

The crude product is stirred in an alkali-resistant vessel of capacity 20 cu. m., provided with a stirrer, with 15 cu. m. water at 80°C. At this temperature caustic alkali solution 40°Bé (200-250 kg.) diluted with the same quantity of water is run in. The solution is tested with "Triacen" paper till it does not become red. A further quantity of caustic alkali (5-10 kg.) may be necessary. The mixture is stirred for 1 hr. at 80°C., filtered hot and washed with

warm water till the wash water does not contain the sodium salt of the desired acid. The filtrate and wash water are collected in an acid-resistant vessel of capacity 22 cu. m., and acidified with sulphuric acid, 96 per cent, (80 kg.) till the solution is acid to Congo paper. After brief stirring, the precipitated nitrocarboxylic acid is filtered, washed neutral with cold water and dried. Yield 560 kg. = 90 per cent of theory.

#### APPENDIX 74

##### INDANTHRENE BLUE RS

In a 5 cu. m. cast-iron vessel with stirrer are introduced 1,000 kg. caustic soda and caustic potash mixture of composition 67-68 per cent potassium hydroxide and 27-28 per cent sodium hydroxide and 200 kg. water-free sodium acetate. Then, at 180°C., in the course of 20 min., are introduced 500 kg.  $\beta$ -aminoanthraquinone (86-87 per cent) (= 436 kg. of  $\beta$ -aminoanthraquinone calculated at 100 per cent) from an automatic balance. After the addition is over a mixture of 60 kg. sodium nitrate, 40 kg. potassium hydroxide and 20 kg. sodium hydroxide, uniformly powdered, is introduced in the course of 2½ hr. The temperature of the melt should not rise above 200°-225°C. It is then stirred for half an hour and sucked off into a cylindrical vessel, 16 cu. m. capacity, containing 11 cu. m. of water at 40°C. The Indanthrene fusion vessel is then rinsed with 2 cu. m. of water. The temperature in the vessel rises to 60°-65°C., and it is cooled to 45°-48°C. in the course of 2 hr. by jacket cooling. It is then vatted by the addition of 750 kg. sodium hydrosulphite (14-15 per cent) under stirring. After allowing to stand for 2 hr., it is filtered on a 36-plate filter press. It is then washed with 1,600 l. water at 25°-30°C. and 150 kg. sodium hydrosulphite (14-15 per cent) and finally washed with water until the filtrate is not very blue but pale green. The filter cake is then blown out with air.

The filter cake is then introduced into a rubber-lined 8 cu. m. oxidation vessel provided with rapid stirring and containing about 1 cu. m. water and 20 kg. sodium hydroxide, and oxidized at 60°C. for 1½-2 hr. 2 lots (total about 490 kg. Indanthrene Blue RS calculated 100 per cent) are then combined and sucked off into a lead-lined cast-iron vessel of 24 cu. m. capacity or in a 14 cu. m. lead vessel, both provided with

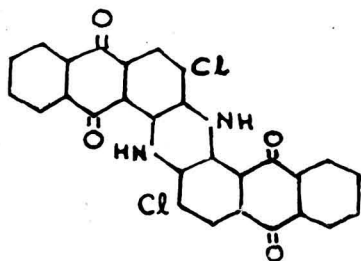
stirring; for each double lot are placed in the vessel 4 cu. m. water at 60°C. and 160 kg. sulphuric acid 66°Bé. The slightly acidic dyestuff is then filtered off in a wooden press, washed neutral and dried (Indanthrene Blue RS Powder crude), or filtered in a rubber-lined Imperial filter and washed neutral.

The press cake thus obtained is reckoned as Indanthrene Blue RS, moist, calculated 100 per cent. Yield per lot 245 kg. Indanthrene Blue RS moist, calculated 100 per cent = 56 per cent of addition = 56.5 per cent of theory.

For the production of Indanthrene Blue RSN, the above product is pasted with 10 per cent Tamol solution and dried in 6-7 hr. The dried product is then finely powdered in a Raymond mill and mixed in a mixer with sodium carbonate and disodium phosphate and marketed as Indanthrene Blue RSN.

#### APPENDIX 74a

##### INDANTHRENE BLUE BC



Indanthrene Blue RS is chlorinated in sulphuric acid (92 per cent), containing manganese dioxide, to the dichloro derivative of the azine. The filter-pressed material is then dissolved in oleum and reduced to dichloroindanthrone by the addition of anthracene disulphonic acid.

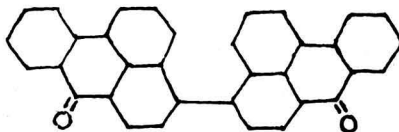
In an iron vessel of capacity 3.2 cu. m. fitted with a rapidly revolving stirrer are introduced sulphuric acid (96 per cent; 3,500 kg.) and Indanthrene Blue RS powder calculated at 100 per cent (350 kg.), and stirred till a complete solution is obtained. Water (175 l.) is now added in a thin stream, when the temperature rises to 50°-55°C. Finely powdered manganese dioxide (about 90 per cent purity; 17.5 kg.) is then added and the vessel closed. At 50°-55°C. chlorine gas (210-230 kg.) is led in at an hourly rate of 15 kg. When the chlorine content of the product reaches to 13.7 per cent, the product is filter pressed

and stirred with sulphuric acid 58°Bé (500 kg.) and diluted with water (400 l.). The sulphuric acid percentage of the mixture should not now go below 55°Bé. The product after dilution has a strength of 58°Bé. After standing for 40 hr., the product is filter pressed and filter cake washed with sulphuric acid 58°Bé (1,000 kg.).

The washed filter cake is now dissolved in a mixture of oleum (24 per cent; 2,000 kg.) and sulphuric acid (96 per cent; 500 kg.), the temperature not being allowed to rise above 350°C. Anthracene disulphonic acid (50 kg. calculated at 100 per cent) is now added and the temperature brought to 60°C. The mixture is kept till a test shows that the product is reduced; time taken is 10 hr. After cooling to 30°C., oleum (24 per cent; 300 kg.) is added and the solution precipitated under stirring in a 15 cu. m. vessel with warm water at 50°-60°C. The dyestuff is filtered and washed neutral with warm water. About 370 kg. Indanthrene Blue BC calculated at 100 per cent is obtained.

#### APPENDIX 75a

##### 4: 4'-DIBENZANTHRONYL

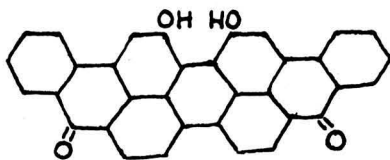


In a 3 cu. m. iron vessel provided with a stirrer are introduced pure isobutanol (385 kg. = 450 l.), powdered caustic potash (90 per cent; 500 kg.) and water-free sodium acetate (60 kg.). It is heated by closed steam to 140°-145°C. in about 2 hr. when isobutanol distils over and is returned from the condenser. It is stirred at this temperature for 1 hr. and cooled to about 100°C., when finely powdered sublimed benzanthrone (200 kg.) is introduced in the course of 1 hr. By the addition of benzanthrone the reaction mixture becomes rather viscous, so that the number of revolutions of the stirrer is reduced to 15-20 r.p.m. (Forset-Enor device). The reaction is exothermic, so that towards the end of the addition of benzanthrone the inner temperature is 112°C. It is stirred at this temperature for 1 hr. Then isobutanol (100 kg.) is added into the thick reaction mixture whereby the inner temperature falls to 105°C., and the fusion mixture becomes thinner, so that the

reaction mass can be stirred at the full speed of the stirrer. The reaction mixture is now cooled to 90°C. by water, cold water (1,000 l.) added, and by further cooling the temperature brought to 25°-30°C. The reaction mixture is then stirred for 30 min., and the lower 30 per cent aqueous potassium hydroxide layer is run off. In the vessel there is a deep-green paste which is a suspension of leucodibenzanthronyl in isobutanol. It is diluted with 400 l. water, and a solution of 80 kg. bleaching liquor in 600 l. water is run in, stirred for 30 min., and an azeotropic mixture of isobutanol with water is distilled over (inner temperature 90°-92°C.). This azeotropic mixture separates into 2 layers, the upper layer containing 83 per cent isobutanol and the lower layer 5 per cent isobutanol. The lower watery layer is run into the vessel till the inner temperature rises to 101°-102°C. The light-green mass is sucked off into a 22 cu. m. diluting vessel in which are introduced 2 cu. m. warm water. The vessel is rinsed with warm water and the diluting vessel filled with water to 8 cu. m., heated with open steam to 90°-95°C. and stirred at this temperature for 1 hr. It is then filtered through an iron filter press and washed with warm water. The pressed cake is dried under vacuum in an iron 3 cu. m. "Venuleth". Yield of dibenzanthronyl (82 per cent) is 200 kg. which is 82.3 per cent of theory.

#### APPENDIX 75b

##### 2:2-DIHYDROXYDIBENZANTHRONE



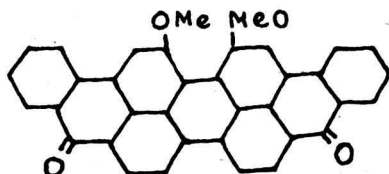
In a 3.2 cu. m. iron vessel provided with a stirrer are introduced 96 per cent sulphuric acid (2,000 kg.), 75 per cent sulphuric acid (2,500 kg.) and 4:4'-dibenzanthronyl 100 per cent (155 kg.). The mixture is stirred till the whole of the dibenzanthronyl is dissolved. After cooling to 15°C., pyrolusite (192 kg.) is added in 2-4 hr. in such a manner that two-thirds of it is added at 15°-25°C. and a third at 25°-35°C. through a 5 mm. mesh sieve. It is kept at 35°C. for 2 hr. and then filtered through an iron press.

The pressed cake (dihydroxydibenzanthrone and manganese sulphate plus

matrix) is stirred at ordinary temperature in an iron stirring vessel of 3.2 cu. m. capacity with monohydrate (3,000 kg.), till the dihydroxydibenzanthrone goes into solution. It is filtered through 2 resistant, revolving filters, each of 7 cu. m. capacity, and washed with 96 per cent sulphuric acid (3,000 kg.). The filtrate is diluted with water to 50° Bé in a lead diluting vessel of capacity 10 cu. m., cooled to 50°C., and filtered. The pressed cake in 3 portions is stirred in a resistant stirring vessel of 24 cu. m. capacity with water (15 cu. m.), and bisulphite solution 38° Bé (1,200 kg.) added. The mixture is boiled with open steam for 4 hr., filtered, washed neutral, and dried under vacuum at 130°C. Yield of dihydroxydibenzanthrone 100 per cent is 161 kg. (98 per cent of theory).

#### APPENDIX 75c

##### INDANTHRENE BRILLIANT GREEN FFB POWDER (CRUDE)



In an enamelled pressure vessel of capacity 3 cu. m. provided with a stirrer and an oil bath for heating is introduced trichlorobenzene (1,800 kg.). From another vessel provided with an agitator is introduced a suspension of caustic potash (200 kg.; dry content 99 per cent) in trichlorobenzene (250 kg.). The contents of the vessel are now heated to 180°-190°C. At this temperature is introduced from another vessel a suspension of dihydroxydibenzanthrone (150 kg.; calc. 100 per cent) in trichlorobenzene (300 kg.).

The vessel is now heated to boiling and maintained at this temperature for 1½-2 hr., when a mixture of trichlorobenzene and water (120-150 kg.) distils over. After cooling to 206°-208°C., methyl benzenesulphonate (300 kg.) is added in about 3 hr. in small lots. The contents of the vessel are now brought to the boil and maintained at this temperature for 3 hr.

After cooling to about 180°C. the contents of the vessel are filtered off and the filter cake is washed with hot trichlorobenzene (1,000 kg.) at 180°C. The filter cake is



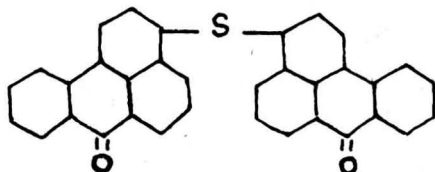
freed from trichlorobenzene and dried. Indanthrene Brilliant Green FFB Powder (crude) obtained is 141 kg., which is 94 per cent on the dihydroxydibenzanthrone used.

The dye (300 kg.) is made into paste form by dissolving in sulphuric acid (90 per cent; 4,500 kg.), immersing in cold water, filtering and washing neutral.

Trichlorobenzene used in the above reaction is recovered, about 100 kg. being lost in each batch.

## APPENDIX 76

## DIBENZANTHRONYL SULPHIDE



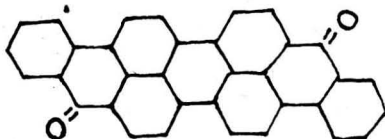
In a wrought iron autoclave of capacity 4 cu. m. for 15 atm. pressure with an elliptical iron stirrer and steam jacket, 1,420 kg. sodium sulphide solution 15 per cent (= 213 kg. sodium sulphide calculated 100 per cent) and finely powdered sulphur (45 kg.) are heated at 80°C. under stirring for 30 min. to bring the sulphur into solution, then cooled to about 30°C., and the operation carried on without stirring.

About 1,500-1,800 kg. bromobenzanthrone press cake (= 800 kg. bromobenzanthrone calculated 100 per cent) are added, while a weak current of nitrogen is passed through. Now the autoclave is closed and to remove the air nitrogen at 1.5 atm. pressure is passed in and again released. Then the apparatus is filled with warm water up to 3.7 cu. m., closed again and the air once again displaced by introducing nitrogen. The mass is now heated to 135°C., whereby a pressure of 3.5-4.5 atm. is developed and stirred at this temperature for 40-50 hr. After cooling to 50°C., the mass is pressed into a wrought iron 14 cu. m. capacity stirring apparatus provided with "Balcken" stirrer, containing 2 cu. m. warm water. The diluted content is filled into 12 cu. m. with washing water. After the addition of 70 kg. caustic soda 40°Bé the product is filtered in an iron filter press, and washed to neutrality with warm water (duration of washing is about 12 hr.). The filtrate and the washing flow into a vat containing waste sulphuric acid. The pressed mass

is dried in a vacuum drying cupboard with low pressure steam. Yield, 648 kg. of dibenzanthronyl sulphide dried = 102.2 per cent of theory.

## APPENDIX 77

## ISODIBENZANTHRONE



In a 3 cu. m. iron stirring vessel of capacity 3 cu. m. provided with a steam jacket are introduced isobutanol (600 kg. = 750 l.) and powdered caustic potash (90 per cent; 300 kg.). These are heated to boiling and maintained under reflux for 1 hr. (inner temperature about 140°C.). After cooling to about 70°C., finely powdered dibenzanthronyl sulphide (200 kg.) is introduced under stirring. The reaction mixture which is now at 60°C. is heated to 126°-128°C. whereby the alcohol begins to reflux. After about 2 hr. the reaction is complete.

A fusion sample diluted with water and vatted with hydrosulphite and alkali gives only a little dark filter residue. A yellow filter residue, which gives a blue solution with concentrated sulphuric acid and likewise a yellow outflow of filtrate from the filter, shows the presence of dibenzanthronyl sulphide.

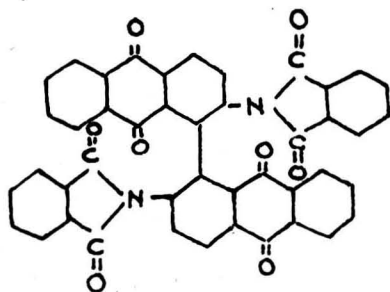
When the correct test is obtained, pure isobutanol (100 l.) is pumped into the melt, cooled to 100°C., and water or water separated during isobutanol recovery (100 l.) added, whereby the temperature falls to 60°C. Then an azeotropic mixture of isobutanol and water is distilled over by heating the mixture to 90°-92°C., the lower watery layer of dilute isobutanol (5 per cent) being run back till the inner temperature is 101°-102°C. The dark-violet melt is introduced into a rubber-lined 14 cu. m. diluting vessel in which 8 cu. m. of water are placed, any substance remaining in the vessel being rinsed with water into the diluting vessel. This is heated with open steam to 95°-100°C., maintained at this temperature for 1 hr., and air blown through it without heating for 8 hr. The oxidation is complete when a sample on filtration does not give a red-violet filtrate. By the addition of cold water the temperature is

brought to 60°C., filtered through an iron press and washed well with warm water. The filter-pressed material is dried in an iron "Venuleth" under vacuum. Yield about 187 kg. of about 80 per cent isoviolanthrone (80.6 per cent of theory).

The watery isobutanol containing about 18 per cent by weight of water is recovered in a fractionating column. About 6 per cent of isobutanol is lost in the process.

#### APPENDIX 79

##### DIANTHRAQUINONYL PHTHALIDE POWDER (CRUDE)



It is prepared in 2 stages. In the first stage 1-chloro-2-aminoanthraquinone is condensed with phthalic anhydride in presence of ferric chloride, when water is split off, and 1-chloro-2-phthalimidoanthraquinone is formed. In the second stage 2 molecules of 1-chloro-2-phthalimidoanthraquinone are linked together by the action of metallic copper.

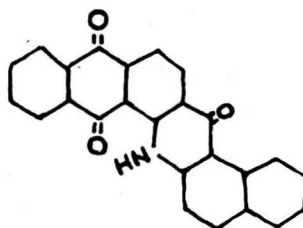
*Details of Process: First Stage* — In an iron 1.5 cu. m. pressure kettle, provided with stirrer and oil bath, is introduced first 170 kg. trichlorobenzene and, under stirring, 233 kg. 1-chloro-2-aminoanthraquinone 90 per cent (200 kg. at 100 per cent), followed by 132 kg. distilled phthalic anhydride and 4.4 kg. sublimed ferric chloride. The kettle contents are heated for 5-6 hr. at 215°-220°C. inner temperature, and for 1 hr. at 220°-230°C., whereby water and trichlorobenzene are distilled off. To facilitate the removal of water, a feeble stream of nitrogen is sucked through the kettle. After the attainment of 220°C. inner temperature, a minimum of 90-100 kg. of the mixture should be distilled (controlled through weighing). The melt is held at 230°C. for 5 hr. and cooled down to 100°C.

*Second Stage* — The melt is diluted with 300 kg. trichlorobenzene and heated again to boiling temperature (217°-220°C.) and

held for 3 hr. at the boil, during which a slow stream of nitrogen is maintained through the kettle. Water distils over mixed with trichlorobenzene such that in the stated time 100 kg. mixture are collected. The kettle is cooled down to 200°C. and by hand within 1 hr. are introduced 88 kg. copper powder. The mixture is heated to boiling temperature and maintained for 4 hr. Then the hot melt is diluted with 600 kg. trichlorobenzene, the kettle cooled to 130°C., and the product filtered in a closed filter press. The filter cake is washed with 700 kg. trichlorobenzene, and then dried in the Venuleth. Yield, about 459 kg. dianthraquinonylphthalide powder crude 48 per cent = 220 kg. calculated 100 per cent.

#### APPENDIX 82

##### INDANTHRENE RED RK POWDER (CRUDE)



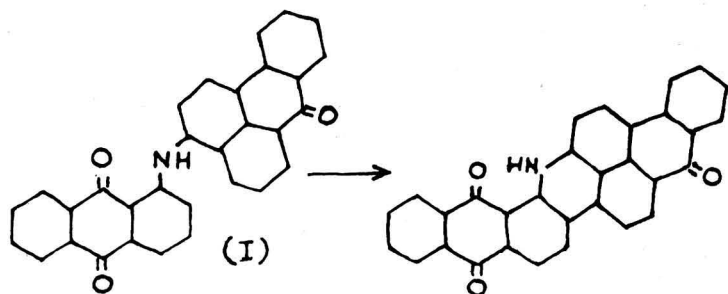
1-Nitroanthraquinone-2-carboxylic acid is heated in *o*-dichlorobenzene with  $\beta$ -naphthylamine in presence of arsenic trichloride to give 1- $\beta$ -naphthylaminoanthraquinone-2-carboxylic acid, and this is cyclized by heating to boiling.

In an enamelled vessel of capacity 3 cu. m. fitted with a stirrer and an oil bath are introduced *o*-dichlorobenzene (3,000 kg.), nitrocarboxylic acid (calc. 100 per cent; 150 kg.),  $\beta$ -naphthylamine (162 kg.) and arsenic trichloride (33 kg.). Under stirring the temperature is raised to boiling in about 4 hr. (oil bath temperature about 235°C.). After about 3 hr., when the inner temperature is 155°C., the distillate is collected in a vessel of capacity 3 cu. m. in which a solution of caustic soda (20 kg.) in water (800 l.) is placed. When the inner temperature reaches 181°C., about 500 kg. of distillate is collected. The reaction mixture is now kept vigorously boiling (inner temperature 184°C.) for 3 hr., in which period for every 30 min. about 300 kg. of distillate are collected. The total distillate collected in 6 hr. is 1,800 kg. The reaction mixture

is then maintained for a further 4 hr. under slow boiling so that a very small quantity of distillate passes over (oil bath temperature about 205°-210°C., inner temperature 183°C.). The contents are now allowed to cool without stirring, blown into a filter press, and the filter cake is washed with *o*-dichlorobenzene (1,500 kg.). The filter cake is then freed from *o*-dichlorobenzene with steam, and dried.

The solvent is recovered from the distillate by separating from the aqueous sodium arsenate solution, and distilled in a vacuum pan with addition of soda (35 kg.). The dichlorobenzene lye from the filter press is treated with a third of its weight of concentrated sulphuric acid, stirred, and then with the addition of soda (12 kg.) distilled in a vacuum pan. Yield 151.5 kg. of Indanthrene RK Powder Crude calculated 100 per cent. The dyestuff is converted into a paste by dissolving in sulphuric acid (96 per cent), and precipitating with water.

## APPENDIX 83

INDANTHRENE OLIVE GREEN B  
(PASTE)

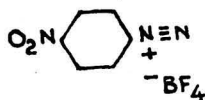
3-Benzanthronyl-1-anthraquinonylamine (I) obtained by the condensation of 3-bromobenzanthrone with  $\alpha$ -aminoanthraquinone is cyclized in isobutanol potash to give the above dyestuff.

In a vessel of capacity 3 cu. m. provided with a stirrer and a steam jacket are introduced isobutyl alcohol pure (1,000 kg.) and caustic potash 90 per cent (150 kg.). By heating under stirring the temperature of the mixture is raised to boiling, and powdered (I) (200 kg.) is added together with isobutanol 200 kg. in about 30 min. In about an hour's time the reaction sets in which is shown by the thickening of the mixture. The contents of the vessel are

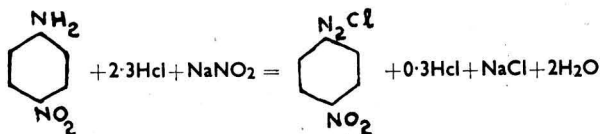
heated for 1 hr. more and then in 2 hr. isobutanol is distilled off. Cold water (700 l.) is now added and the inner temperature brought to 80°C. Isobutanol is now steam distilled for about 6 hr. The reaction mixture is then diluted with water, blown out with air, filtered at 60°C. and washed acid free. Yield of Olive Green B 100 per cent is 200 kg. About 85 per cent of isobutanol used is recovered.

## APPENDIX 91

## NITRAZOL CF EXTRA (PNA-OS = BOROFLUORIDE)

*p*-NITROPHENYLDIAZONIUM BOROFUORIDE

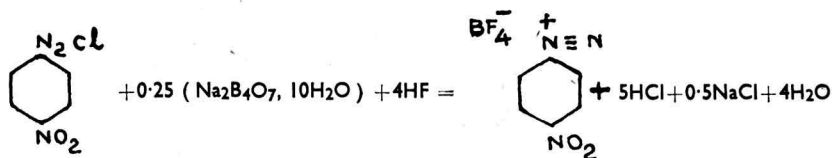
## Diazotization —



In a small diazotization vessel are introduced 32.5 l. water, 35 kg. ice and 203 kg. hydrochloric acid 30 per cent (= 60.8 kg. 100 per cent). To this, 22.5 per cent hydrochloric acid is added in about 3 hr., a mixture of 100 kg. *p*-nitraniline 100 per cent and 52.5

kg. sodium nitrite 100 per cent (5 per cent excess of the theoretically required amount). The temperature is maintained at 5°-10°C. The time for diazotization is 2½-3 hr. The mixture must always show the presence of nitrite and acid (direct test), and it is finally stirred for 2 hr. to complete the reaction, then cooled to 0°C. Then are introduced 1 kg. carboraffin dry and, after 10 min., 2 kg. kieselguhr. After stirring for 10 min. at 0°C., the mixture (360 l.) is filtered, and the residue washed with 35 l. water.

*Separation of Diazonium Borofluoride* — In the clear diazo solution are introduced 83 kg. borax Ia refined powder



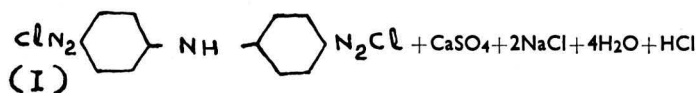
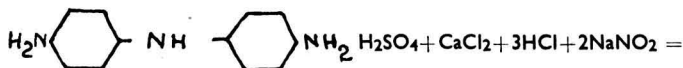
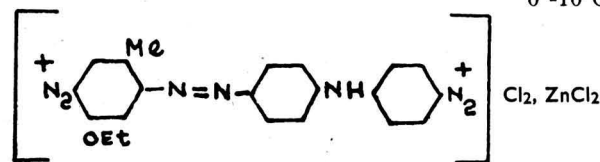
(= theoretical + 20 per cent) and 69.9 kg. hydrofluoric acid 100 per cent (= theoretical + 20 per cent) in 3 aliquots. The two are added alternately, beginning with borax, which, for the purpose of solution, is stirred for half an hour whereby the reaction remains acid. Before the hydrofluoric portion is run in, it is cooled to 0°C. After the addition of the last portion of hydrofluoric acid, the mixture is stirred for 2 hr., and cooled to 0°C. The volume is now 500 l. The precipitate is then filtered, washed with 37.5 kg. ice water and centrifuged. Yield, moist, 178 kg.

**Mixing & Drying**—The centrifuged diazonium salt is mixed with dehydrated Glauber's salt in the ratio of 2:1, and dried at 40°-45°C. for 36 hr.

**Standardization**—The standardization is carried out with alumina 23 per cent, and the balance with Glauber's salt and vacuum salt, so that the concentration corresponds to 22.5 per cent of the Fast Base. Yield is 95-96 per cent. In a small diazotization system, 300 kg. base 100 per cent can be handled.

#### APPENDIX 95

##### FAST BLACK SALT G (ESG—DS)



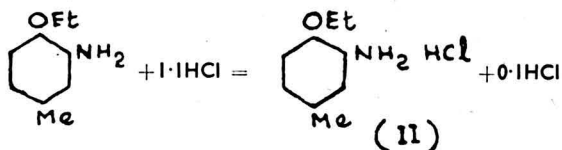
1. **Tetrazotization of p: p'-diaminodiphenylamine Sulphate**—In the large pre-warmed diazotization vessel are introduced 1,000 l. water at 80°C., and, then, under

stirring 100 kg. diaminodiphenylamine sulphate moist 100 per cent (222 kg., purity 45 per cent), followed by 186 kg. calcium chloride lye (30 per cent by weight = 55.8 kg. 100 per cent). Hereby separates coarse crystalline calcium sulphate. At the end of the addition, the mixture is cooled to room temperature, under stirring, 184 kg. hydrochloric acid 30 per cent (= 55.2 kg. 100 per cent) are added, and cooled to 20°C. by brine. For the diazotization, the mixture is cooled to 0°C. by adding 447 kg. ice. At the beginning of the diazotization, about 25 l. of the base suspension are withdrawn. The total acidity reaches to: 1,000 kg. added water, 130.2 kg. water from calcium chloride solution, 73 kg. water from the base sulphate moist, 184 kg. hydrochloric acid, and 446.8 kg. ice water; the result being 1,834 kg. diluted hydrochloric acid of 3 per cent by weight. Here the sulphuric acid is not calculated as sulphate of the base. Then, under brine cooling, diazotization is carried out with 133.2 l. sodium nitrite solution 52.1 vol. per cent = 69.4 kg. 100 per cent = theory, diluted with 213 l. water (i.e. with 346.2 l. sodium nitrite solution 20 vol. per cent). The diazotization time is about 60 min. and the temperature 0°-10°C.

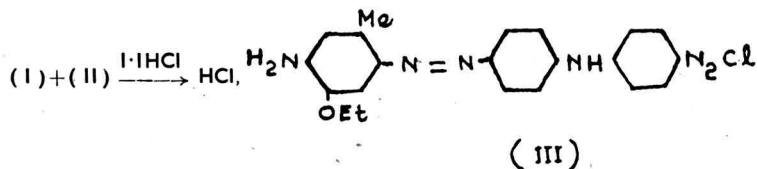
The speed of diazotization is at first quick, and is then slow. Test for nitrite. The diazotization is complete when the test shows a yellow stain. No large nitrite excess is permitted, and any excess is now removed with the withdrawn sulphate of the base (25 l.). Finally, some nitrite solution is added so that there is a slight excess of nitrite. The volume of the diazo solution with foam = 2,600 l. This is filtered, and the filter washed with 2 × 250 l. water. The volume of filtered

diazo solution = 2,600 l.

2. **Preparation of Amino-p-cresol Ethyl Ether ("Kresaminathyl")**—In the dissolving vessel are introduced 500 l. water at



70°-80°C., and without stirring, 77.8 kg. Kresaminathyl 100 per cent (= 78.4 kg. of purity 99.2 per cent = theory + 2.5 per cent). After about 20 min. the Kresaminathyl liquefies. Now are run in under stirring 69 kg. hydrochloric acid 30 per cent (= 20.7 kg. 100 per cent = theory + 2.5 per cent). The base goes completely in solution. Carboraffin dry (1 kg.) is added, stirred for 15 min., diluted with 500 l. water, and the mixture filter pressed. The vessel and press are rinsed with 300 l. water. The volume of solution, now in the coupling vat, is made up to 2,950 l. at 5°-10°C. by about 1,000 l. water and about 500 kg. ice.



### 3. Coupling to form the Intermediate Product

The tetrazotized solution is, under stirring, introduced into the coupling vat. Time of addition is 15 min. and coupling temperature 5°-10°C. The diazo vessel is rinsed with 100 l. water. The coupling is feeble at the beginning, and in order to quicken the coupling, sodium acetate solution is run in under the surface of the liquid as described in (4). Time of running in 2 hr.; temperature 5°-10°C. The intermediate substance now separates.

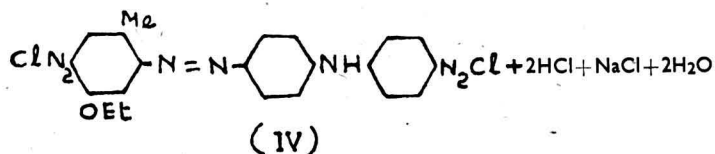
*Test*—A test with litmus paper should show no more red colouration (also no excess of tetrazo salt); it must give with diazotized *p*-nitroaniline solution a brown colouration (detection of excess of Kresaminathyl solution). After 4-5 hr. stirring the coupling is finished. In order that the intermediate product should separate as the hydrochloride, usually 96 kg. hydrochloric acid 30 per cent (= 28.8 kg. 100 per cent) and 400 kg. rock salt are added and stirred

for half an hour. Volume with foam = 6,500 l. Now the intermediate substance is collected in the filter press, and the coupling vat rinsed with about 1,000 l. mother liquor. The press is then blown dry.

4. *Preparation of Sodium Acetate Solution*—For 100 kg. diaminodiphenylamine sulphate 100 per cent are employed 213 kg. sodium acetate crystals dissolved in 600 l. water at 35°-40°C. under stirring, whereby all goes into solution and the temperature is 5°-10°C. The volume is now 725 l., and this sodium acetate solution is run in as described in (3). The dissolving vessel, after the removal of acetate solution, is rinsed with 50 l. water.

5. *Diazotization of Intermediate Substances (III)*—The intermediate substance (III) is removed from the filter press and suspended in the coupling vat with about 5,000 l.

water at 30°C. The volume is made up to 6,000 l. at 23°C. Then are added 123 kg. hydrochloric acid 30 per cent (= 36.7 kg. 100 per cent). After stirring for half an



hour, diazotization is effected with 69.9 l. sodium nitrite lye 52 per cent (36.4 kg. 100 per cent = theory + 5 per cent), diluted with 112 l. water, so that we have a 20 per cent nitrite solution. The diazotization temperature is 23°-25°C., and diazotization time 1 hr. The intermediate product goes into solution with a red-brown colour.

*Test for Nitrite & Acid*—The solution is stirred for half an hour after it shows the nitrite reaction. Hereafter are added 30 kg. kieselguhr. After stirring for 15 min. the mixture is filter pressed and washed with 600 l. water. The press is blown dry. Volume of filtrate 7,600 l.

6. *Separation of Zinc Chloride Double Salt*—The clear filtrate is treated with 76.4 kg. zinc chloride 100 per cent

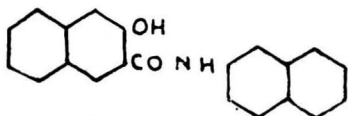
(= theory + 10 per cent) as lye of about 125 vol. per cent or dissolved in 100 l. water. The zinc chloride solution is run in very slowly. Volume 7,600 l. Then the whole is salted with 1,000-1,100 kg. vacuum salt (= 15 per cent of volume). The salting has to begin very slowly. After stirring for half an hour at 15°C., the product is sucked off. Yield, moist, is about 380 kg. (purity about 38 per cent; water about 28 per cent).

**Mixing & Drying**—The moist diazo salt is mixed in a Roland mixer with 220 kg. Glauber's salt R and 25 kg. zinc sulphate (anhydrous). Then it is dried on a Haveg sheet in a current of air for 48 hr. at 40°-50°C.

7. **Standardization of the Colour Salt to 20.5 per cent Purity**—The dried, purified diazo salt is mixed in a mixing machine for a short time, and then analysed. It is mixed with anhydrous zinc sulphate and Glauber's salt till a concentration of 20.5 per cent (about 22 per cent) of the base is obtained. This strongly diluted diazo salt is then powdered in a "Simplex-Perplex grinder". It is necessary to pulverize the salt very well and uniformly, 685 kg. Fast Black Salt G 20.5 per cent.

#### APPENDIX 96

##### NAPHTOL AS-SW



The raw materials used are pure toluene,  $\beta$ -naphthylamine, phosphorus trichloride and 2-hydroxy-3-naphthoic acid.  $\beta$ -hydroxy-naphthoic acid (250 kg.) is introduced in the reaction vessel, and over it toluene (1,800 kg.) is run in, and the two are stirred together. Under stirring is then added  $\beta$ -naphthylamine (190 kg.) and the contents of the vessel heated to 40°C. and stirred for 30 min. until all the  $\beta$ -naphthylamine is dissolved. The temperature is then raised to 75°C. in about 30 min., and at 75°-85°C. phosphorus trichloride (80 kg.) is added in the course of about 5 hr. At the end of this addition the temperature is raised to 107°C. in the course of 3 hr. and the contents maintained at 107°-111°C. for 20 hr. At the end of the reaction the

contents of the vessel are cooled to 40°C., and siphoned off into a distillation vessel, to which caustic soda (75 kg.) and water (1,000 l.) are added. The product is worked up as usual. From the above batch 396.7 kg. of product are obtained of purity 97 per cent, the yield thus being 95.3 per cent of theory. The m.p. of Naphtol AS-SW is 245°-247°C., and setting point 240°C.

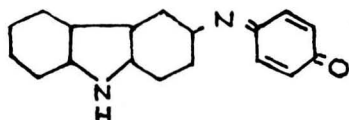
#### APPENDIX 106

##### *p*-NITROSOPHENOL

One adds in about 5 hr., at 0°C., an aqueous solution of 800 kg. phenol, 730 kg. caustic soda solution (33°Bé) and 698 kg. sodium nitrite (100 per cent) in 5,400 l. water to 6,400 l. of a solution of 1,586 kg. sulphuric acid (95 per cent pure) in water under stirring. The temperature is maintained at 0°C. by the addition of ice. The mixture is allowed to settle for 1 hr. and the supernatant liquid siphoned off. The residue is centrifuged and washed with ice water. Yield about 980 kg. paste = 840 kg. dry = 816 kg. 100 per cent pure.

#### APPENDIX 107

##### R BASE



In a 1,300 l. vessel, provided with a powerful agitator and cooling coils as well as jacket cooling, is placed 9,700 kg. of sulphuric acid (95 per cent pure), and cooled to 0°C. Herein is added 8 kg. "anthracene residue" and 800 kg. carbazole (about 98 per cent pure) in about 2 hr. After solution it is allowed to stand for 3-6 hr. and cooled to -28°C. In another vessel, provided with cooling coils, sulphuric acid (95 per cent; 4,300 kg.) is cooled to -10°C., and herein 592 kg. nitrosophenol, 100 per cent pure (= 670 kg. paste), is dissolved. After cooling to -27°C. the solution is transferred into the condensation vessel of 11,500 l. capacity. Under more powerful cooling, the carbazole solution cooled to -28°C., is added to the nitrosophenol solution in 2½-3 hr., so that the temperature is maintained at -23°C. (volume 9,200 l.). The indophenol solution is stirred afterwards

for an hour. An excess of nitrosophenol should always remain.

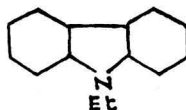
The reduction is carried out in 3 portions. A third of the indophenol solution is run into a lead tub of 30,000 l. capacity, provided with an agitator, in about 1 hr., to a mixture of 10,000 l. water, 3,000-4,000 kg. ice and 170 kg. of iron powder. During the time of addition 7,000-8,000 kg. of ice is added to maintain the temperature below 0°C. The temperature should be maintained close to -5°C. After additional 6 hr. of stirring, the temperature comes to 0°C. The reduction liquor is then green in colour. It is warmed to about 20°C. in about 2 hr. by means of steam, and then pumped at high pressure. The next day it is filtered through a filter press and washed with water until free of acid. The precipitate is once more treated with 12,500 l. of water and filtered. It is first washed with water, then with acidified water until free of iron. The paste from the whole batch (about 4,000-4,500 kg.) is dried on a cylinder with a steam pressure of about 4 atm. Yield 1,300 kg. dry (1,212 kg. 100 per cent pure; 92.4 per cent of theory).

*Hydron Blue RR* — The ingredients and the thionation process are the same as with *Hydron Blue R*. To distinguish *Hydron Blue RR* from *Hydron Blue R*, the finished dye paste is not acidified, but directly dried. The product is redder than the *R* mark, but is very variable in colour, tone and yield.

*Hydron Blue R (Powder)* — In the fusion pot, 1,710 kg. sodium sulphide ( $\text{Na}_2\text{S}$  conc.), 2,160 kg. sulphur and 1,800 kg. *R* base (leucoindophenol from carbazole and *p*-nitrosophenol) with 8,000 l. butanol are refluxed for 36 hr. The butanol is then distilled off in 6 parts. To each part in the distillation vessel are added 600 l. salt water and 1,200 l. distilled water, and one-sixth of the melt pumped in every 3 parts are transferred together to the desulphurizing vat, and desulphurized with 200 kg. sodium sulphide (crystals) for 1 hr. at 60°-65°C. The product is filtered through the press and washed free of sodium sulphide with 12°Bé salt water. The paste is then taken in about 12,000 l. distilled water, acidified with hydrochloric acid, stirred for  $\frac{1}{2}$  hr. at 50°C., filtered and washed with water until neutral. The paste is made up to about 20-22 per cent with water and dried to specification. Yield, about 4,250 kg. of the standard.

## APPENDIX 114

## N-ETHYLCARBAZOLE



In an agitated autoclave of 4,900 l. capacity, *N*-ethylcarbazole (1,800 kg.) and caustic potash (630 kg.) are heated to 150°-160°C., and carbazole (1,340 kg.) is then added. The autoclave is closed and heated in 6 hr. to 270°C. Water of reaction, some carbazole and secondary products pass over. The formation of potassium carbazole is now over. The temperature is allowed to fall to 215°C., and the valve leading to the receiver closed. Connection is established with an ethyl chloride cylinder containing 650 kg. ethyl chloride, which is heated to 10 atm. by means of a boiling water bath. The temperature falls to about 188°C., and in a run of about 16 hr. it is brought to 234°C. It is now allowed to cool to 160°C. in 8-12 hr., and excess ethyl chloride is blown over. After standing for 3 hr., during which potassium chloride is deposited, the supernatant base (1,600 kg.) is siphoned off into cold water, in which it solidifies. Then a further 1,600 kg. are siphoned off, and employed for the next operation. The residual potassium chloride is brought into solution in hot water, the base filtered through a filter press, and on cooling separated. This base is purified and then used as a solvent for the next batch. Yield, 1,533 kg. = 98 per cent of theory.

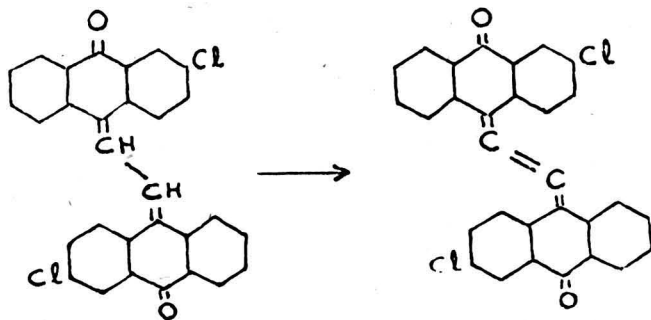
## APPENDIX 126

## 3-NITROCARBAZOLE

To dilute nitric acid from 1,600 l. water and 378 kg. nitric acid (40°Bé) in a stone-ware vessel with wooden stirrer, are added 300 kg. iron powder and finely powdered carbazole 100 per cent. The stirrer and vessel partition are rinsed with 200 l. water. The mixture is heated to 75°-80°C. with direct steam, and kept at this temperature for 4 hr. After addition of 1,000 l. cold water, it is filtered through a suction filter and washed neutral with cold water. The

product is dried at 50°-60°C. Yield, 370 kg. = 97.2 per cent of theory.

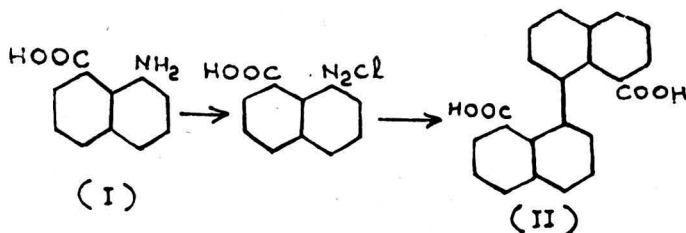
#### APPENDIX 129 ANTHRONETHYLENE



In 1,200 l. of spirit and 104 kg. caustic potash, 120 kg. of anthronethane are quickly added at 75°C. and a current of air is passed in closed vessel through a disc of 2.2 mm. orifice for 6-7 hr. at 76°C. until a blue-end product is just perceptible under the microscope. Then it is filtered through a pressure filter and washed with spirit. 2 parts are stirred with 1,500 l. of water and 30 kg. of hydrochloric acid, filtered, washed and dried. Yield, 78 kg.

#### APPENDIX 133

1:1'-DINAPHTHYL 8:8'-DICARBOXYLIC  
ACID



800 kg. of the acid (I) as paste are dissolved in 2 cu. m. water and 140 l. of soda lye; 61 kg. nitrite are added, and then a mixture of 450 kg. hydrochloric acid, 1,600 kg. ice and 1 kg. nitrite at 0°C. Neutralized with 800-900 l. soda solution, the diazo suspension is run into a mixture of 4 cu. m. water, 800 kg. ice, 150 kg. bicarbonate, a

cuprous chloride solution corresponding to 100 kg. copper, and 300 kg. ammonia at 15°-20°C. It becomes clear after the addition of 100 kg. ammonia and the acid (II) is precipitated from the filtrate with 450 kg. sulphuric acid, filtered, washed and dried in vacuum. Yield, 125 kg. as 100 per cent acid.

#### APPENDIX 137 PYRAZOLGELBKALI

A mixture of 110 kg. pyrazoleanthrone and 215 kg. caustic alkali, prepared in a mixing drum, is mixed in a 1,000 l. fusion kettle with stirring with a solution of 5 kg. caustic alkali in 45 kg. spirit, and the kettle closed. The temperature rises through heat of reaction to 100°C.; it is then raised to 125°-130°C. and held at that temperature for 6 hr. The fusion is then complete. It is then blown under pressure at 110°C. and diluted slowly with 500 l. cold water. It is then run under pressure into the oxidation kettle (6,000 l.), adjusted to 330 l. at 60°C., oxidized with air, filtered and the paste hydraulic pressed (80 atm.). It is dried *in vacuo* at 80°C. Yield, 100-125 kg. dry; 89 per cent of theory.

#### APPENDIX 138

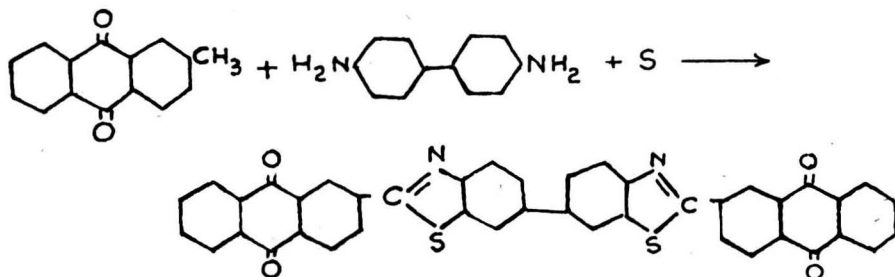
##### INDANTHRENE YELLOW GF

(a) *Crude Melt*—In a 1,000 l. melting vessel with ethyl carbazole bath are taken 500 kg. of lump sulphur at 225°C. Within 5 hr., through a funnel and screw conveyer, a uniform mixture prepared by grinding together of 120 kg. of  $\beta$ -methylantraquinone and 50 kg. benzidine are added. Temperature 225°-230°C. The inner temperature is kept for 2 hr. at 240°C., further for

2 hr. at 250°C. and finally 2 hr. at 260°C. At 260°-265°C. it is stirred for 3 hr. In the end the thick melt is drained out. The cooled melt is ground in a disintegrator.

(b) *Desulphurization*—In a 10,000 l. vessel, 5,100 kg. crystalline sodium sulphide are melted with indirect steam and brought to 100°C. In 1 hr. are added 1,830 kg.





ground melt and stirred for 3 hr. more at 102°C., diluted to 9,000 l. and filtered at 80°-85°C. After washing with a solution of 150 kg. sodium sulphide in 1,500 l. water, and then with hot water till filtrate is colourless, the product is dried at 65°-70°C. Yield, 525 kg. crude dry dye.

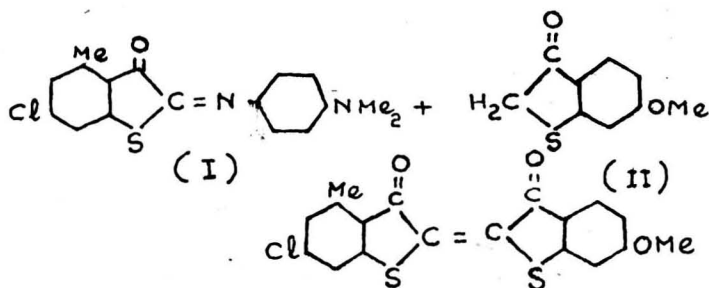
(c) *Solution in Sulphuric Acid*—To 4,200 kg. 96 per cent sulphuric acid in a 5,600 l. vessel at 50°-100°C. are added in 1 hr. 525

kg. crude dye. Stirred for 4 hr. at 100°C., the solution is cooled to 20°C. and poured in 5,000 kg. ice and 9,000 l. water in 20 cu. m. stone vessel and stirred for 3 hr. Final temperature 10°C. Filtered through wooden press with nitrocellulose cloth and washed with cold water for 4 hr. The green paste weighs 2,100 kg. of which 21 per cent is obtained on drying.

(d) *Pure Paste*—The green paste is stirred in a vessel of 5,000 l. with 1,000 l. water for 5 hr. It must show a neutral reaction. Then it is poured in an alkaline stone chlorinating vessel of 22 cu. m. under stirring. According to experience the necessary hypochlorite liquor = 3,500 l. (14 per cent chlorine) and caustic soda to bring caustic soda content to 2.5 per cent are added. Stirred for 4.5 hr. and allowed to remain overnight, then heated to 100°C. with direct steam. When chlorine solution is not sufficient, more is added till potassium iodide paper after 1 hr. still gives the best. Mass is diluted to 15,000 l. and filtered through wooden press, and washed with hot water till the washing are neutral to turmeric. Pure orange wet paste, 1,700 kg. = 440 kg. dry.

## APPENDIX 140

## INDANTHRENE SCARLET B

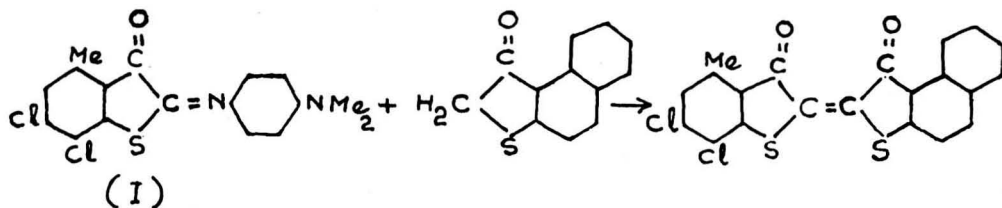


In a double-walled, enamelled vessel are introduced dry chlorobenzene (1,600 l.), "rosa-anil" (I) (132 kg.), methoxythionaphthene (II) (79 kg.) and piperidine (8 kg.). These are heated to 115°C. and maintained for 2 hr. at 115°-118°C. At this stage, condensation of an insoluble dye takes place. The dye is filtered off at 50°C. and washed thrice with 350 l. of warm chlorobenzene (50°C.) till the filtrate is light in colour. The filter cake is freed from chlorobenzene by steam and dried at 70°-80°C. The yield is about 190 kg. of standard dyestuff.

## APPENDIX 142

## VAT BROWN IVD

The "anil" (I) (114.5 kg.), 2:1-naphthoxythiophene (60 kg.) and piperidine (6 kg.) are stirred in dry chlorobenzene (1,350 l.), and heated to 115°C. The temperature is maintained at 115°C. for 2 hr., and then cooled to 70°C., and the product filtered. The filter cake is washed thrice with 300 l. warm chlorobenzene each time, freed from chlorobenzene with steam, and dried. Yield about 160 kg. paste or 113 kg. dry powder.

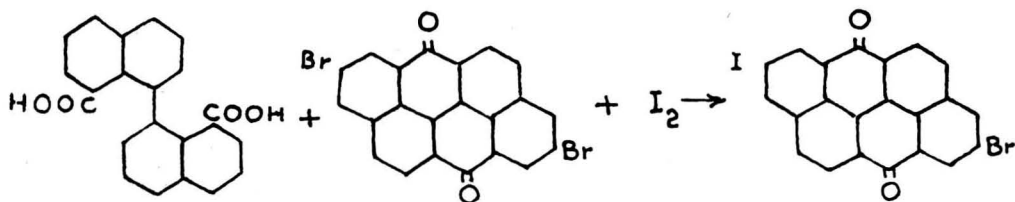


## APPENDIX 147

## VAT SCARLET RM

Dinaphthylidicarboxylic acid (89.4 kg.), dibromoanthrone (80 kg.), and arsenic acid (101 kg.) are dissolved in monohydrate (1,600 kg.) at 45°C. in the course of 1 hr., and diluted with water (16 l.) when the

red colouration, which then turns green. The contents of the vessel are allowed to remain overnight. Then, under cooling, oleum 65 per cent (490 kg.) is run in at 35°C. After cooling to 25°C., iodine (1.3 kg.) and bromine (90.5 kg.) are added. The contents of the vessel are now heated and agitated. In the course of an hour the temperature is

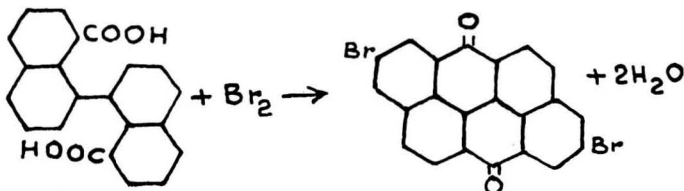


temperature rises to 60°-70°C. Next day iodine (85 kg.) is introduced and the contents heated to 132°C. in 2-3 hr. and maintained at 132°-135°C. for 5 hr. It is then cooled, and with the addition of water (400 l.) the sulphuric acid concentration is brought to 85 per cent. It is now filtered and the filter cake washed 5 times with 85 per cent sulphuric acid (200 l. each time). The filter cake is agitated with 2,000 l. water at 45°C. for 4 hr., refiltered and washed free from acid. The filter cake is now stirred for 1 hr. with warm water (2,000 l.) at 80°-90°C. and soda lye (240 kg. 33°Bé). After this it is filtered and washed. The yield is 550-650 kg. paste = about 195 kg. dry = 86.2 per cent of theory.

## APPENDIX 148

## INDANTHRENE BRILLIANT ORANGE RK

In an enamelled vessel of capacity 2,000 l. are introduced monohydrate (1,550 kg.) and 1:1'-dinaphthyl-8:8'-dicarboxylic acid (188 kg.) under cooling. Solution takes place first with a

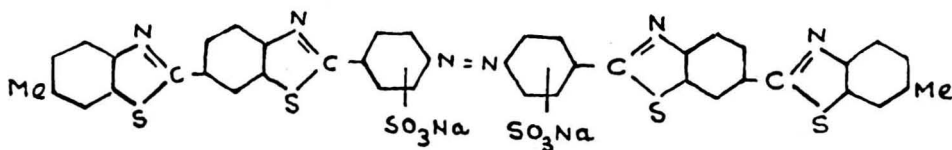


brought to 30°C., and then more slowly to 65°C., where it is maintained for 3 hr. After cooling to 50°-55°C., warm water at 60°-70°C. (450 l.) is run in. The contents are now filtered off and washed neutral. By the introduction of water the sulphate are hydrolysed and light orange-coloured dibromoanthrone results. The dyestuff obtained in paste form is 750-800 kg., the yield being 90 per cent of theory.

## APPENDIX 153

## SIRIUS LIGHT YELLOW RT

487 kg. of primuline acid (m.w. 373) are suspended as paste in 2,000 l. of water, and dissolved with about 270 kg. of soda lye solution of 33°Bé. It is diluted with water and ice to 8,000 l., and at 3°C. 115 kg. sodium hydroxide lye of 33°Bé and, then, with good stirring, 2,400 l. of sodium hypochlorite



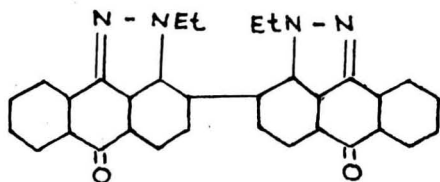
(224 kg. active chlorine) are added in 6-8 min. The temperature rises thereby to 7°-9°C., then slowly to 15°-17°C. After standing overnight, excess chlorine smell should faintly persist; otherwise, a little hypochlorite solution is added. The dye is salted out with rock salt 650 kg. as a 11°Bé solution. Yield, about 780 kg., standardized at about 1,780 kg.

120 kg. "Pyrazolgelbkali" are ground with 200 l. chlorotoluene for 18 hr. in a flint mill, and discharged into the alkylation kettle. The mill is rinsed with 900 l. chlorotoluene. Total charge = 1,000 l. = 1,150 kg. chlorotoluene. Then are added 112 kg. ethyl *p*-toluenesulphonate and 12 kg. powdered caustic alkali. The mixture is heated at 125°-130°C. for 9 hr. when alkylation is complete. Then it is filtered hot. The cake is washed, stirred thrice with 200 l. chlorotoluene at 100°C., filtered off, and finally washed into the suction apparatus with 100 l. chlorotoluene and sucked dry. The adhering chlorotoluene is steam distilled. The dye is filtered and washed neutral with water. Yield about 140 kg. wet or about 80 kg. dry.

The filtrate of chlorotoluene is distilled with steam; yield about 95 per cent of the used chlorotoluene.

#### APPENDIX 154

#### DIETHYL RED (INDANTHRENE RUBINE R)



## A Method for the Preparation of Dioctyl Sebacate

DIIOCTYL SEBACATE HAS COME INTO USE AS a plasticizer<sup>1</sup> as it imparts not only low temperature flexibility to polyvinyl chloride and its copolymers, but also possesses non-volatility characteristics obtainable with dicapryl phthalate or dibenzyl sebacate. Methods for the preparation of this plasticizer are not reported in literature. A method employing *p*-toluene sulphonic acid as catalyst has been worked out in these laboratories. The usual catalysts — conc. sulphuric acid and dry HCl gas — gave poor yields. The method of preparation is briefly as follows:

Sebacic acid (10 parts), secondary octyl alcohol (13 parts), and *p*-toluene sulphonic acid (1 part) were dissolved in benzene in a round-bottom flask and the mixture refluxed for 20 hr. The water formed was removed through a trap. The reaction mixture was extracted with ether, the

etheral solution washed with dilute sodium carbonate solution and dried over anhydrous sodium sulphate. After removal of ether, the residue was distilled under 5 mm. pressure and the fraction, boiling between 255°-257°C., collected. A yield of 34.5 per cent pure dioctyl sebacate on the weight of sebacic acid was obtained. The product is a colourless, odourless liquid; sp. gr. at 15°C., 0.920; sap. val., 258; acid val., 0.4; ref. index at 20°C., 1.4498.

J. P. VERMA  
KARIMULLAH

Chemical Laboratories  
Council of Scientific & Industrial Research  
Delhi

#### REFERENCE

1. *Rubber Age*, 1945, 56, 633.

# A Study of the Colouring Matter of *Tambul* Seeds\*

T. R. SESHADRI

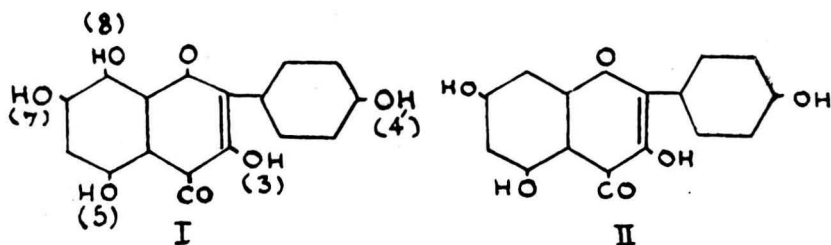
Andhra University, Waltair

**F**LAVONES and flavonols forming the most important group of anthoxanthins were originally valued highly as mordant dyes. In this respect they have been almost completely displaced by synthetics. More recently, however, their physiological properties have been attracting attention<sup>1</sup>. A precise knowledge of these yellow colouring matters is useful in other directions also. For example, it gives a deeper insight into the botanical classification of plants. As the result of the endeavours of chemical investigators in various countries, considerable progress has been recorded not only in the discovery of new types of compounds, but also in the development of newer and more efficient methods of studying their molecular structure and effecting their synthesis which will lead ultimately to a comprehensive picture of their evolution and function.

Hydroxy flavones and flavonols occur free in a number of cases. But it is more common to meet with them in a combined form. One or more of the hydroxyl groups are protected either by glycoside formation or by methylation or methylenation. Exact knowledge as to why these modifications are needed in the plant is not yet available. But it could be safely stated that factors relating to solubility, stability to oxidation and light absorption are involved. When there are a number

of hydroxyl groups in a molecule capable of protection in the above manner, the exact position or positions involved is of some importance because considerable variation in properties can arise thereby. Methods of locating these positions have been explored continuously and marked success has been achieved during the past few years. The study of the colouring matter of *tambul* seeds provides an example of such work on partial methyl ethers.

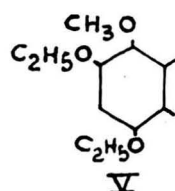
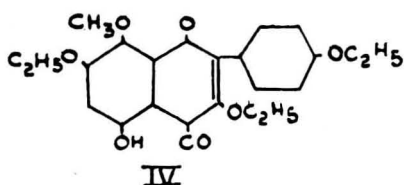
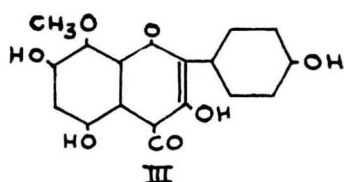
It is better to start with the discovery in 1935 of herbacetin (I) by Neelakantam and Seshadri<sup>2</sup> in the Indian cotton flowers, *Gossypium herbaceum*. The constitution of the new flavonol was soon determined at Waltair<sup>3</sup> and confirmed by synthesis at Oxford<sup>4</sup>. But there was considerable difficulty in isolating it from the cotton flowers, because it occurs along with 3 other flavonols, gossypetin, quercetin and kaempferol, partly free and partly as glucoside. The method of synthesis also was not easy at that time though a considerably simpler one has recently been discovered<sup>5</sup>. In order to supply other workers who were in need of small authentic samples, a search for more convenient sources was made. The flowers of *Thespesia populnea*<sup>6</sup> yielded herbacetin, but it was again associated here with kaempferol and the question of separation existed. Bose and Bose<sup>7</sup> subsequently reported the



\* Paper presented at the Symposium on Plant Products held during Silver Jubilee Celebration of the Indian Chemical Society (January 1949).

isolation in a very small yield (0.006 per cent) of tambulin from *tambul* seeds which form a well-known Indian drug and contain a sweet-smelling essential oil. This was shown to be a trimethyl ether which could be demethylated to herbacetin. Since the seeds were readily available and no difficult separations were involved, they appeared to form a satisfactory source for the quick preparation of small samples of herbacetin. We<sup>8</sup> were actually successful in obtaining much better yields (0.03 per cent) of the colouring matter than previous workers. Surprisingly, however, it was not tambulin, but a new chemical

But in cases where there are a number of free hydroxyl groups, this procedure has not worked satisfactorily. Neither does the decomposition take place well nor is the identification of the fission products easy. Consequently, in the study of partial methyl ethers, we have been adopting the practice of protecting these free hydroxyls by ethylation and then subjecting the mixed methyl ethyl ether to fission. By this means not only the decomposition of the flavonol molecule and identification of the products are rendered smooth and precise, but even confirmation by synthesis becomes comparatively easy.

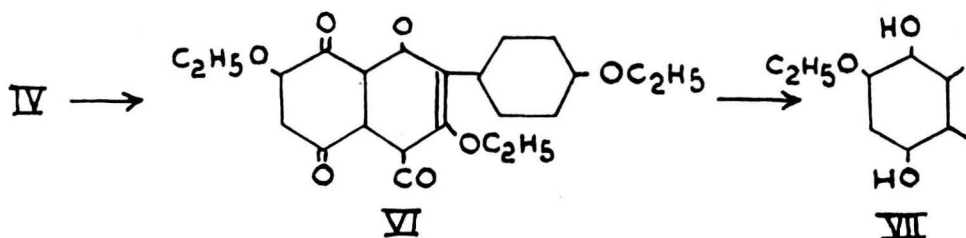


entity which we named tambuletin. It was a monomethyl ether yielding herbacetin on demethylation and thus our original object of preparing this flavonol was attained.

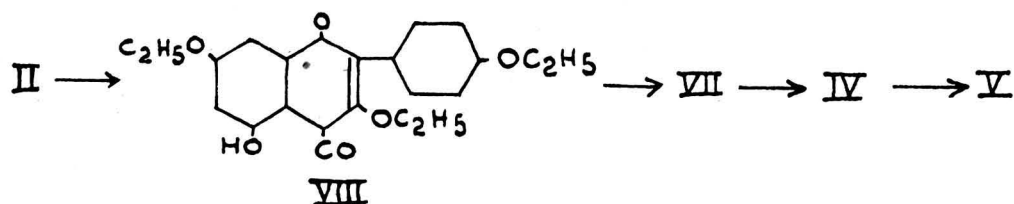
Next the chemical constitution of tambuletin was investigated in detail. Its properties and colour reactions indicated that it had the methoxyl group in the 8- position (III). It differed markedly from herbacetin (I) and resembled closely kaempferol (II) in the reactions with dilute alkali, alcoholic ferric chloride and *p*-benzoquinone. It could, therefore, be concluded that the hydroxyl in the 8- position was immobilized by ether formation. Though these colour reactions are very useful for purposes of diagnosis, errors have arisen in the past by depending too much on them. Confirmation by more definite methods had to be sought. It was usual to subject the new substance (partial methyl ether) to fission with alkali and identify the products of decomposition.

Actually in the case of tambuletin<sup>9</sup> though ethylation was carried out, there was no need to resort to alkali fission. Simpler methods whose usefulness had been fully established by our earlier work were adopted. Tambuletin could be ethylated in two definite stages yielding a tri- (IV) and a tetra-ethyl (V) ethers. The former exhibited the reactions for a free hydroxyl in the 5- position and was oxidized readily with nitric acid yielding a quinone, reduction of which produced a quinol. The final product was found to be identical with a synthetic sample of 3 : 7 : 4'-triethoxy-5 : 8-dihydroxy flavone (VII). It could, therefore, be concluded that the oxidation of *o*-triethyl-tambuletin involved demethylation and this could happen only if the methoxyl group was present in the 8- position. The transformations involved were represented as follows.

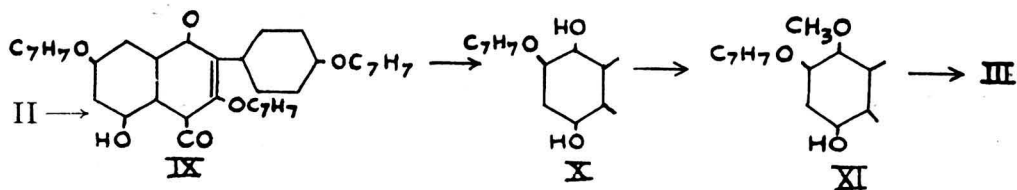
More conclusive evidence was provided by the synthesis of the tri- (IV) and tetra-ethyl



(V) ethers of tambuletin. This work as well as the synthesis of the quinol (VII) could be readily accomplished by the application of the new discovery of facile nuclear oxidation in the flavone series<sup>15</sup>. Kaempferol (II) was the starting point; it was subjected to partial ethylation to form *o*-triethyl-kaempferol (VIII). Oxidation with alkaline persulphate yielded quinol (VII). Partial methylation with dimethyl sulphate and anhydrous potassium carbonate in acetone solution converted it into the 8-methyl ether, the resistant 5-hydroxyl being left out. This was identical with *o*-triethyl-tambuletin (IV). Subsequent ethylation yielded 8-monomethyl-3:5:7:4'-tetraethyl ether of herbacetin which was identical with *o*-tetraethyl tambuletin (V). The course of the various stages in the synthesis had been well established earlier by detailed study of simpler examples.



The synthesis of tambuletin<sup>10</sup> itself was carried out as the next stage. Here again kaempferol was the starting material and nuclear oxidation and partial methylation were employed. But some of the hydroxyl groups had to be protected by partial benzylation and the benzyl groups removed finally. There was some difficulty in the benzylation and in the oxidation of the benzyl ether. But with adequate precaution the reactions could be accomplished satisfactorily.



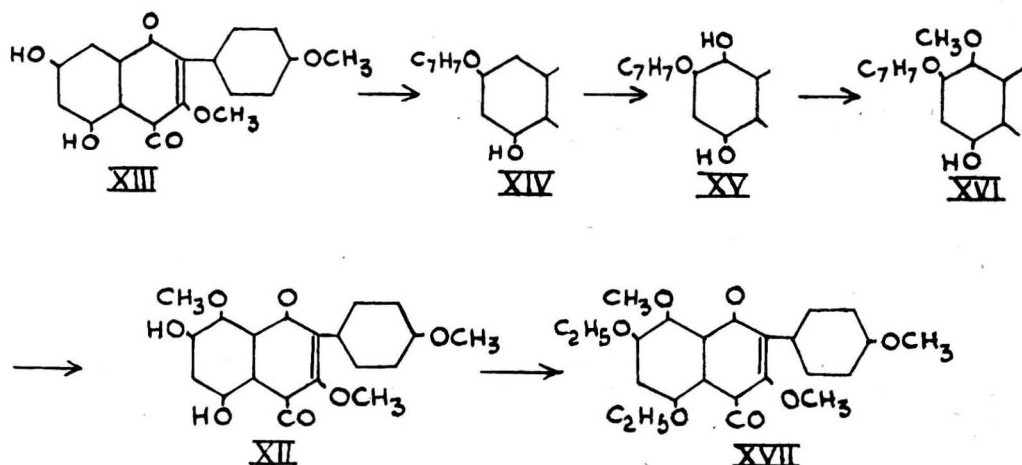
It is rather surprising that in the number of extractions of *tambul* seeds that we carried out, only tambuletin was isolated and no tambulin as originally reported by Bose and Bose<sup>7</sup>. This is a puzzle which remains still

to be solved. From their study of tambulin Bose and Bose<sup>7</sup> proposed that it was 3:8:4'-*o*-trimethyl herbacetin (XII). We could prepare a substance of this structure starting from 3:4'-dimethyl ether of kaempferol (XIII) and passing through the stages indicated below<sup>11</sup>.

The synthetic product differed markedly from tambulin. Its melting point was much higher and so also was the melting point of its acetyl derivative higher than that of tambulin acetate. That the structure of the synthetic substance was correct was confirmed by conversion into its diethyl ether which was found to be identical with a synthetic sample of 3:8:4'-trimethoxy-5:7-dithoxyflavone (XVII). Consequently the constitution of tambulin had to be revised. Bose and Bose showed that a methoxyl was definitely present in the 4'- position because

anisic acid could be isolated as a product of fission with alkali. Since the compound failed to reduce alkaline *o*-dinitrobenzene or chloropentamine cobaltichloride, there was proof that it did not have *ortho* or *para* dihydroxy groups. It was, therefore, concluded that a methoxyl was in the 8- position. This was also justified by analogy with tambuletin which has a methoxyl in the same position. They considered that the third methoxyl was in the 3- position, since the

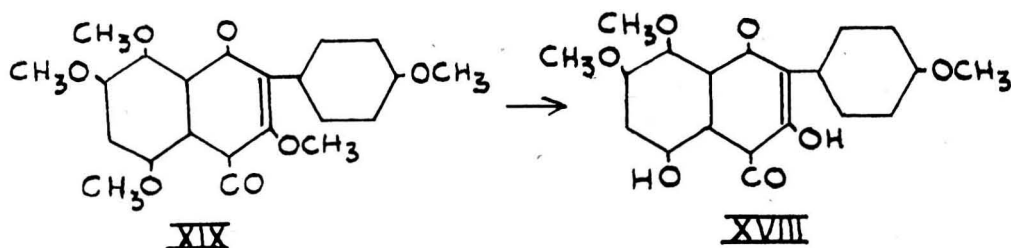
compound was stable to aerial oxidation in alkaline solution. This last evidence did not appear to be quite dependable. For we had found in the course of other work that compounds like galangin and kaempferol

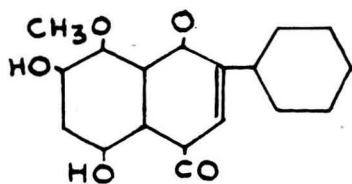
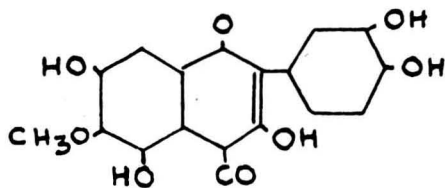
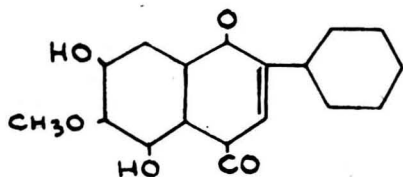
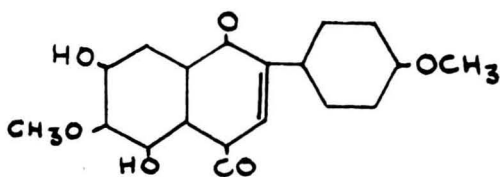


having a free hydroxyl in the 3- position and a small number of hydroxyl groups in the other parts of the molecule are fairly stable when exposed to air in alkaline solution (milder condition), and Perkin<sup>12</sup> had recorded that when air is aspirated through an alkaline solution of galangin-3-methyl ether (more drastic condition), it is oxidized with the formation of benzoic acid and phloroglucinol. The method cannot, therefore, be considered to be conclusive for the present purpose. In view of the above-mentioned synthetic work, the third methoxyl group should be located elsewhere. Of the two possible positions, 5 and 7, the former is known to be resistant to methylation and is the last to be affected in the plant or in the laboratory. Consequently, position 7 seemed to be more probable. Further, the constitution of 7:8:4'-o-trimethyl herbacetin (XVIII) seemed to be in accord with the lower melting point of tambulin. This could be checked by synthesis. A substance of this constitution could be most conveniently made by the partial demethylation of herbacetin-pentamethyl ether (XIX) using anhydrous alumi-

nium chloride in nitrobenzene solution. This method of simultaneous attack of the 3- and 5- methoxyl groups had been adopted successfully in a number of analogous cases and the synthesis of partial methyl ethers of galangin, kaempferol and quercetin reported earlier<sup>13</sup>. The reaction was found to proceed smoothly in the present case<sup>14</sup> and the product was found to have the properties reported for tambulin; a similar agreement was found between the acetates. Hence the constitution of tambulin should be considered to be that of the 7:8:4'-o-trimethyl ether of herbacetin (XVIII).

In conclusion a word is necessary regarding the structural interest in the colouring matter of *tambul* seeds. In tambuletin and tambulin, the hydroxyl in the 8- position is found to have undergone methylation in preference to those of the 7- and 3- positions, though biogenetically there is clear indication that the former should have a later origin than the latter. The reasons for this preferential partial methylation and its mechanism are not yet known. Under this category comes the flavone methyl ether wogonin



**XX****XXI****XXII****XXIII**

(XX) which is found in the roots of *Scutellaria baicalensis*. A parallel series is presented by the 6-methyl ethers like patuletin (XXI), oroxylin-A (XXII) and pectolarigenin (XXIII) in which the 6-position is preferentially methylated.

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# REVIEWS

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**A Source Book in Greek Science**, by Morris R. Cohen ( McGraw-Hill Book Co. Inc., New York ) 1948, pp. xvi+579.

THE PRESENT BOOK IS THE FIFTH IN THE series of source books in the history of the sciences published under the general editorship of Prof. Gregory D. Walcott. The earlier volumes in the series dealt with astronomy, mathematics, physics and geology respectively. A source book in medieval science is also to be issued, and, further, a volume containing the most important contributions of the major sciences from 1900 to 1950 is planned for publication by about 1960; and, according to the general editor, a similar volume is to be issued each half century thereafter.

Stimulated largely by the growing concern in recent years of scientists by the impact of science on society, the history of science and its social implications has attracted considerable attention. Of all the adventures of the human mind, the scientific adventure has proved the most fruitful and profound. In fact, the very idea of "progress" is comparatively modern: it is a result of the scientific and technological revolution. As W. K. Clifford, noted for his "profound and orderly understanding", said in his book *The Commonsense of the Exact Sciences* published in the last century: "*scientific thought is not an accompaniment or condition of human progress, but human progress itself*". In our own day, Sarton, the distinguished historian of science, has emphasized in his *Study of the History of Science* that "the acquisition and systematization of positive knowledge are the only activities which are truly cumulative and progressive. The history of science is the only history which can illustrate the progress of mankind." With the increasing specialization that is inevitable with the development of science and with its growing utilization by society, it has become all the more necessary for scientists, and no less for others, to acquire a proper understanding of the limitations of science and of the factors that determine its progress and development. This understanding, and also a balanced science education, is greatly

helped by a study of the history of the growth of science under different civilizations and social environments and how it has acted and reacted on society. The President of the Harvard University, Dr. J. B. Conant, has vigorously advocated in his highly interesting book, *On Understanding Science*, the historical approach in science teaching. Dr. Conant has urged that the strategy of science can best be understood by a study of typical case-histories and he has provided most illuminating illustrations of them in his book, taken mostly from 17th and 18th century science.

The publication of the present source book in Greek science is most opportune and the work is assured of a wide welcome. As the author points out in the preface, the extracts from Greek science presented in the book cannot take the place of a history of science, nor can they offer a systematic study of Greek science or of the position it occupied in Greek civilization. "But no history of Greek Science can convey an adequate picture, unless the readers see something of the original source on which the historian relies. The presentation of these sources seems all the more desirable, because the material on which we draw is often fragmentary, and scattered in many diverse volumes." The study of the volume will help in correcting, what the author calls, an established popular error, namely that the Greeks were merely speculators and that the natural science began in the 17th century with Galileo and his successors.

The book opens with a section on mathematics, and it is in mathematics, particularly in geometry, more than in any other branch of science that the Greeks made their greatest contribution. We find here the proof (Euclid), oft-repeated for the last two thousand years, that  $\sqrt{2}$  is an irrational number. We read here (page 20) the well-known theorem that the number of primes is infinite. An interesting extract from Archimedes is on the sand-reckoner. The sand-reckoner described a system of representing numbers different from the usual Greek system, and it allowed representation of any number, however large. To illustrate the utility of the system, Archimedes estimated the

number larger than the number of grains of sand necessary to fill the universe. (Curiously enough, the estimate of the Syracuse philosopher-scientist is comparable to the number of fundamental particles in the universe estimated more than 2,000 years later by Eddington.)

Extracts are given from Aristarchus of Somos (the Copernicus of antiquity) on the sizes and distances of the sun and moon. Also extracts are given dealing with the precession of the equinoxes. Ptolemy's arguments for a fixed earth are reproduced from his *Almagest*. The section of mathematics and astronomy covers a little less than 200 pages.

Next comes the section of physics of about the same length. We find here extracts from Aristotle on the equations of Aristotelian dynamics. Aristotle maintained, among other things, that "within bodies moving with their proper motions, the larger moves quicker". Again, we have an extract from Philoponus (page 279) contending that Aristotle was wrong in his assumption that a larger weight falls in a given time through a greater distance than a smaller weight does in the same time. The Greek mechanics, like their astronomy and a good deal of physics, was based not on experimental observation but on deductions drawn from general principles—the general principles being assumed on philosophical and speculative rather than on scientific grounds. Witness, for instance, Aristotle's dictum that nature abhors vacuum.

Reasoning by analogy (for instance the analogy between macrocosmos and microcosmos) played a significant part in the derivation of principles. This gave them a wide sweep, but as the analogies were often superficial (in the scientific sense), the principles so derived were of little value in natural science.

Interesting extracts are given relating to siphon, simple machines, and so on. The last 150 pages of the book are devoted to zoology, botany, animal and human psychology. Here we have interesting extracts taken from Aristotle who, for his acumen as a biologist, ranks supreme in the same sense that Archimedes does in the realm of mathematics and physics. Here one finds reference to Indian elephants (page 537). Interesting extracts from Aristotle relate to the senses of animals: "There is no doubt but that fishes have the sense of taste. . . With regard to sight and hearing

we cannot make statements with thorough confidence or on irrefutable evidence."

It is obviously difficult to summarize the contents of a work of this kind, and more so for a reviewer who can claim no expert knowledge of this fascinating subject of Greek science. The book covers an extensive field and it appears that practically every work of interest and importance finds a reference in it. It is a book that should find a place in every library concerned with science and its teaching.

D. S. K.

**The Chemistry and Manufacture of Indian Dairy Products**, by K. S. Rangappa & K. T. Achaya (Bangalore Printing & Publishing Co. Ltd., Bangalore City), 1948, pp. 189. Price Rs. 10.

THE IMPORTANCE OF DAIRY INDUSTRY HAS not been adequately recognized in India and it is, therefore, an encouraging sign when a book on the chemistry and manufacture of Indian dairy products appears in the market. As has been correctly pointed out by Professor J. C. Ghosh in his foreword to this book, the Indian dairy products "are fundamentally different in nature from the dairy products in Europe and America" and hence the problems connected with these products need considerable research and extensive application of the results of research before the dairy industry can be built up on a firm scientific foundation. Unfortunately, however, research in dairy industry, not being as glamorous as that in many other subjects, has not been financed or encouraged as much as its importance would deem it necessary. What little has been achieved by a few workers in Government laboratories or research institutions needs to be extended and more encouragement by way of better-equipped laboratories, conditions of employment, etc., is needed before the results of our research investigations can be usefully employed by the industry. It would, however, be unsafe to incorporate into a text-book results which have not been tried out under a variety of conditions or confirmed by a few other workers. For example, on page 72, quoting the work of Roy and Bhatnagar, the authors of the book point out that during fermentation of milk its nitrogen content increases. To the best knowledge of this reviewer, this observation of nitrogen fixation in milk

has not been recorded by any other worker in India, Europe or America. It would have been interesting to read some hypothesis postulated to explain this rather unusual phenomenon.

The book has been divided into 3 parts: part I—milk and unfermented milk products; part II—fermented milk products; and part III—ghee. Since this book deals primarily with dairy products peculiar to India, products such as ice-cream and milk powder, which are also being manufactured and used in India to an increasing degree in recent years, should have been included.

Discussing the bacterial quality of milk marketed, the authors quote reference (page 51) to a book published in 1916 to point out that "it is encouraging that the samples were free from pathogenic organisms like *B. Typhus*, *tuberculosis*, *diphtheria* or *cholera*". This is most misleading, since it is well known that the detection of pathogenic organisms in milk is quite a difficult and laborious procedure. Incidentally, the sentence does not read well in a book, especially, since the name of the first is now recognized as *Eberthella typhosa* and the latter 3 are names of diseases and not organisms. The authors emphasize that under Indian conditions it is quite easy to produce milk of bacterial count of 30,000 or less per ml. (page 49). Actually, in the results reported in Table XXX, even in the herd milk at the Government farm of *Indian Dairy Research Institute*, the bacterial count was about 100,000 per ml. It will need a considerable reorganization of our dairy methods before milk of a bacterial count of the order of 30,000 per ml. can be produced. Again, on page 55, it is pointed out that "And it is to this practice (of heat treatment of milk) that the freedom from milk borne diseases in India is to be mainly attributed". It is well known that in many homes the milk is not heated to a sufficiently high temperature or for sufficiently long periods to ensure the complete destruction of pathogenic organisms. If we do not hear of diseases transmitted through milk, this reviewer believes that it is largely due to lack of adequate public health surveys and "follow ups" of epidemics rather than the absence of such milk-borne epidemics.

The arrangement of the textual matter is good, although many printer's mistakes

need corrections. To cite a few glaring examples: page 140, line 15, "interreaction ship" instead of "inter-relationship"; page 141, "voriations" instead of "variation", etc. The get-up of the book is excellent although many research workers in the field may find the price of Rs. 10 somewhat beyond their means.

I.

**Turning and Boring Practice & Drilling and Surfacing Practice**, by Fred H. Colvin & Frank A. Stanley (McGraw-Hill Book Co. Inc., New York), 1948, pp. xv+531. Price \$4.75.

ALTHOUGH THE AUTHORS HAVE WRITTEN two books separately, one on *Turning and Boring Practice* and the other on *Drilling and Surfacing Practice*, in the opinion of the reviewer these are considered as two volumes under general machine-shop practice. A comprehensive survey of modern machine-shop practice becomes increasingly difficult because of the constant changes in machines, methods and materials employed. For example, where formerly the planer and shaper machines handled all flat-surface work, the milling machine, grinder and more recently the broaching machine are now used almost exclusively on production work. In addition to new machines, new methods have come into practice with the introduction of new materials.

The authors have made these points quite clear in these two volumes and, in their opinion, all those connected with a machine shop, be he an executive or a workman, should have wider knowledge of factors which affect the smooth working of a machine shop. The authors have also emphasized the fact that principles of machine operation have remained unchanged even though vital changes in the use of machines have come to stay in a modern machine shop.

In the first volume of the work, the authors concern themselves with turning and boring practices. This volume is divided into five sections. The chapters on turret and semi-automatic lathes are written very exhaustively and very few books give so many details about the new techniques employed in production work. Boring has received special attention in a separate section. A large number of illustrations given in this section showing how to execute large boring operations will be found very useful.

The section on cutting tools for different materials contains valuable information such as use of carbide tools, suggestion and methods for machining various metal parts, cutting oils and other coolants. Also described in the same section are the methods to be employed in case of non-metallic materials such as rubber and plastics.

As drills and drilling machines are used to a greater extent than other machines in machine-shop practice, it is quite appropriate that the authors have devoted a complete volume for "drilling and surfacing practice". This volume runs into six sections describing in detail drilling, reaming and tapping, planers and shapers, milling, milling cutters and broaching.

These volumes are very authoritative covering almost all the fundamental machine-shop operations. A special feature of this work is the inclusion of a large number of tested, new workshop practices to make difficult jobs easier, and many short-cuts employed in the mass production are also elaborately discussed. Great stress is laid on the economy of time and money in all the operations.

The authors need no introduction and their work is so thorough and exhaustive that the reviewer strongly feels that these books will prove to be valuable additions to all technical libraries and that all those connected with machine-shop industry could use them with great advantage.

**Handbook of Indo-Australian Trade — 1947**, compiled by E. V. S. Maniam & M. L. Gupta (Bureau of Economic Research, Kanpur), 1948, pp. x+60. Price Rs. 5.

THIS PUBLICATION WHICH IS THE RESULT of an economic survey of the two countries, is a sequel to the widespread desire felt in both the countries for increased trade relationship. It presents in a handy form valuable information regarding the possibilities of Indo-Australian trade.

It may be recalled that an Indian Industrialists' Delegation visited Australia in 1945 and a similar Delegation from that country came to India in 1946 to explore ways and means of fostering trade between the two countries. The Bureau of Economic Research, Kanpur, was asked to prepare a factual memorandum for the guidance and information of the Australian Delegation, and the present volume is an enlargement on the report.

The handbook gives, in a concise form, facts and figures which are not easily available elsewhere. It is an exhaustive survey of the economic resources of the two countries and indicates the deficiencies of each country which can be met by the other. The different offices under the Commonwealth Ministry, Diplomatic and Trade Consulates and others directly and indirectly concerned with the administration of trade, commerce and industry have been mentioned to facilitate trade inquiries. The names and addresses of the different industrial and commercial bodies in Australia have also been given and these will be very helpful to Indian exporters.

The handbook also contains a directory section, giving names and addresses of Australian and Indian manufacturers and import and export houses in the two countries. The directory is classified under different heads and alphabetically arranged for quick reference.

**The Manufacture of Iron and Steel, Vol. I—Iron Production**, by G. Reginald Bashforth (Chapman & Hall Ltd., London), 1948, pp. viii+228. Price 21s. net.

THE AUTHOR HAS NEATLY COVERED IN FAIR detail practically all the aspects of iron production. It should prove extremely useful to students of metallurgy. A typical feature of the book is the list of references relating to the voluminous data condensed in the text.

The contents are well laid out and balanced. Reference to Bihar and C.P. haematite iron ore belts has not been made in relation to India's iron ore deposits, although Mysore magnetite deposits have been referred to. It may be pointed out that the Bihar and Bengal iron ores form one of the richest iron ore deposits of the world. Further chapters deal effectively with the technical processes involved in iron and ferro-alloys' production.

The language of the book is precise and the information contained should prove useful both to students as well as to industrialists for reference and study. The technical details of the processes are dealt with comprehensively. The pages on electric reduction of pig iron, however, lack adequate reference to the Swedish practice. This may be ascribed perhaps to lack of space.

The book is a very useful addition to textbooks on iron production.

B. R. NIJHAWAN

# Developments in Vegetable Oil Extraction

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M. N. KRISHNAMURTI

*Volkart Brothers, Bombay*

**D**URING the past 25 years, the vegetable oil extraction industry has undergone a considerable change with respect to processes, equipment and practices. As a result, the industry has now changed over from pressure extraction with expellers or hydraulic presses giving a comparatively low yield of oil, to processes based on advanced knowledge of chemical engineering principles yielding higher percentages of oil. This transition has been, for the most part, from an industry requiring a very modest capital investment in simple equipment to an industry representing a substantial investment in complicated chemical engineering equipment and auxiliary processes. Consequently, this higher yield of oil obtained from seeds or pressed cakes leaves the meal with a higher percentage of protein concentrate which provides higher nutritional value from the point of view of cattle feed. Moreover, the meal produced can be used with complete satisfaction for human consumption.

Soya-bean or, for that matter, any oil seed with an original oil content of less than 30 per cent is seldom pressed by mechanical expellers in America or on the Continent. For seeds with an oil content exceeding 30 per cent, such as groundnut, a combination of expeller-solvent extraction operation is recommended. Most of the earlier installations in Europe and the United States adopted this process for extracting oil from soya-beans, for this was a comparatively low oil-bearing seed with 20-22 per cent oil content, and mechanical extraction was expensive, as the oil content in the residual cakes could not be reduced to less than 4.5-5 per cent. The solvent extraction process, on the other hand, yielded a cake containing only 0.5-1 per cent oil. Later installations have demonstrated the benefits of solvent extraction of oils from pressed cakes obtained by expellers from groundnut which contain 8-10 per cent of residual oil, and there were several mills operating in Germany employing expeller cum solvent extraction processes before the war.

## **Process**

The raw materials, whether they be nuts, beans or pressed cakes, with an oil content not exceeding 30 per cent, are treated in the preparatory milling equipment, converting them into flakes of suitable size and thickness with moisture regulated to 12 per cent and temperature lower than the lower boiling limit of the solvent. The flaked material then enters the extractors where it contacts the solvent, and oil is removed by washing and diffusion. The mixture of oil and solvent termed "miscella" is clarified by filtration and, subsequently, by centrifuging. The mixture is then distilled to remove the major part of the solvent, and finally stripped in rectifying columns to remove the last traces of moisture and solvent. The solvent-free oil is drawn off the base of the stripping column and pumped to storage tanks. The solvent vapour from the evaporator and distilling columns is condensed and water removed by decantation before returning the solvent to the extractor. The solid residue from the bottom of the extractor enters dryers where the last traces of solvent are driven off by heat and condensed. The solvent-free meal is conditioned by toasting followed by cooling, sifting and bagging in the warehouse.

## **Equipment**

A solvent extraction plant consists mainly of the extraction apparatus, evaporator units, distilling and rectifying columns, "miscella" storage tanks, filters, meal dryers and accessories. The Anderson extraction plant discussed here is of American design. It has an extraction column embodying the patented settling chamber device for "miscella" fines and a discharge device which is a modification of Anderson's expeller application. The settling chamber device consists of a number of vertical tubes through which the "miscella" flows. A disc in which there is a triangular opening covers the top of the tube permitting the intermittent discharge of "miscella" from each tube as the disc revolves. While the "miscella"

in each tube is stationary, the fines settle into the lower part passing out through the bottom of the column with the flakes of the material.

One of the latest models of the continuous solvent extraction plant is now operating near Wooster, Ohio, U.S.A. The plant does not require any elaborate factory installation. It is placed in the open with no building of any sort, thus eliminating explosion hazard inherent in housed equipment, especially when hexane is used as a solvent. The extraction unit is located 50 ft. away from the preparation building. It is thoroughly insulated and weather-proofed. The equipment is largely pre-assembled and compact, different pieces of the unit supporting one another so that no special building or frame-work is needed except for a concrete footing and foundation slab.

#### Remote Control

The outdoor extraction unit is remotely controlled from a room in the preparation department by one operator. Push-button station, complete with pilot lights for all equipment, are grouped in accordance with the operating sequence. All principal equipment of the extraction unit are electrically interlocked. It is, therefore, necessary to start the plant in proper sequence and interruption to any unit shuts down equipment ahead of it. The grouping of all instruments at one location gives the operator very close control and allows finer co-ordination of the entire operation.

The vapour scrubbing device located between the main condensers and the solvent recovery dryers afford protection to the main condenser by separating dust that might come off with the vapours from the solvent recovery dryers which, if allowed to enter the condenser unit, affect the efficiency of the condensers, thus increasing the solvent loss. The equipment is designed with two main condensers, each having a sufficient capacity to take care of the entire plant in case any cleaning operation should ever be necessary while the extraction unit is in operation. The evaporator, vaporizing chamber and stripping column are built in one column with the further advantage of using waste heat from one element as the heat source for the other saving in steam, piping and insulation. This solvent extraction unit is elegant in design, practically trouble-free, and requires less labour and space.

#### Solvents

The selection of the most suitable solvent depends on the following considerations:

The solvent desirable for vegetable oil extraction should take up the oil selectively and dissolve a minimum quantity of wax, resin and colour; otherwise this may result in a dark crude oil involving high refining costs. The solvent should be low boiling with a narrow boiling range and chemically inert, leaving no odorous residue. Among the solvents now in use are alcohol, carbon-disulphide, chloroform, ethyl ether, methyl chloride, acetone, benzene and petroleum ether. In Europe, the common solvent recommended by the manufacturers of equipment is benzene, b.p. 70°-90°C. This has an aromatic flavour and, in order that no aromatic smell is present in the products, the meal is de-aerated in the process. In the United States, where the continuous system is followed, the solvent recommended is normal hexane, b.p. 62°-65°C. Both the solvents have good selectivity, excellent stability and low evaporation residue, but they are inflammable. The Japanese have reported the use of alcohol as a solvent for the batch extraction of soya-beans in a plant at Darien, Manchuria. They have published data on the miscibility of soya-bean oil in various ethanol water ratios. The data shows that absolute alcohol (99.52 per cent) is miscible in all proportions with soya-bean oil above 67.3°C. At the lower alcohol concentration, 95-92 per cent, the oil solubility is greatly reduced, and complete miscibility is not attained even at the boiling point, 78°C. However, the solvent proportion of 95 per cent alcohol may be brought into a practical solvent range by operating under sufficient pressure to bring the temperature to 90°C.; obviously, the separation of the miscella from the oil-bearing material must also be carried out under pressure. Both 95 per cent alcohol and absolute alcohol remove water from oil-bearing meal at the moisture content usually employed in solvent extraction, and quickly becomes diluted to such an extent as to be of no further use as an oil solvent unless rectification and dehydration be used to remove the water. To minimize the dilution of alcohol, the Japanese recommended that the moisture content of the oil-bearing material be reduced. In fact, under the ordinary conditions of drying, the moisture content of soya-beans is often reduced below 3 per cent and an actual improvement

takes place in the efficiency of alcohol as an oil solvent.

### Solvent Loss in Tropical Climates

Solvent loss is the chief consideration in all solvent extraction units. Various equipment manufacturers have adopted variations in design in order to see that the solvent loss is kept within a reasonable limit. In all the designs the local considerations as to air temperatures, humidity and temperature of cooling water available, physical condition of raw material while entering the extractors, have been given careful consideration. The average summer temperature of cooling water available in this country is about 90°F., while the temperature of water on the Continent and in the United States is only 65°F. To cool the temperature of water from 90°F. to 65°F. requires a big refrigeration unit, which means the process is uneconomical. For this the condenser should be designed for a high flow rate of cooling water; if the solvent loss turns out to be high, the vent gases can be directly led into a dephlegmator unit where it can be cooled by a small refrigeration unit and condensing the solvent vapour sufficiently well below the vapour dew point.

### Cost Estimates per 24-hour Day Basis for Solvent Extraction of Groundnut Pressed Cakes

#### Raw Materials—

	Rs. a. p.
50 tons oil cake of approx. 10% oil content at Rs. 130/- per ton	6,500 0 0

#### Utilities—

(a) 70,000 lb. process steam at 140 lb. per sq. in. corresponding to 4.6 tons coal at Rs. 30/- per ton	138 0 0
(b) 13,750 gal. make-up water requirement, i.e. 5% of the total water circulated at Re. 1/- per 1,000 gal.	14 0 0
(c) 1,500 kWh. electricity at ½ anna per unit	47 0 0
(d) Solvent hexane based at 1% loss equivalent to 120 gal. per day (Price Rs. 3/- per gal.)	360 0 0
	185 0 0

#### Labour & Supervision—

1 Technical manager—Rs. 900/- per month	
3 Solvent plant operators—Rs. 1050/- per month	
3 Assistant solvent plant operators—Rs. 600/- per month	
3 Mechanical engineers—Rs. 900/- per month	
3 Asst. mech. engineers—Rs. 450/- per month	
3 Boiler engineers—Rs. 600/- per month	
6 Preparatory plant tenders—Rs. 600/- per month	
Depreciation at 10% on Rs. 10,00,000 per year	335 0 0
	TOTAL PER DAY
	7,579 0 0

#### Credit for Sale Proceeds

4 tons of oil based at 8% recovery and selling at Rs. 1,125/- per ton	4,500 0 0
46 tons oil cake at Rs. 100/- per ton	4,600 0 0
	TOTAL
	9,100 0 0

Profit per day Rs. 1,521/- (45% over investment)

#### REFERENCE

1. BEEKEL, A. C. : *J. Amer. Oil Chemists Soc.*, 1948, 25, 7.

## Non-Technical Notes

### RUBBER-LINED EQUIPMENT FOR CHEMICAL INDUSTRIES

RUBBER OFFERS A HIGH DEGREE OF RESISTANCE to many corrosive liquids and, therefore, rubber-lined equipment is finding increased applications in the processing of corrosive substances in chemical industry. Rubber can be made to adhere strongly to materials like iron, steel, wood and concrete.

The usual practice followed in industry for the rubber-lining of equipment is to make suitably prepared sheet rubber adhere to the basic material by the use of adhesives. Rubber latex has been used only on a limited scale in rubber-lining.

Methods for using latex for the rubber-lining of equipment have been developed. The standard procedures have been modified and adapted to suit Indian conditions.

Actual working details of the processes are covered by Indian patents, and the "know how" will be made available to parties taking up commercial production according to these patents.

### Process

The methods employed for rubber-lining are:

- (i) electro-deposition of rubber; and
- (ii) ionic deposition of rubber.

(i) *Electro-deposition*—This method is particularly suited for small metallic equipment of complicated shape. The article to be lined is first subjected to a pre-treatment. It is then immersed in a properly compounded latex bath from which rubber is anodically deposited on the article.

Methods have been developed to improve the mode of preparation of the latex bath, and also to improve the quality of the rubber-lining obtained.

(ii) *Ionic Deposition*—This method can be used for rubber-lining chemical equipment of large size.

Films of suitably compounded latex are made to adhere to pre-treated surfaces of articles employing the principle of ionic deposition. A wide range of rubber compositions to suit the requirements of individual industry has been developed.

In addition to the general application of this method, special mention may be made of the fact that wooden boxes can be rendered suitable for use as storage battery containers by lining with rubber.

### Raw Materials

The raw materials and chemicals required for these processes are easily available in India.

### Equipment

The process can be advantageously exploited by existing rubber-goods manufacturers. Most of the equipment required would be available with them. For general guidance, a list of equipment needed is given below.

*Approx. cost*

	Rs.
(1) One porcelain ball mill (without motor; approx. capacity, 2 gal.)	800
(2) One mixer (without motor; to be fabricated locally; approx. capacity 2 gal.)	300
(3) One vulcanizer (to be constructed locally)	1,000
(4) Three motors	1,200
(5) Three rubber-lined, mild-steel tanks; (i) zinc plating, (ii) rubber deposition, (iii) washing, each 3'×3'×4', @ Rs. 200 each (rubber-lining may be done locally)	600
(6) One air compressor (with motor and shaft system)	2,500
(7) One spray gun (medium size)	200
(8) Building with 2 rooms, 13'×15' each	5,000
<b>TOTAL</b>	<b>11,600</b>

### Cost Sheet

#### *Electro-deposition Basis* —

Manufacture of 50 sq. ft. rubber-lined area per day of 8 hours:

#### *Working Capital* —

	Rs.
1. Power at 1 anna per unit, 400 kW. for zinc plating and 1,425 kW. for rubber-lining of 50 sq. ft.	114·06
2. Rubber chemicals including pure zinc anodes and water	20·00
3. Depreciation	1·00
4. <i>Management &amp; Labour</i> —	
1 Assistant chemist at Rs. 250/- p.m. (24 working days)	} 25·4
4 Coolies at Rs. 90/- p.m. each (24 working days)	
5. Miscellaneous	5·00
<b>TOTAL</b>	<b>165·46</b>

Estimated cost to rubber-line 1 sq. ft. area: Rs. 3/4/0.

#### *Spraying Basis* —

Manufacture of 100 sq. ft. rubber-lined area per day of 8 hours:

	Rs.
1. Power at 1 anna per unit (1,200 kW.)	75·00
2. Chemicals including water	40·00
3. Depreciation	1·00
4. <i>Management &amp; Labour</i> —	
1 Chief chemist at Rs. 350/- p.m.	} 43·00
6 Coolies at Rs. 90/- p.m. each	
1 Steno-typist, at Rs. 150/- p.m. (24 working days)	
5. Miscellaneous	3·0
<b>TOTAL</b>	<b>162·00</b>



# NOTES & NEWS

## Mold Amylase by Submerged Fermentation

THE MOLD-BRAN METHOD AND the amylo method are two procedures normally employed in commercial practice for the production of mold enzymes. From the standpoint of distillery operation, however, both these methods possess certain disadvantages. In mold-bran production, considerable labour, processing space and equipment, as well as a cereal bran substrate are required, while in the amylo process the entire mash volume must be pre-liquefied and aerated under pure-culture conditions. As a result of a survey conducted at the Northern Regional Research Laboratory, Peoria, U.S.A., a few molds have been isolated which are capable of producing starch-hydrolysing enzymes when cultured under submerged conditions (*Ind. Eng. Chem.*, 1949, **41**, 100). Laboratory studies showed that culture liquor from one strain, *Aspergillus niger* NRRL 337, completely replaced malt when added to corn mashes at the rate of 10 to 20 per cent of the final mash volume.

Pilot-plant experiments were conducted to obtain data on the following points: extent of enzyme production in deep tank fermentations under varying rates of aeration; the relationship between enzymatic potency of the mold culture liquor and the quantity required to replace malt completely; alcohol yields obtainable by the continuous use of fungal amylases for the saccharification and fermentation of corn; and design and cost of operation of submerged process for production of fungal enzymes.

The results showed that saccharification was satisfactory and alcohol yields were comparable to those obtained with malt when mold culture liquor equivalent to 6 to 10 per cent of the final mash volume was used. The cost of amylase produced by this process is estimated at 6.06 cents per bushel of grain processed, as compared with 12.1 cents in the case of malt.

Corn meal, starch, molasses, maltose and commercial glucose were found to be suitable sub-

strates. Corn-steep liquor, stillage, soya-bean meal and animal tankage were found to be good sources of protein. Calcium carbonate appeared to be beneficial in maintaining optimum pH levels during fermentation. Sterile air is supplied at a rate of 0.25 vol. per vol. of medium per min. Mechanical agitation was also employed. Malt stillage can be replaced satisfactorily with mold stillage as the substrate for mold cultivation and can be used continuously.

## Concentration of Latex

A PROCESS FOR THE RAPID CONCENTRATION of Type III GR-S latex is described (*Ind. Eng. Chem.*, 1949, **41**, 156). The process, which is an improvement on the German method of latex concentration, permits a more rapid concentration of GR-S latices to solids content considerably higher than were achieved by the German method. The process involves gelation by the addition of an electrolyte (sodium chloride) and cooling, and subsequent processing of the mix to yield a fluid and rapidly filterable mass.

The sample of GR-S latex to be concentrated is analysed for soap and rubber content, and if the soap content is less than 0.25 milliequivalent per gm. of rubber, a solution of potassium soap of K. wood rosin is added to bring the soap content to 0.28 to 0.29 milliequivalents. The pH is maintained between 10.5 to 11.5 by the addition of requisite quantity of sodium hydroxide solution. In the next stage 3N. sodium chloride is slowly added to the latex to give a final concentration of approximately 0.24N. sodium chloride in the final volume of latex and salt solution. It is essential that all the additions to the latex are made with agitation. The salt latex-salt mix is cooled as soon as possible after preparation by surrounding the vessel with an ice-bath. During cooling the mass should be agitated continuously. The cooling is continued through the gelled stage and the subsequent thinned stage until the temperature of 5°-8°C. is reached. The material is now ready for filtration

through a double-walled Buchner funnel, pre-cooled by ice-water. It is preferable to over-concentrate the latex and dilute it back to the required concentration. The filter cake is transferred to a closed container, allowed to warm up to room temperature and blended by agitation.

Exceptionally stable latices of 60 to 70 per cent solid contents and above can be obtained by this method, and they exhibit all the characteristics of a good latex. Pilot-plant studies have shown that the process works well in large-scale practice, and suitably prepared Type III GR-S latex can be filtered readily on an Oliver filter.

## Physical Constants of Leather & Collagen

INVESTIGATION CONDUCTED AT the National Bureau of Standards, U.S.A., on leather has added fundamental data on the physical and physico-chemical properties of leather and collagen (*J. Franklin Inst.*, 1949, **247**, 155). A knowledge of these constants may permit utilization of leather in applications requiring specific properties possessed by leather but as yet unknown. The effect of tannage and other treatment on values of such constants may yield valuable information concerning the structure of collagen and the mechanism of tanning.

Since leather is a porous material containing as much as 60 per cent void space, most measurements of physical properties of the leather substance present obvious difficulties. One method of solving this problem lies in completely filling the leather with a liquid and ascertaining the properties of the liquid-leather system. This approach has been used at the Bureau's leather laboratory to obtain data on the cubical expansivity of leather. Using water as the confining liquid, dilatometric measurements of a water-leather system were made and compared with measurements on a similar system containing water alone. As a result of these measurements it was found that the average expansivity of 540 × 10<sup>-6</sup>/°C. applied for collagen and all leather except chrome-vegetable leather. The average expansivity of the latter tannage is 340 × 10<sup>-6</sup>/°C. These averages have reproducibilities as indicated by standard deviations of 13 × 10<sup>-6</sup>/°C. and 37 × 10<sup>-6</sup>/°C., respectively.

Leather, when heated in water above a well-defined temperature, depending on tannage and other factors, undergoes shrinkage. The temperature at which shrinkage begins is defined as the shrinkage temperature and is considered a measure of the efficiency of the tanning process — the higher the temperature, the more thorough the tannage. During the dilatometric studies it became evident that the apparent shrinkage actually represented an increase in real volume. This increase in volume amounted to approximately 1 per cent and was irreversible in character although the, purely thermal expansion was found to be nearly completely reversible. It was also observed that this increase in real volume occurred very slowly at temperatures well below the shrinkage temperatures of the specimens found by regular test methods. This indication that shrinkage was a rate process was substantiated by a rate measurement which indicated that the expansion occurring during shrinkage follows a first order process.

### Improved Guayule Rubber

THE IMPROVEMENT OF GUAYULE rubber by shrub retting has been recently investigated (*Ind. Eng. Chem.*, 1949, **41**, 346). By retting, the resinous contaminations of the crude rubber are reduced to about half the quantity present in unretted guayule rubber, tensile strength is increased by 50 per cent and some other physical properties are improved.

For rapid retting, shrub moisture content is to be maintained between 35 to 55 per cent on a wet weight basis. Temperatures should never exceed 60°C.; for unmixed or occasionally mixed shrub, the safe limit is 55°C.; for continuously mixed shrub, temperature should be maintained from 25°C. to 35°C. All degrees of mechanical reduction from whole shrub to finely crushed shrub may be used. More finely cut or crushed shrub will ret more rapidly, but is suitable only for mixed rets, while coarsely cut or whole shrub may be used only for unmixed rets. The natural pH of the shrub is satisfactory for initiating the retting process.

The methods of retting, which have been investigated, fall into 2 categories: those not involving any disturbance of the shrub during the retting period, and

those involving occasional or continuous mixing. In the first category, floor retting and bale retting gave satisfactory retting. Depth of piling must be correlated with the degree of mechanical reduction and the retting time will be from 1 to 2 weeks. Whole shrub can be retted in the bale following parboiling, defoliation, and rebaling, with a retting period of approximately 2 weeks. In the second category are included floor retting with occasional mixing, and drum retting with continuous mixing.

Retting on the floor with periodic mixing (4 to 6 times a day) requires that the shrub be rather finely cut (0.5" followed by crushing), the depth of piling be closely correlated with the size of cut, and the retting period be adjusted to 6 or 7 days. Shrub cut to 0.5" on a rotary cutter, piled 10" deep, and retted for 7 days has been found to give excellent improvement in quality and optimal recovery of rubber. Temperatures may range up to 60°C. or even slightly above in parts of the shrub, but the moisture content should be kept in the range of 45 to 50 per cent by adding water as needed. Drum retting provides the neatest and easily controllable method of retting. Aeration, moisture, and temperature can be kept under continuous control at the desired level, and very finely reduced shrub can be used without the danger of anaerobiosis. Continuous and uniform maintenance of optimal conditions permits a reduction of the retting period to 3 days.

### Plating of Plastics

A PROCESS, SIMILAR TO ELECTRO-deposition of rubber films from aqueous latex, has recently been developed at the Columbia University for the production of plastic-coated products (*Ind. Eng. Chem.*, Feb. 1949, 15A). Sheets of uniform thickness are obtained and complex "hollow" shapes can be "plated" from a plastic emulsion and can be conducted in ordinary plating tanks. However, this technique produces a film less than a millimeter thick and building up of thicker films is time-consuming and does not produce a uniform sheet.

The latices used may be prepared from any plastic material which will form a stable aqueous emulsion containing 55 per cent solids. A plating tank divided

by a thin barrier of porous porcelain is used. The latex is placed in the anode compartment of the plating tank, the cathode chamber being filled with a 50 per cent sodium chloride solution. The porous divider is necessary to prevent hydrogen evolved at the cathode from migrating to the anode and interfering with the deposition of the plastic. Copper cathodes may be used since the cathode does not dissolve into the electrolyte.

The choice of the anode is important. Nickel, which has proved impracticable for the electro-deposition of rubber, is especially effective for plastics. Zinc, which is widely used for rubber deposition, destabilizes vinyl type of plastics. Passivated metals such as aluminium tend to form a spongy film.

Metal-free films can be formed by depositing the plastic on a porous barrier, or mould of alundum or porous porcelain. Mercury, used as an anode either behind the barrier or contained in the body of the mould, forms insoluble anode products which do not penetrate the barrier.

If the coating composition contains a plasticizer, the film is coherent and firm but does not attain its maximum tensile strength until it has been baked for half an hour at 80° to 105°C. Unplasticized latices produce soft, crumbly films but these can be cured at a temperature of about 150°C. for an hour to produce a fused, hard sheet.

The tensile strength of the cured films is superior to that of dipped or sprayed film and approaches that of moulded products. This property could be enhanced by using emulsifying agents in the latex which would either volatilize or combine with the polymer during heat treatment.

The power requirements for electrolysis are small. When depositing on metal anodes, 2 to 4 v. are required, about one-fifth to one-tenth the potential difference used in the electro-deposition of rubber, deposition efficiency is about 2,500 gm. per faraday and at rates as high as 100 kg. per kW./hr. Best efficiency is obtained at rates between 10 and 20 kg. per kW./hr. with current densities of about 20 ma. per sq. cm. Under these conditions a 0.125" film can be deposited in about 15 min. on metal electrodes. Porous forms reduce the rate of deposition by about 15 per cent.

### Optical Glasses

A NUMBER OF NEW OPTICAL glasses, their properties and applications have been described (*Nature*, 1949, 163, 412).

With the introduction of barium crown glass, many new types of photographic lens became possible. This glass greatly improved all lenses of the air-spaced type. In 1934 samples of unusual glasses with characteristics  $n_D$  in the region of 1.85 and  $\nu$  the reciprocal relative dispersion of 47.0, were in existence.

Lanthanum is very soluble in boric acid, and its contribution to higher refractivity without increase of dispersion is remarkable. The oxides of tantalum, thorium and tungsten are soluble in the lanthanum borate base glass in amount up to 35 per cent. These new borate glasses are very stable and hard. They are harder than flints, stable to the atmosphere, and amenable to optical shop practices of moulding, grinding and polishing.

Early in the development work it was found that the rare-element borate glasses were extremely corrosive to all known pot refractories and platinum was employed for the actual production of these glasses. This was justified on the basis that no platinum would be lost by contamination, and that the glass, once homogenized, could be poured in its entirety, free from striae, into a single slab or into cast shapes without striae, seed, bubbles or other defects usually attending a glass made in a refractory pot. The first of the new glasses to be made had a refractive index of 1.7445 and a reciprocal relative dispersion of 45.8. The glass was slightly yellow, but further work established the origin of the yellow colour, and finally glasses were produced as colourless and homogeneous as any other optical glass.

Since 1940 the types of these glasses in production have been extended, and at the present time the following seven types are being made:

	$n_D$	$\nu$
EK — 110	1.69680	56.2
— 210	1.73400	51.2
— 310	1.74500	46.4
— 320	1.74450	45.8
— 330	1.75510	47.2
— 450	1.80370	41.8
— 448	1.88040	41.1

All these glasses contain thorium, and in the case of folding

camera, in which the lens may rest for a long time in close proximity to film, the radio-activity of the thorium may be a disadvantage. A thorium-free equivalent of EK — 320 is available.

The most useful of these new glasses were flint glasses containing titanium oxide and fluorine in addition to silica. The best glasses lie in the region 45 per cent silica, 28 per cent titanium oxide, and 27 per cent sodium fluoride, ranging in optical properties from  $n_D$  1.65/ $\nu$  29 to  $n_D$  1.58/ $\nu$  36.6. The glasses can be moulded, are very resistant to tarnish, and are easily fabricated by the usual optical methods.

The fluosilicate flints are almost as useful to the optical designer as the high-index glasses, since they extend the possible difference between crown and flint refractive index and between crown and flint dispersion. With these glasses, three-element lenses have been designed to give even better performance than the usual four-element types. K. H. Sun of the *Kodak Research Laboratories* has successfully produced novel mixtures in some 20 quite different glass fields including fluo-borate, fluo-germanate and fluo-phosphate systems.

An interesting group of glasses are those containing no oxides and composed entirely of fluorides. These glasses show the characteristic low refractive index and extremely low dispersion previously available only in fluoride minerals. The refractive index in most of the glasses is approximately 1.38 to 1.39 and the  $\nu$ -value, 100. Moreover, the glasses are transparent to below 300  $\mu$  in the ultra-violet and to 5  $\mu$  in the infra-red, so that they may be very useful in the making of instruments requiring optical transparency over a wide range of wavelength. Difficulties are met with in the production of these all-fluoride glasses, but it is possible that these difficulties may be overcome in the near future.

### Failure of Railway Materials

WITH INCREASING SPEEDS, heavier revolving masses and greater overall weights of main-line locomotives, the need for sound materials both in the track and rolling stock has become of paramount importance. Fatigue and other causes of

fracture in engineering materials and parts have been widely investigated in recent years, and the root causes are now fairly well understood. So far as railway track materials are concerned, transverse fissures in rails at one time were a source of frequent discontinuity in service, although such occurrences have now been greatly reduced by improved methods of manufacture. As regards rolling stock, the highly stressed portions of locomotives are susceptible to fracture arising from fatigue cracks usually originating in faulty design or due to indifferent machining and finishing of surfaces.

The failures of rails through transverse fissures were first observed in the United States, where, up to 1932, no fewer than 65,281 cases had been reported, representing an average of 6.14 failures per 100 miles of track per annum. The true transverse fissure starts in the railheads, developing from a tiny shatter crack or hair-line crack and gradually spreading upwards and outwards until the unaffected sectional area is so weakened that it suddenly fractures transversely under a passing wheel load. The origin and development of hair-line cracks in the sectional thickness of steel is believed to be due to intergranular weakness derived from factors associated mainly with steel-making, fabrication and half-treatment of the product. Data contained in the technical literature is not unanimous in attributing this phenomenon to any well-defined set of conditions. It is, however, understood that steel-making conditions involving the solubility of gaseous elements like hydrogen, oxygen and nitrogen in steel, the conditions of deoxidation in final processes of steel-making, teeming conditions and, last but not the least, factors associated with the heat treatment and fabrication of the product particularly in its final rate of cooling, all play a part in bringing about a state of intergranular rupture in the finished product. Unfortunately, the Sperry method is not sensitive enough to detect hair-line cracks themselves so that rails so affected cannot be sorted out and scrapped at the mill; the rails must be in service long enough for the original hair-line crack to develop before its presence can be magnetically detected. The Sandberg controlled cooling process has consi-

derably reduced the risk of hair-line crack formation in rail-heads through controlling and retarding the rate of cooling of the product in a temperature range of 200°-400°C.

Published literature on this phenomena does not record occurrence of fracture in railway tyres. A number of other cases of transverse-fissure failure in tyres have been examined, each tyre steel conforming to standard specifications in respect of chemical and physical tests and free from metallurgical abnormalities. In a few cases the tyres that failed were fabricated from the same cast, which is in itself significant (*Iron & Coal Trade Rev.*, 1949, 158, 6).

### A New Austenitic Alloy

A NEW ALLOY FOR GAS TURBINE blades has been developed by a British firm. Known as G32, it is an austenitic alloy having a cobalt base and other main ingredients are nickel and chromium. It has also a small percentage of niobium.

G32 has been derived after 600 experimental melts. Further detailed tests are expected to show that it will be a worthy successor to G18B (hitherto predominant in the field of gas turbine discs). While, like G18B, primarily evolved for turbine engines, G32 has been shown to have other uses and is being produced for furnace parts such as cradles and structured members (*BIS*).

### Calcium Carbonate Extender Pigments

AN IMPORTANT CLASS OF Extender pigments is the calcium carbonate class. A study of 5 calcium carbonate varieties, viz. dry ground calcite, wet ground calcite, "Witcarb regular" (medium-fine), fine-particle calcium carbonate and "Witcarb R" (ultra-fine) have been studied and the effect of particle size on the optical properties of the dried paint films and on the rheological properties of the fluid paint has been investigated (*Ind Eng. Chem.*, 1949, 41, 390).

The carbonates cover a particle size range from 0.5 to 3.9 microns, corresponding to specific surface areas from 0.32 to 0.55 sq. m. per gm. The oil absorption values increase with surface area, showing that surface area rather than dif-

ferences in the nature of the surface is the dominant factor. Formulations obtained with low acid value linseed oil also indicate that the particle size is primarily responsible for the variations observed in their properties.

Hiding power, lightness and gloss of a paint film increase regularly with decrease in particle size of the carbonate. Improvement in the above properties by the ultra-fine particle precipitated calcium carbonate appears to be due to improved dispersion of the binding pigment brought about by the calcium carbonate particles which may function as grinding or anti-flocculating agents.

Decreasing particle size of the calcium carbonate extender is accompanied by a regular increase in consistency and yield values of the paint.

### Potato Farming in the United States

WITHIN RECENT YEARS, THE average yield of potatoes per acre over the potato-growing territory in U.S.A. has more than doubled. In 1948 the average for the United States reached 204.5 bushels per acre. For these greatly increased yields, 4 developments are chiefly responsible: breeding of new varieties, better fertilizers, better sprays and the wide availability of certified seed stock.

Most important in the prevention of the spread of disease is the development of disease-resistant varieties of potato plants. Since 1932, under the national potato-breeding programme of the U.S. Department of Agriculture, at least 34 new varieties of various virtues have been distributed to the growers.

Of second importance in the control of disease at planting time is the programme of inspection and certification. A corps of government experts have the task of examining seed potatoes at various stages of their field growth and during the harvest, to the end that all seed potatoes they certify as acceptable will have, at most, only a tiny percentage of diseased plants.

The practice of crop rotation has been found to be a very effective additional control measure. If one or two crops of some other vegetation not susceptible to potato diseases is planted after the potato crop,

the disease organisms, or a large majority of them, die of starvation before they can affect the potatoes.

The fourth control measure, which is proving effective against the rhizoctonia, is the disinfection of the seed, either the whole tubers before they are cut, or the cut seed pieces. One recommended adjunct to disinfection is the suberization of seed pieces. This treatment causes the pieces to form over themselves a layer of cork cells which prevents their rotting in the moist ground. Since this method consists only of putting the seed pieces in a room with the proper humidity and heat for a short time, it is becoming a widespread and very rewarding practice.

The fifth control measure, now very common in areas where late blight and beetles harm huge numbers of the plants, is the spraying or dusting of the potatoes with various chemical mixtures. Among them is a DDT mixture which seems to have splendid effect on flea beetle, Colorado potato beetle, and leaf hopper.

In successfully raising seed potatoes which meet the stringent seed certification standards, farmers have taken advantage of a procedure which requires immensely more work at planting time but makes the rest of the growing and inspection of seed tubers much easier and safer. This is the "tuber unit" system of planting. All the seed pieces from single tubers are planted side by side, and a space is left at the end of each set of seed pieces before another tuber's pieces are planted. A corollary of this procedure is that the potato seed must be cut on the field at the time of planting, and after each set of seed pieces is planted, the knife used for cutting must be disinfected.

A good deal more care is necessary for this method of planting than when the farmer merely fills his seed hopper with pieces and starts off on his tractor. But once planted to tuber units, the crop becomes highly controllable.

Another necessary step in the programme of constant vigilance against potato diseases is "rouging" where diseased plants are removed by a close physical examination before they have chance to infect whole areas (*USIS*).

## Tropic Proofing

DURING THE WAR YEARS MUCH study and experiment was devoted to tropic proofing and to the packing of the equipment of every type used by Allied forces in Burma, Africa and other tropical theatres of war. The Directorate of Chemical Research and Development, Ministry of Supply, U.K., on the suggestion of the Department of Scientific & Industrial Research, have compiled in a booklet (H.M.S. Office, London; 1949, price 9d), the results of the extensive investigations of methods of protecting materials and equipment from deteriorative effects of storage and use in tropical climates. This publication is an up-to-date version of the wartime pamphlet published in 1945 and is now made available for general information of the civilian exporters to tropical countries.

The first 3 chapters in the pamphlet are devoted to methods of testing the efficiency of various treatments against fungal and insect attack. The remaining 8 chapters give information about the work done for the protection during storage and use in the tropics of various classes of materials and equipment, viz. plastics, rubber, leather, wood, textiles and cordage, paper and packing materials, paints and varnishes, lubricants and temporary metal preservatives and finally optical and electrical equipment. A list of Allied government publications is added as an appendix.

Brilliant sunshine, high temperature and humidity conditions and attack by molds and insects are the main causes of deterioration of the above type of materials and equipment in the tropics. Information relating to the choice of materials and the protective measures to be employed, including the use of appropriate fungicides and insecticides where necessary during use and storage, has been given under each head. The choice of any particular method of protection should generally be governed by a full consideration of all the factors likely to be involved and preferably after comparative trials. On the whole, the information contained in this pamphlet, although requiring to be supplemented by further details in many cases, is of immense practical interest

to manufacturers, traders and users of these materials and equipment in India.

The Defence Services in this country do already have a proper appraisal of the magnitude and importance of the problem of tropic proofing and have their own development organizations working on its different aspects, but it should be the task of the Indian Standards Institution to produce appropriate specifications for general adoption by the trade in India.

C. S. RAO

## Specifications for Radio Components

A SPECIFICATION DESCRIBING THE general conditions and procedure for durability testing of components for radio and other electronic equipment is published by the *Radio Industry Council*, U.K. The Specification No. RIC/11 is the work of the *R.I.C. Technical Specification Committee* in consultation with the *British Radio Equipment Manufacturers' Association*, the *Radio Communication & Electronic Engineering Association* and the *Radio Component Manufacturers' Federation* and is the first produced by the Committee. It covers approximately the same ground as the Inter-services Specification No. RC.S/11, but caters for the industry's requirements as distinct from those of the Services.

The object of the *Technical Specification Committee's* work is to produce a series of radio component specifications designed to ensure a high standard of reliability and performance for British components during use, transit and storage. Components are examined and their properties measured before and after they are subjected to the tests and their performances under test are laid down in the relevant component specifications. Components are classified under heads according to their ability to withstand extremes of temperature and humidity.

Supplementary design acceptance tests for vibration, salt atmospheric corrosion and mold growth are described.

Copies of the specification can be obtained (price 1s) from the *Radio Industry Council*, 59 Russel Square, London, W.C. 1.

## Television in Medical Training

FOR THE FIRST TIME IN EUROPE an audience of 200 medical

practitioners and students have been able to follow a surgical operation by television. This was on the occasion of the anniversary of Holland's oldest university at Leiden. Whilst an operation was being performed in the operating theatre of the hospital at Leiden, this was televised and projected on 2 screens of 1.30 x 1 m. set up in the lecture hall in another wing of the hospital.

Philips Eindhoven, who undertook the technical arrangement of this unique experiment in television after the most painstaking preparations, produced for the benefit of the audience a wonderful picture which demonstrated beyond all doubt the enormous possibilities of television as an aid to medical training.

## PATRA Research Laboratory

A NEW RESEARCH CENTRE WITH some 25,000 sq. ft. of floor area, to house the scientific and technological laboratories of the *British Printing, Packaging & Allied Trades Research Association* was recently opened at Leatherhead, in Surrey.

The PATRA laboratory stands on a 4.5 acre site and is designed and sited to allow further expansion. The building is approximately 200' long and 50' wide and of 3 floors. An outstanding feature is the technological laboratory which runs from the basement floor up to the top-storey floor level. This 20' of height and 2,000 sq. ft. of floor area will permit the installation of printing and packaging machinery. Letterpress and offset printing plants are installed here. Also at basement level are the laboratory for mechanical testing of packages, the process camera room and the electro-deposition and stereotyping department, and litho plate making section. To ensure constant working conditions, the atmosphere of the technological laboratory is controlled at 65°F. and 65 per cent relative humidity.

The package-testing laboratory is similarly controlled, but contains a variable humidity container conditioning the room with an operating range from 32°F. to 140°F. and 16 to 95 per cent relative humidity. The main laboratory here is equipped with rotating drums for testing the resistance of containers to rough handling with a drop-test apparatus.

Stores, boiler room and air-conditioning plant are housed in the basement.

At ground-floor level there is the entrance hall, flanked by a reception office and a committee room. Many of the scientific instruments used in PATRA's research work are designed and made in a workshop on this floor. The packaging research laboratory here contains a "make-up" table and equipment for making experimental packages and containers.

Paper and optics laboratories equipped for the study of the opacity, gloss and printing qualities of papers are also on the ground floor.

Study of the flow and deformation of inks and adhesives is carried out in the rheology laboratory (ground floor). The entomology, mycology and microscopy laboratories are also on this floor. Infection and damage to packages, books and papers by insects and molds, a major item on PATRA's research programme, is carried out here.

Scientific research on permeability in relation to packaging and packaging materials is the principal task of the packaging laboratory on the ground floor. Here, there is a variable high temperature room (for storage trials at elevated temperatures, designed to operate at controlled conditions between 65°F. and 140°F. and 10 per cent and 95 per cent R.H.) and a constant high temperature room (to operate at constant tropical conditions, 100°F. and 90 per cent R.H.).

Among the laboratories on the top floor of the new centre is that for surface chemistry where the desensitization of litho plates, contact angle measurement (for fundamental observations of the wettability of surfaces by oil and water), litho-plate-grain measurement, anti-corrosion processes for zinc and aluminium litho plates, and research into the influence of silica in desensitizing litho plates are among the tasks undertaken. On this floor is also the general enquiry laboratory for dealing with the workshop problems of individual members, and the laboratory dealing with absorption, oxidation and drying of printing inks.

Finally the building contains offices, a conference room, and the technical library—the key to which is a card index of over 100,000 items covering books,

periodicals, pamphlets, patents, catalogues and other matter of informative interest to the printing and packaging industries.

### Multi-purpose Laboratory in the Himalayas

THE PROPOSAL FOR SETTING UP of a high altitude laboratory in the Himalayas was first mooted by the Government of India in 1947. The proposed laboratory will have separate departments dealing with the study of snow, glaciers and their contribution to Indian rivers, astronomical and astrophysical observations, cosmic rays, biological and geological research, and research on the flora and fauna of the Himalayas.

An exploratory party, sent out last year to select a suitable location, recommended a spot near Kauri pass, especially the 12,458' Ghorosan peak area which can be easily connected by a motor road with Joshimath, lower down on the route to the plains. 500' below Ghorosan lies a vast meadow known as *Aulivugyal* (pasture land) which would be suitable for the scientists' colony. The establishment of a hydro-electric station and arrangements for water supply would not present any insuperable difficulties at this spot.

A second exploratory party is being sent again by the Government of India this summer to gather more complete data before undertaking the project.

### Institute of Radio Physics

THE WEST BENGAL PREMIER, Dr. B. C. Roy, laid, on April 21, the foundation-stone of a Calcutta University institute which will, for the first time in India, provide for a post-graduate study and research in radio physics and electronics. Built with a capital grant of Rs. 5½ lakhs and an annual recurring grant of Rs. 49,000 from the Government of India, the institute will be located close to the institute of nuclear physics of the University College of Science. It is proposed to start in the institute new research schemes on microwaves, both fundamental and applied to television.

In recognition of the research work carried out by the University, the Council for Scientific & Industrial Research of the Commonwealth of Australia

have offered on permanent loan a complete set of ionospheric equipment to the University. A field-station for this apparatus would be built in the agricultural farm-land offered to the University by the Government of West Bengal at Haringhata, about 35 miles from Calcutta.

### Indian Roads Congress — 13th Annual Session, 1949

THIS SESSION OF THE CONGRESS was inaugurated by the Hon'ble Dr. Srikrishna Sinha, Premier of Bihar, on 4th February 1949 at Ranchi. 237 road engineers from all over India attended the session which was a pronounced success.

In his address, the retiring President of the Congress, Shri S. N. Chakravarti, stressed the important part that roads play in all development programmes, and referred to the many valuable contributions made by the Congress for the improvement of roads in all its aspects such as specifications, standards and design. He emphasized the importance of research on roads and welcomed the establishment of the Central Road Research Institute at Delhi. He advocated the establishment of a non-lapsing fund for road construction, avoiding waste of public money, and rendering possible for road engineers to complete their road development schemes according to programmes based on availability of resources. The President laid particular stress on the need for developing village roads for the benefit of the common man and the country as a whole.

9 committees and sub-committees dealing with specifications and standards, bridges, highway layout, research organization, soil research, test track, etc., held their meetings during the session and many technical papers were read and discussed.

Provincial chief engineers held a meeting to discuss programmes of road development in the different regions of the country, and measures for co-ordinating the work on national highways.

The exhibition organized by the Indian Roads Congress attracted much attention. The Roads Organization of the Government of India, Provincial Governments, the Indian Roads Congress Office, and firms dealing with road materials and machinery took part. Charts, maps

and statistical tables of road development programmes in India, soils and bituminous products, models of different types of timber bridges, etc., were exhibited. The Military Engineering Services had a separate section in which machinery and modern plant for road construction were on show. Considerable interest was evinced by the delegates in the mobile laboratory for bituminous road work exhibited by the *Shalimar Tar Products Ltd.*, Calcutta, and in the road rollers (steam and Diesel type) exhibited by the Government of Bihar.

Mr. S. M. Gross, Vice-President of the *International Division of the American Road Builders Association*, gave an interesting lecture on "Modern Drainage Practice in Highway Construction" and described some of the methods of bridging adopted on American roads.

Informative and instructive scientific films on road-building and road-making machinery added greatly to the interest of the road engineers who had gathered in full strength at Ranchi to discuss technical topics and exchange views on problems confronting them in their professional work. The Congress concluded with visits to the Barakar Bridge on the Grand Trunk Road and to the *Tata Iron & Steel Works* at Jamshedpur.

R. K. N. I.

### Committees on Geophysical Studies

AN IMPORTANT STEP FOR THE development of the study of geophysics in India has been taken by the appointment of 2 committees by the *Central Board of Geophysics*.

One committee, consisting of 5 members, will draw up a planned programme for geophysical studies in Indian universities and the scientific departments of the Government of India. Another committee of 7 will prepare plans of work regarding physical and biological oceanography covering as large an area as possible of the Indian seas.

The *Central Board of Geophysics*, which was constituted in March this year and held its first meeting recently, has decided to approach the Government of India for 5 overseas scholarships for specialized training in geophysics this year. Government will

be also approached for sanction for the establishment of magnetic and seismographic observatories at Dehra Dun in the immediate future. The establishment of a Central Geophysics Institute in India for advanced study and research in the science is also among the *Board's* plans.

### National Planning Committee

THE NATIONAL PLANNING COMMITTEE, which was appointed by the Indian National Congress 10 years ago, has completed its work. The report, which was adopted at the Committee's final meeting, has, as its appendices, the reports of its 29 sub-committees. The sub-committees' reports are published in 26 volumes, collectively known as the *National Planning Committee Series*.

The Committee defined its own objectives, laid out a bold code of wide instruction for the guidance of these sub-committees and prescribed clear-cut norms and stages for the completion of its comprehensive task.

The reports of the 29 original sub-committees and 2 *ad hoc* sub-committees deal with the following: (1) manufacturing industries; (2) chemical industries; (3) population; (4) trade; (5) power and fuel; (6) women's role in planned economy; (7) rural marketing and finance; (8) labour; (9) river training and irrigation; (10) animal husbandry and dairying, fisheries and horticulture; (11) national housing; (12) education (general and technical); (13) communications; (14) engineering industries and industries connected with scientific instruments; (15) mining and metallurgy; (16) soil conservation and afforestation; (17) insurance; (18) land policy and agricultural, labour and insurance; (19) rural and cottage industries; (20) industrial finance; (21) crop planting and production; (22) currency and banking; (23) public finance; (24) transport services; (25) national health; and (26) national planning, its principles and administration.

### Central Silk Board

THE GOVERNMENT OF INDIA HAVE set up a Central Silk Board to provide for the development under central control of the raw silk industry in the country. The Board will advise the Central Government on all matters relating to the raw silk industry

including the import and export of raw silk. Among its other functions, the Board will devise means for improved methods of mulberry cultivation, rearing, developing and distributing healthy silk-worm seed, reeling and the supply of technical advice to filature and *charkha* reelers and help to improve the marketing of raw silk.

The Board will consist of 27 members with the Minister for Industry and Supply as Chairman. By the Act under which the Board is constituted, it is empowered to levy a cess on the silk industry.

### Agricultural Census

IN PURSUANCE OF A RECOMMENDATION made by the *FAO* for organizing an agricultural census in 1950 in member-countries, the Government of India have appointed a technical committee consisting of Mr. W. R. Natu, Economic and Statistical Adviser, Ministry of Agriculture, as Chairman and the following as members: Prof. P. C. Mahalanobis, Statistical Adviser, Cabinet Secretariat, New Delhi; Dr. P. V. Sukhatme, Statistical Adviser, Indian Council of Agricultural Research; Dr. V. G. Panse, Deputy Director (Research), Institute of Plant Industry, Indore; Dr. B. Natrajan, Economic Adviser to the Government of Madras; Mr. G. M. Sankpal, Director, Bureau of Economics and Statistics, Government of Bombay; Mr. K. Kishen, Statistician to the Department of Agriculture, United Provinces; Mr. N. C. Chakravarty, Department of Agriculture, Forests and Fisheries, Government of West Bengal; Mr. H. C. Kothari, Statistical Officer, Government of Jaipur; Mr. K. S. Sursinghji, Director of Agriculture, Government of Saurashtra; Mr. B. A. Bombawale, Secretary to the C.P. Government Land Records Department; and Mr. J. S. Sharma, Research Officer, Directorate of Economics and Statistics, Ministry of Agriculture (*Secretary*).

The terms of reference of the committee are: (a) to examine the forms of returns at present used by the different provinces and States for routine collection of data and evolve standard forms of returns; (b) to consider whether any special modifications are necessary in the scope of enquiry in respect of areas where the system of land survey or

record of rights is not introduced ; (c) to explore the directions in which the data collected through the census of agriculture can be usefully integrated with the data collected through the census of population.

#### Village Oilseeds Industry Board

THE INDIAN OILSEEDS COMMITTEE, at its 4th annual general meeting, recommended the setting up of a village oilseeds industry board and a training institute at Nagpur for the development of the village oil industry. The board named *The Ghani Advisory Board of the Indian Oilseeds Committee* will study the progress of the schemes sanctioned for *ghani* development and organization of co-operative societies. It will also supervise the activities of the training institute.

To encourage crushing of seeds in village *ghanis* the Committee requested the Provincial Governments to adopt a uniform policy with regard to exemption limits from sales tax, and suggested that the limit should be Rs. 15,000 in all the provinces.

#### The Metal Market Review— 1949 Annual Number

TO MARK THE COMPLETION OF the first year of its publication the *Metal Market Review* has brought out a well got-up and highly informative special annual number (Vol. 1, No. 25, 1st Feb. 1949). The issue contains a series of articles written by well-known authorities in various branches of metallurgical industries and metal trade.

The number is divided into 4 parts. The first part deals with topics of general interest pertaining to metal trade and non-ferrous metallurgical industry. The second part is mainly concerned with the ferrous metallurgy. The third part contains valuable statistics. The last part

is a useful "who is who" in metal trade and industry.

#### Grant for Andhra University

THE GOVERNMENT OF INDIA have sanctioned a grant of Rs. 770,000 to the Andhra University non-recurring, and Rs. 88,000, recurring, for the expansion and development of the Technology Department including the organization of a new branch in pharmaceuticals. The buildings are estimated to cost Rs. 380,000 and the equipment Rs. 390,000.

#### Grant for College of Technology, Madras

THE GOVERNMENT OF INDIA sanctioned for the Alagappa Chettiar College of Technology, Madras, a sum of Rs. 2,92,000 for the college buildings, and Rs. 1,13,000 for equipment. They have also sanctioned for the 2 departments of Chemical Engineering and Textile Technology an ultimate recurring grant of Rs. 40,000 and 32,000 respectively. The grants are made on the understanding that the number of admissions will be increased and the admissions will be made from candidates applying from different provinces.

#### Announcements

*Grant for Preparation of History of Chemistry in Ancient and Mediaeval India*—The Government of West Bengal have contributed a sum of Rs. 5,000 towards the preparation of the "History of Chemistry in Ancient and Mediaeval India" (incorporating Sir P. C. Ray's "History of Hindu Chemistry") undertaken by the *Indian Chemical Society*.

*The Indian Standards Institution* has issued the following 5 Draft Indian Standards for comment by those interested in them: Two standards on Mica; Coniferous Timber; Procedures for Testing Cotton Textiles and

Cordages for Resistance to Attack by Micro-organisms; and Draft Indian Standard Style Manual. These can be obtained by application to the Secretary (Publications), Indian Standards Institution, Block No. 11, Old Secretariat, Delhi.

Comments on these Standards should reach the Director, Indian Standards Institution, Block No. 11, Old Secretariat, Delhi, by June 30, 1949.

## REPORTS FROM STATES & PROVINCES

(continued)

cultivators as a winter crop in central districts and for conducting trials of Sea Island varieties on the west coast. The target is to evolve a strain of staple length 11/16" and 1 1/8" over the 4.5 lakh acre winter crop area, yielding 4 lakh bales and 6 lakh acres in the west coast districts under Sea Island cotton yielding 1 lakh bales of 1 1/4" to 1 1/2" staple length.

#### Cycle Factory

THE GOVERNMENT OF INDIA HAVE provisionally agreed to the establishment at Madras of a cycle factory by *Messrs B.S.A.* bicycle manufacturers with a capacity of 1 lakh cycles per year.

Permission has also been granted for the setting up by a British firm of a plant for the manufacture of earth-moving machinery used in building roads and dams.

## UNITED PROVINCES

#### Soil Laboratory

THE AGRICULTURE MINISTER OF the province opened the Regional Soil Laboratory at Aligarh on April 21. The laboratory, the first of its kind in India, will undertake soil survey for the benefit of agriculturists.



# Reports from States & Provinces

## MYSORE

### Manufacture of Telephones

THE TELEPHONE INDUSTRIES Ltd., Bangalore, has gone into production and is assembling and finishing telephone instruments at the rate of 300 per week.

The factory is now housed in 2 huge hangars on the site of the proposed building. An extensive plot of 365 acres have been cleared on the Bangalore-Madura Trunk Road for putting up the factory buildings and residential quarters. There will be 8 huge workshop sheds with administrative blocks. Water and light have been laid. To facilitate quick transmission a railway siding is provided. The buildings are to be completed by 1951. The factory when in full swing is expected to provide employment for 10,000 persons.

### New Engineering College

DAVANGERE, AN IMPORTANT commercial centre and third largest city in Mysore State, is to have an engineering college from the coming academic year. The Government have also sanctioned the establishment of a polytechnic in this city.

## BIHAR

### Power Alcohol Plant

THE REVENUE MINISTER OF Bihar inaugurated the biggest power alcohol plant in India at Mirganj in Saran district on April 13. The plant has a daily capacity of 3,000 gal.

## BOMBAY

### Penicillin Factory

THE RS. 3 CRORE PROJECT FOR the manufacture of penicillin, sulphadiazole and anti-malarials was approved at a recent conference held in Bombay and presided over by Dr. Syama Prasad Mookerjee, Minister for Industry and Supply, Government of India. The Conference approved of the suggestion to locate the penicillin factory at Dehu Road, 16 miles from Poona.

## NEW DELHI

### Machine Tools Factory

NEGOTIATIONS FOR AN AGREEMENT with the *Oerlikon Machine Tools Buehrle & Co.*, Zurich-Oerlikon, Switzerland, for the establishment in India of a factory to manufacture machine tools have been successfully concluded. The capital cost of the factory is estimated at Rs. 12 crores and the value of the production at Rs. 7 to 8 crores a year. The factory is expected to be completed within a period of 4 years.

The agreement provides *inter alia* that the Swiss firm will render all technical assistance in the erection of the factory, training of Indian personnel and direction and supervision of the production of the factory for 20 years from the date the production starts. They will also have a small financial interest in the project.

The Swiss firm will train Indian workmen, engineers and other technical personnel required for the factory in Switzerland. The Government of India will meet only the travelling and living expenses of these trainees and no fees for training will be charged. The firm will also set up a training school in India for the purpose of training Indian workmen, engineers and other technical men required for the factory and provide the necessary expert personnel and equipment to train such men. They will endeavour to train Indian personnel in such a way that within a period of 10 years from the date of the starting of the factory there would be sufficient number available to hold not less than 85 per cent of the technical posts in the factory.

## MADRAS

### Millet Research

THE OPENING OF FOUR MILLET research stations has been sanctioned by the Government as a temporary measure in the scheme for making the province self-

sufficient regarding millets. Narasapatam in Vizagapatam district, Ongole in Guntur district, Tirupattur in North Arcot district, and Ariyalur in Tiruchirappalli district will be the places at which the new research stations will be established.

The work of these stations will be to produce high-yielding millets suitable to different parts of the province within a period of 5 years. The cost of the scheme is estimated at Rs. 2 lakhs.

### Paper Research

THE GOVERNMENT HAVE SANCTIONED a scheme for establishing a paper research station in North Malabar for the purpose of giving scientific assistance to paper industry in South India. The scheme will run for a period of 4 years in the first instance. The station is to be located near Payyanoor.

### Limestone Deposits

A REPORT ON THE MINERAL resources of Tiruchendur and Nanguneri taluks of Tirunelveli district shows large deposits of good limestone estimated at 9,000,000 tons in Sattankulam area. This area is ideally suited for the location of cement industry also.

### Ergot Production in the Nilgiris

THE SCHEME FOR THE EXTENSIVE production of ergot on the Nilgiri Hills has been extended by the Government for a further period of 3 years from the current year. The subventions to the cultivators of rye has been increased to Rs. 10-12-0 per lb. of dry ergot, in addition to the free supply of culture for spraying and the necessary equipment. Since the inception of the scheme in 1944 the Agriculture Department has produced 5,745 lb. of ergot.

There has been considerable improvement in the quality of ergot produced. In 1944-45 when the scheme started, the alkaloid content of ergot was only a little over 0.20 per cent and at present it has gone up to nearly 0.45 per cent.

### Growing of Long-staple Cotton

THE GOVERNMENT HAVE SANCTIONED a 5-year scheme for the evolution of a suitable strain of long-staple American cotton for

(continued on page 252)

# INDIAN PATENTS

The following is a list of a few of the Patent Applications accepted in the *Gazette of India*, Part II, Section I, for April 1949.

## Plastics & Plasticizers

38615. I.C.I. LTD.: Improved anion exchange resins: *Subjecting a chlorinated paraffin wax and an alkylene polyamine to an elevated temperature.*

## Inorganic Chemicals

37423. DELHI CLOTH & GENERAL MILLS CO. LTD., SHARMA, JAIN & CHIPALKATTI: Process for the conversion of titanium into soluble state from materials containing titanium such as bauxite sludge: *Bauxite sludge in dried form is ground and mixed with sulphuric acid and heated.*

40185. ANGLO-IRANIAN OIL CO. LTD.: Catalytic desulphurization of a petroleum fraction: *Passing the distillate vapour over a cobalt-molybdate-alumina catalyst at 700°-800°F. at a pressure of 250 lb./sq. in.*

40505. N. V. ZUID-NEDERLANDSCHE SPIRITUS-FABRIEK: Method of working up waste liquor of fermentation industries in which molasses are used: *Partly evaporated liquor is burnt to reduce the potassium sulphate to sulphide and finally treating the sulphide with carbon dioxide to obtain potassium carbonate.*

## Organic Chemicals

38339. SHARP & DOM INCORP.: Preparation of benzoic acid esters of secondary-alkyl (secondary) amino-propanols and butanols and the products of said methods: *Reacting benzoic acid anhydride or halide with secondary alkyl amino propanol or butanol.*

38502. I.C.I. New leuco sulphuric esters of vat dyestuffs: *Forming the leuco sulphuric ester in the presence of organic amide in which hydrogen atoms attached to amide nitrogen have been replaced by hydrocarbon radicals.*

38554. I.C.I. LTD.: New dyestuffs of the anthraquinone series: *Sulphonating the product obtained by reacting 2:3-dichloro-1:4-diamino-anthraquinone with an alkali metal-phenoxide varying a phenyl group in para position.*

38555. I.C.I. LTD.: Vat dyestuff: *Sulphonating in the presence of organic amide in which hydrogen atoms attached to amide nitrogen atom have been replaced by hydrocarbon radicals.*

38556. I.C.I. LTD.: New leuco sulphuric esters of vat dyestuffs: *Sulphonating in the presence of an organic amide in which hydrogen atoms attached to amide nitrogen atom have been replaced by hydrocarbon radicals.*

38658. I.C.I. LTD.: Vat dyestuffs: *Sulphonating naphthalhioindigoid dyestuffs which contain chloro and/or nitro groups in the naphthalene nuclei.*

38690. LES USINES DE MELLE: Process for the production of acetone and butyl alcohol by fermentation: *A non-fermentable acid is supplied to fermenting mash by an amount*

*not higher than equivalent of the amount of neutralizing agent added.*

40175. N.V. DE BATAAFSCHE PETROLEUM MAAT-SHAPPIJ: Process for producing alcohols: *Reacting ethylene with water in the presence of a catalyst latter consisting of activated bentonite clay impregnated with hydrofluoric acid.*

## Food & Kindred Products

40544. LEVER BROTHERS & UNILEVER LTD.: Manufacture of margarine: *Successively warming, cooling and subjecting margarine to mechanical working.*

28820. BROWN: Grain flour: *Temperature of cereal maintained below the brittle point of flour cells from the time it is tempered until final milling operation.*

## Instruments

37154. DAKSHINAMURTHY: Humidity measurement devices: *Circuit consisting of wet and dry thermocouples and one or more galvanometers with switch means to connect the thermocouples selectively with the galvanometer.*

## Leather & Leather Products

40231. NEUMANN: A process for preserving hides and skins: *After usual liming, bating and scudding the pelts are treated with preservative comprising common salt and sodium metaphosphate.*

## Medical Research & Practice, Drugs & Pharmaceuticals

39121. MARCELLE FLEISCHMANN FOUNDATION INC.: Penicillin preparations: *Comprising calcium penicillin in beeswax and peanut oil.*

40233. MERCK & CO. INC.: Processes for manufacturing therapeutically valuable salts of penicillin G: *Reacting in solution an amine salt of penicillin G and a salt of alkali metal or alkaline-earth metal salts.*

40469. MERCK & CO. INC.: Procaine salt of penicillin: *Reacting penicillin or a salt of penicillin with procaine or a salt of procaine.*

## Metals & Metal Products

38508. METALS-GAS CO. LTD. & R. HUNDER: Processes for coating a metal or alloy: *Passing a continuous stream of a gaseous halide of the coating metal over the metal to be coated.*

## Miscellaneous

40957. JANSEN: Ceiling boards: *Having coconut fibre dust as base.*

38683. I.C.I. LTD.: Seed dressing: *Comprises gamma-benzene hexachloride and an organic mercurial seed disinfectant.*

38845. I.C.I. LTD.: Seed dressing: *Consisting of gelling clay mixed with water soluble dispersing agent and an organic mercury seed disinfectant.*

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No. 6

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## CONTENTS

**A Note on the Chemistry of *Solanum torvum*** .. 97  
G. V. Krishnamurti & T. R. Seshadri

**Potassium Chromate as a Deleading Agent for the  
Determination of Clerget's Sucrose** ... 99  
S. V. Ramanaiah & C. Narasingarao

**Some Properties of Acidic Oxycellulose** ... 103  
K. S. Bhujang & G. M. Nabar

### Letters to the Editor

**SYNERGISM OF PTERYGOSPERMIN WITH OTHER ANTI-  
BACTERIAL AGENTS** ... .. 111  
R. Raghunandana Rao & ( Miss ) Mariam George

**A SYNTHESIS OF KELLIN** ... .. 112  
V. V. S. Murti & T. R. Seshadri

**A SYNTHESIS OF MYRISTICIN & ELEMICIN** ... 113  
K. V. Rao, T. R. Seshadri & T. R. Thiruvengadam

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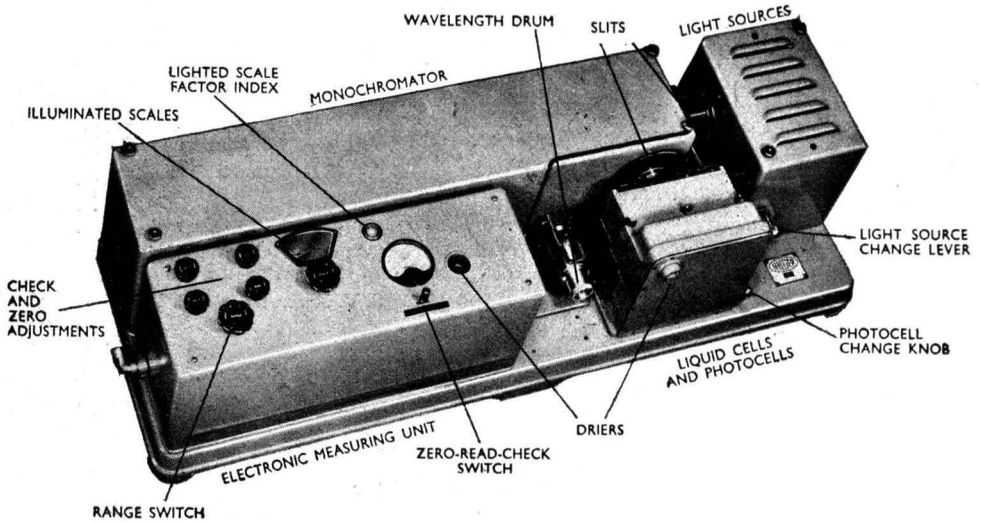
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## A Note on the Chemistry of *Solanum torvum*

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**S***OLANUM torvum* is a tomentose shrub growing throughout South India. It bears white flowers and round fruits that are bitter. They are eaten as a vegetable and are considered to be of medicinal value particularly for the enlargement of liver and spleen for which no proper specifics seem to be known<sup>1</sup>. Dry *torvum* fruits are readily available in commercial quantities. Fruits of other *Solanum* species have been recently examined and new alkaloids discovered<sup>2</sup>.

The chloroform extract of the dried drug powder yields about 1.7 per cent of oily matter from which a crystalline solid melting at 248°-249°C. (decomp.) separates out in a very small quantity. It exhibits all the properties of a sterol glycoside, forms an acetate melting at 165°-166°C. and on hydrolysis yields glucose and a sterol melting at 134°-135°C. The acetate of the aglucone melts at 128°-129°C. These agree with the data given by Jantzen and Gohdes<sup>3</sup> for sitosterol-*d*-glucoside and the aglucone sitosterol. It may be recalled here that Syed and Kanga<sup>4</sup> reported the isolation of a substance melting at 248°C. from the fruit of *S. xanthocarpum*. Though they considered it to be a sterol and named it carpesterol, its solubility, high melting point and large content of oxygen would suggest that it is also in all probability a sterolin. The remaining fixed oil resembles closely in its characteristics similar oils obtained from *S. xanthocarpum* and *S. nigrum*.

The fat-free residue on extraction with alcohol yields an alkaloid which has been purified and examined. It melts at 275°-276°C. and gives characteristic reactions of the members of the *Solanum* group. On hydrolysis it yields an aglycone which is

identified as solasodine, along with glucose, rhamnose and galactose. The original glycoalkaloid should, therefore, be solasonine<sup>5</sup>. The identity has been established by comparison with a sample of glycoalkaloid isolated from *S. xanthocarpum*<sup>6</sup> and also with its products of hydrolysis. The yield of solasonine is about 0.1 per cent and the *torvum* fruit is, therefore, a comparatively poor source of the alkaloid. But this drug can more easily be obtained from *S. xanthocarpum*. The claim recorded by Kirtikar and Basu that *S. torvum* can relieve enlargement of liver is noteworthy. A similar statement has been made by Nadkarni<sup>7</sup> with regard to *S. xanthocarpum*. This property may, therefore, be mainly due to the glycoalkaloid solasonine. No detailed physiological experiment on this point seems to have been done so far.

### Experimental

Dried fruits of *Solanum torvum* were coarsely powdered. The powder (100 gm.) was successively extracted in a Soxhlet with petroleum ether, chloroform, alcohol and water using 6 hours' of extraction for each solvent. Details of the various fractions are given below.

SOLVENT	YIELD OF EXTRACT %	NATURE OF THE PRODUCT
Petroleum ether	1.5	Dark brown viscous liquid with aromatic odour. Mainly fixed oil with a little wax
Chloroform	0.3	Brownish-yellow waxy matter, bitter to taste
Alcohol	10.2	Brown and resinous. Very bitter. Mostly bitter material with some inorganic matter
Water	7.0	Reddish brown and astringent to taste. Mostly inorganic material along with tannin

*Sterolin & Oil*—The coarse powder (2.5 kg.) was extracted with chloroform thrice in the cold using 48 hr. for each extraction. The major bulk of the solvent was recovered by ordinary distillation and the last portion removed under reduced pressure. The residue (42.0 gm.) was viscous in consistency and dark brown in colour with a characteristic aromatic odour. On cooling and allowing it to stand for an hour, a crystalline solid separated out. It was filtered and washed with benzene. It crystallized from chloroform-alcohol mixture as shining rectangular plates and prisms melting at 248°-249°C. Yield, 0.1 gm.

The filtrate (80 c.c.) was treated with more benzene (40 c.c.) and the solution heated in a water bath with animal charcoal and filtered. The clear yellow liquid was distilled to recover most of the solvent, the last traces being removed under reduced pressure. The oil thus obtained was greenish yellow when viewed in thick layers, and bright yellow when viewed in thin layers. Yield, 40.0 gm.

The crystalline solid melting at 248°-249°C. was sparingly soluble in chloroform, moderately in alcohol and insoluble in ether. It was tasteless and responded to Molisch test for carbohydrates and to Libermann-Burchard reaction for sterols. Hence it was a sterolin. On acetylation with acetic anhydride and a few drops of pyridine and crystallizing the product from absolute alcohol, the acetyl derivative was obtained in the form of colourless, shining needles melting at 165°-166°C. When the sterolin was hydrolysed with aqueous alcoholic sulphuric acid and the aglucone (sterol) crystallized from alcohol, it was obtained as colourless, shining, rectangular plates melting at 134°-135°C. The acetate of the aglucone melted at 127°-128°C. The acid filtrate containing the sugar was neutralized with barium carbonate, filtered and the filtrate concentrated to small bulk. The syrup yielded glucosazone on treatment with excess of phenyl hydrazine reagent (cf. Jantzen and Gohdes<sup>3</sup> for sitosterol-*d*-glucoside).

The oil obtained was an yellow, viscous liquid with a fine aromatic odour and burning with a smoky flame. It did not solidify on cooling even up to -5°C. but only became thick. The following are the properties of the oil: acid number, 10.4; saponification number, 183.5; Hehner number, 90.8; unsaponifiable matter, 2.3 per

cent; iodine number, 113.5; mean mol. wt. of fatty acids, 298.6. There is close agreement between this oil and those obtained from *S. xanthocarpum*<sup>8</sup> and *S. nigrum*<sup>9</sup>.

*Glycoalkaloid*—The defatted fruit was then extracted with cold alcohol thrice using 2 days for each extraction. The combined alcoholic extract was distilled to recover most of the solvent, the last portions being removed by evaporation on a water bath. The brown residue was extracted with warm water containing a little acetic acid (250 c.c.). The aqueous extract was cooled and shaken with ether to remove waxy and oily impurities. The clear aqueous solution was brought to boiling and dilute ammonia added when a granular dark-yellow solid separated out. It was allowed to settle and the supernatant liquid decanted. The precipitate was dissolved in dilute acetic acid (5 per cent) and reprecipitated by ammonia. The process was repeated twice. Finally it was crystallized from ethyl alcohol (60 per cent) first and then from methyl alcohol when it separated as long, shining needles melting at 275°-276°C. Yield, 2.0 gm.

It had a marked bitter taste. It was soluble in chloroform, alcohol and water and insoluble in ether, benzene and petroleum ether. It contained nitrogen and gave a precipitate with Mayer's reagent. It dissolved in strong sulphuric acid forming an yellow solution changing to orange-red and finally brown. It gave mild frothing with water and a positive test for carbohydrates. The glycoalkaloid was hydrolysed with sulphuric acid (7 per cent) and filtered. The filtrate was examined for sugars and the presence of glucose and rhamnose was established by the preparation of their osazones and that of galactose by conversion into mucic acid. The precipitate of aglycone salt was washed free of acid, crystallized from alcohol and boiled with dilute ammonia to liberate the free base. The mixture was filtered hot and the precipitate was washed and crystallized from alcohol when it came out as shining, star-shaped, white plates melting at 200°-202°C. (found: C, 75.3; H, 10.7; C<sub>27</sub>H<sub>43</sub>O<sub>2</sub>N, H<sub>2</sub>O requires C, 75.2; H, 10.4 per cent). The aglycone was insoluble in petroleum ether and cold ether and soluble in hot ether, chloroform and alcohol. It dissolved in strong sulphuric acid forming a red solution, turning brown on keeping. When an alcoholic solution of the substance was

treated with sulphuric acid, green fluorescence characteristic of solasodine was produced. Further it gave a series of colour reactions with anisaldehyde and acetic-sulphuric acid. The acetyl derivative of the aglycone was obtained in the form of narrow plates melting at 195°-196°C. On refluxing with methyl iodide and crystallizing the separated solid, the aglycone formed a hydroiodide melting at 283°-284°C. (found: C, 59.9; H, 8.2;  $C_{27}H_{43}O_2N$ , HI requires C, 59.9; H, 8.1 per cent). It also formed "azo-solasodine" melting at 260°-261°C. and a picrate melting at 144°-145°C.

### Summary

The dried fruit of *S. torvum* has been examined in detail. The chloroform extract yields a small amount of a sterolin (sitosterol-*d*-glucoside) and 1.7 per cent of a fatty oil which resembles closely similar oil obtained from *S. xanthocarpum* and

*S. nigrum*. Subsequent alcoholic extract gives an yield of 0.1 per cent of a glycoalkaloid which could be identified as solasonine by a detailed study of its properties and reactions and of the products of hydrolysis. The identification has been confirmed by direct comparison of the glycoalkaloid and its hydrolytic products with those obtained from *S. xanthocarpum*.

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## Potassium Chromate as a Deleading Agent for the Determination of Clerget's Sucrose

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THE method of double polarization for the determination of sucrose was first suggested by Biot; according to Brown<sup>1</sup>, this method was further developed by Clerget, who specified the length of the observation tube and the temperature at which polarization readings are to be taken. Much literature has since accumulated indicating the complexity of this determination; new modifications have been suggested, and the influence of optically active bodies and of reversion and condensation products, either present in the sugarcane or formed during the decomposition of fructose and other unstable bodies has been examined.

Among the more important modifications of determining Clerget's sucrose, mention may be made of deleading with (a) anhydrous

sodium carbonate; (b) potassium oxalate; and (c) sodium phosphate. Noel Deerr<sup>2</sup> developed the neutral polarization method employing equivalent quantities of barium hydroxide, aluminium sulphate and sulphuric acid. Based on similar considerations, and keeping in view the influence of the acid on invert polarization, Jackson and Gillis<sup>3</sup>, in their method IV, proposed the addition of sodium chloride solution (a neutral salt) to the direct polarization liquid. The International Committee on Uniform Methods of Sugar Analysis tentatively recommended Jackson-Gillis' method IV as the standard for adoption.

Zerban<sup>4</sup> re-examined the question of double polarization of sugar products and found that Jackson-Gillis' method IV gives correct values only with pure sugars, either

alone or in the presence of reducing sugars, and that the method fails when amides, amino acids and reversion and condensation products were present, the only reliable method in such cases being yeast inversion.

In view of the importance of sucrose determination to the sugar industry, the method of determining Clerget's sucrose was re-examined from the stand-point of complications introduced by lead and deleading agents on polarization. A new and simple method of deleading with potassium chromate, excess of which can be exactly controlled by using silver nitrate solution as an external indicator, has been devised and tested.

Sucrose is calculated from the general equation,\*

$$S = \frac{100(D-I)}{143.2 - 0.0676(m-13) - \frac{T}{2}}$$

employing half normal solutions, and Clerget's constants were obtained by reference to Table 15 of *Methods of Chemical Control, Hawaiian Sugar Technologists' Association* 1931 (p. 130). For sucrose estimation by yeast inversion, 10 c.c. of 5 per cent *B.D.H.* yeast solution preserved with toluene were used.

### Experimental

*Influence of Deleading Agents on the Polarization of Sugar Solutions*—The use of anhydrous sodium carbonate, sodium phosphate and potassium oxalate for deleading sugar solutions prior to direct polarization and inversion is a common practice. Incomplete deleading leads to the precipitation of lead as lead chloride in the inversion flask and introduces a precipitate error. To avoid this, complete deleading is essential, and an excess of deleading agent is usually present in sugar solutions, as there is no simple means for the precise control of deleading. The presence of deleading agents interferes with direct polarization, as may be seen from Table I.

It will be seen that excess of deleading agents introduces errors in the results. The

\* *S* stands for sucrose percentage (Clerget); *D*, direct polarization reading; *I*, invert polarization reading; *T*, temperature of invert solution in centigrade; *m*, normal weight of sugar product, 26 gm. in 100 gm. of solution. In all the calculations half normal weights are taken so that  $0.0676(m-13)$  cancels itself.

TABLE I

50 c.c. of sugar solution + deleading agent in 3 different quantities, viz. 0.5 gm., 1.5 gm. and 2.5 gm.; solution made up to 100 c.c. and polarized. Figures indicate direct polarization readings

	DELEADING AGENT	POLARIZATION READINGS			
		Blank	0.5 gm.	1.5 gm.	2.5 gm.
1.	15% sugar solution + 0.5% glucose by vol.				
	Sod. phosphate	28.5	28.7	28.2	28.9
	Pot. oxalate	28.5	28.8	29.7	28.0
	Sod. carbonate	28.5	26.7	27.0	26.2
2.	15% sugar solution + 1.0% glucose by vol. +				
	Sod. phosphate	29.5	29.8	29.2	29.0
	Pot. oxalate	29.5	29.3	30.5	29.9
	Sod. carbonate	29.5	30.5	30.2	30.1

TABLE II—CLARIFICATION WITH DRY POWDERED ALUMINIUM HYDROXIDE (Vol. of solution 50 c.c.)

SOLUTION	DIRECT POL.	INVERT POL.	CLERGET'S SUCROSE
1. 5 gm. sucrose	9.9 Clarified with al. hydroxide	...	...
	9.9 Unclarified	...	...
2. 5 gm. sucrose + 0.25 gm. glucose + 0.25 gm. fructose	10.3 Clarified with al. hydroxide	1.4	9.28
	10.3 Unclarified	1.4	9.28
3. 5 gm. sucrose + 0.25 gm. of aspartic acid + 0.5 gm. of asparagin	9.3 Clarified with al. hydroxide	1.4	8.43
	9.3 Unclarified	1.4	8.43

usefulness of a new deleading agent, viz. potassium chromate, was investigated as its adoption lends itself to exact control. The results obtained by the use of potassium chromate have been compared with those obtained by Jackson-Gillis' method and by using dry powdered aluminium hydroxide as clarifying agent. That the use of aluminium hydroxide does not introduce any errors while working with pure substances has been repeatedly confirmed. Table II gives one set of such results. From the table it can be seen that the values obtained for direct and invert polarimetric readings and Clerget's sucrose are exactly identical where the solutions are clarified with dry powdered aluminium hydroxide and where no clarifier is employed. Sugar solutions containing different quantities of glucose and water-soluble ash from molasses were prepared, and the sucrose determined by Jackson-Gillis' method. The results obtained are given in Table III.

It will be seen from the table that the results obtained by deleading with potassium chromate correspond to the values obtained by aluminium hydroxide clarification, indicating that for a wide variation in the concentration of glucose and ash, deleading with chromate gives satisfactory



TABLE III

SUGAR SOLUTION	SUCROSE DETERMINED				
	Al. hydroxide	Pot. chromate	Sod. phosphate	Pot. oxalate	Sod. carbonate
20% sucrose solution + $\frac{1}{2}$ % glucose by vol.	18.45	18.45	18.52	18.52	18.57
20% sucrose solution + $\frac{1}{2}$ % ash by vol.	18.61	18.61	18.01	18.01	18.42
20% sucrose solution + 1% glucose by vol.	19.19	18.95	18.60	18.60	18.57
20% sucrose solution + 1% ash by vol.	18.51	18.51	18.08	18.08	18.21
20% sucrose solution + 2% glucose by vol.	19.04	19.04	18.73	18.73	18.57
20% sucrose solution + 2% ash by vol.	17.87	17.89	17.48	17.48	17.79
20% sucrose solution + 3% glucose by vol.	18.57	18.57	17.89	17.98	18.13
20% sucrose solution + 3% ash by vol.	18.35	18.27	18.0	18.0	17.96

TABLE IV — DIRECT POLARIZATION OF SOLUTIONS AFTER DELEADING WITH DIFFERENT DELEADING AGENTS

HORNE'S DRY LEAD ADDED TO 100 C.C.	AL. HYDROXIDE	SOD. PHOSPHATE	POT. CHROMATE	POT. OXALATE	SOD. CARBONATE
1. 10% sucrose + 0.5% glucose solution by vol.					
0.63 gm.	38.8	39.0	38.7	38.6	38.6
1.25 "	38.8	39.0	38.7	38.5	38.6
1.88 "	38.8	39.0	38.8	38.3	38.3
2. 10% sucrose + 1% glucose solution by vol.					
0.63 gm.	41.2	41.3	41.2	41.2	41.1
1.25 "	41.2	41.1	41.2	41.0	41.1
1.88 "	41.2	41.1	41.3	41.0	41.0
3. 10% sucrose + 2.0% glucose solution by vol.					
0.63 gm.	44.1	43.3	44.1	43.7	43.6
1.25 "	44.1	43.0	44.1	43.8	43.0
1.88 "	44.1	43.1	44.1	43.7	42.2

results. Further, while the actual variation in the final values recorded in Table III appears small, the variation in readings of direct polarization are considerable. This will be clear from the readings in Table IV.

Deleading with potassium chromate using test solutions containing different concentrations of glucose and excess of lead does not introduce any errors in direct polarization while the use of other deleading agents leads to errors, especially as the concentration of glucose increases.

Even though fructose as such is not known to exist except in products from unripe sugarcane, experimental solutions containing fructose were prepared and the effect of deleading agents studied. The results (TABLE V) indicate that sucrose values obtained with potassium chromate

and aluminium hydroxide clarification closely agree.

*Effect of Amides & Amino Acids* — Though present in small quantities, asparagine and aspartic acid are known to influence the values for sucrose as determined by Clerget's procedure. The amino compounds are completely removed by deleading with potassium chromate, while they are unaffected when other deleading agents are employed (TABLE VI).

It is clear that the use of potassium chromate eliminates the errors following the use of sodium phosphate, potassium oxalate and sodium carbonate in deleading sugar solutions containing glucose, fructose, amides and amino acids and ash-forming materials. The method has been further compared with Jackson-Gillis' method IV for the analysis of cane juices. The comparative data are presented in Table VII.

TABLE V

SOLUTION	CLERGET'S SUCROSE %				
	Al. hydroxide	Pot. chromate	Sod. phosphate	Pot. oxalate	Sod. carbonate
10 gm. sucrose + 0.5 gm. fructose in 100 c.c. solution	9.41	9.34	8.6	9.0	9.0
10 gm. sucrose + 1.0 gm. fructose in 100 c.c. solution	9.8	9.8	9.8	9.5	9.3

TABLE VI — EFFECT OF AMINO ACIDS

50 c.c. sucrose solution, 10 c.c. 0.25% asparagine or 10 c.c. 1.0% aspartic acid or 10 c.c. each of both treated with lead, delead and made up to 100 c.c. and polarized.

DELEADING AGENT	POLARIZATION READINGS	
	Control	Experimental
1. 15% sucrose solution + 10 c.c. 0.25% aspartic acid by vol.		
Pot. chromate	28.7	28.7
Sod. phosphate	28.7	28.4
Pot. oxalate	28.7	28.4
Sod. carbonate	28.7	28.1
2. 15% sucrose solution + 10 c.c. 1% asparagine by vol.		
Pot. chromate	28.5	28.5
Sod. phosphate	28.5	28.0
Pot. oxalate	28.5	28.1
Sod. carbonate	28.5	28.0
3. 20% sucrose solution + 0.25% fructose + 1.75% glucose + 1% asparagine by vol.		
Pot. chromate	38.8	38.8
Sod. phosphate	38.8	38.4
Pot. oxalate	38.8	38.5
Sod. carbonate	38.8	37.3
4. 20% sucrose solution + 0.25% fructose + 1.75% glucose + 10 c.c. 0.25% aspartic acid		
Pot. chromate	38.0	38.0
Sod. phosphate	38.0	38.6
Pot. oxalate	38.0	38.7
Sod. carbonate	38.0	38.7

It is seen from the above that Jackson-Gillis' method and chromate deleading followed by acid or yeast inversion give closely agreeing results both for raw and clarified juices taken from factory samples.

*Determination of added Sucrose in Molasses & Vinasses* — The application of this method has been extended to the estimation of added sucrose to molasses and vinasses. The results obtained are shown in Tables VIIIA, VIIIB, IXA and IXB.

TABLE VII

	CLERGET'S SUCROSE%		
	Jackson-Gillis' method	Pot. chromate, yeast inversion	Pot. chromate, acid inversion
1. Raw juice	12.42	12.48	12.30
Clarified juice	14.61	14.72	14.54
2. Raw juice	10.23	10.35	10.34
Clarified juice	12.75	12.79	12.78
3. Raw juice	11.69	11.76	11.74
Clarified juice	13.90	13.94	13.94

TABLE VIIIA — ESTIMATION OF SUCROSE ADDED TO EXHAUSTED MOLASSES

Method of deleading	ACID INVERSION		
	Sugar added	Sugar recovered	Variation
Jackson-Gillis	7.63	4.21	-3.42
	11.44	10.98	-0.46
	15.24	17.03	+1.79
	19.06	16.28	-2.78
Pot. chromate	7.63	7.83	+0.20
	11.44	11.9	+0.46
	15.24	15.03	-0.21
	19.06	19.03	-0.03
Pot. oxalate	7.63	9.52	+1.91
	11.44	9.4	-2.02
	15.24	12.56	-2.68
	19.06	18.11	-0.95
Sod. carbonate	7.63	4.11	-3.52
	11.44	11.9	+0.46
	15.24	12.56	-2.68
	19.06	15.66	-3.4
Sod. phosphate	7.63	7.21	-0.42
	11.44	10.05	-1.39
	15.24	17.04	+1.8
	19.06	19.52	0.46

TABLE VIIIB — ESTIMATION OF SUCROSE ADDED TO EXHAUSTED MOLASSES

Method of analysis	YEAST INVERSION		
	Sugar added	Sugar recovered	Variation
Pot. chromate, acid inversion	3.8	3.78	-0.02
	7.63	7.64	+0.01
	15.11	15.01	-0.1
Pot. chromate, yeast inversion	3.8	3.81	+0.01
	7.63	7.75	+0.12
	15.11	15.00	-0.11
Herzfeld-yeast inversion	3.8	4.4	+0.6
	7.63	7.77	+0.14
	15.11	15.01	-0.1

TABLE IXA — ESTIMATION OF SUCROSE ADDED TO DISTILLERY VINASSES BY DIFFERENT METHODS OF ANALYSIS

METHOD	SUGAR ADDED	SUGAR RECOVERED	VARIATION
1. Sod. phosphate, acid inversion	15.26	18.07	+3.81
	19.12	16.56	-2.56
	22.93	21.82	-1.11
2. Pot. oxalate, acid inversion	15.26	13.59	-1.67
	19.12	20.22	+1.10
	22.93	22.29	-0.63
3. Sod. carbonate, acid inversion	15.26	14.06	-1.20
	19.12	21.16	+2.04
	22.93	24.03	+1.10
4. Sod. carbonate, yeast inversion	15.26	15.35	+0.09
	19.12	19.11	-0.01
	22.93	22.79	-0.14
5. Pot. chromate, acid inversion	15.26	15.36	+0.10
	19.12	19.11	-0.01
	22.93	22.79	-0.14
6. Pot. chromate, yeast inversion	15.26	15.36	+0.10
	19.12	19.14	+0.02
	22.93	22.79	-0.14
7. Jackson-Gillis	15.26	16.76	+1.52
	19.12	20.54	+1.42
	22.93	25.07	+2.14

TABLE IXB — ESTIMATION OF SUCROSE ADDED TO DISTILLERY VINASSES IN THE PRESENCE OF AMINO COMPOUNDS

METHOD	1.0 gm. asparagine and 0.22 gm. aspartic acid in 100 c.c. solution		
	SUGAR ADDED	SUGAR RECOVERED	VARIATION
1. Sod. phosphate, acid inversion	15.26	16.92	+1.66
	19.12	18.28	-0.84
	22.93	23.63	+0.70
2. Pot. oxalate, acid inversion	15.26	14.21	-1.05
	19.12	16.69	-2.43
	22.93	24.11	+1.18
3. Sod. carbonate, acid inversion	15.26	16.74	+1.48
	19.12	17.79	-1.33
	22.93	21.39	-1.54
4. Sod. carbonate, yeast inversion	15.26	15.48	+0.22
	19.12	19.06	-0.06
	22.93	22.70	-0.23
5. Pot. chromate, acid inversion	15.26	15.32	+0.06
	19.12	19.10	-0.02
	22.93	22.70	-0.23
6. Pot. chromate, yeast inversion	15.26	15.32	+0.06
	19.12	19.10	-0.02
	22.93	22.70	-0.23
7. Jackson-Gillis	15.26	17.71	+2.45
	19.12	19.87	+0.75
	22.93	22.08	-0.85

According to Saillard<sup>5</sup>, the suitability of a method for sucrose estimation can be assessed by studying the effects of pre-existing salts and nitrogenous bodies in molasses on the polarization of sucrose. For this purpose, distillery vinasses containing these non-sugars but little or no sugar was taken, known quantities of sugar added and the solution analysed for Clerget's

sucrose by different methods. In Table IXA are given the results obtained by applying the chromate method. In Table IXB are given the results obtained when the methods are applied to determine sucrose added to vinasses, asparagine and asparatic acid being also added.

The results indicate that Jackson-Gillis' method gives results which depart from the true values, while the chromate-yeast, chromate-acid and Herzfeld-yeast inversion methods give correct values for sucrose within permissible limits of experimental error.

The above studies show that deleading with potassium chromate provides a

satisfactory means of eliminating the errors due to the presence of ash constituents, amides and amino acids normally present in molasses and distillery vinasses.

The authors record their thanks to Dr. B. Viswanath for suggesting this investigation.

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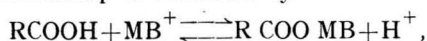
## Some Properties of Acidic Oxycellulose

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CELLULOSE when oxidized gives rise to degradation products of varying properties<sup>1</sup>. It is believed that the oxidizing agents convert the hydroxyl groups in the cellulose molecule either to the aldehydic or to the carboxylic acid stage or to a mixture of both. The products of oxidative degradation, therefore, are generally more acidic in character and also possess more reducing power than the original cellulose. The high affinity of oxycelluloses for basic dyes or for metal ions has been attributed to the presence of carboxylic acid groups. A qualitative method of estimating the extent of carboxylic acid groups formed in oxycelluloses depends on the measurement of the absorption of methylene blue by the sample<sup>2</sup>. Recently, a number of methods for the direct estimation of carboxylic acid groups in cellulose have been suggested<sup>3-6</sup>. Of these methods, the one of Neale and Stringfellow<sup>3</sup> appears to be more convenient and easy to work. Since high methylene blue absorption is accompanied by high carboxylic acid content,

it is considered that the two quantities bear a relationship as indicated by



where  $-\text{COOH}$  represents the carboxyl group of the cellulose material and MB represents the methylene blue ion in the dye solution. The present investigation was undertaken with a view to see to what extent these two quantities were related to one another.

In a recent communication, Davidson<sup>7</sup> has stressed the importance of the methylene blue absorption in determining the carboxylic acid content of oxycelluloses. After a systematic study of the effect of various conditions such as the  $\text{pH}$  of the methylene blue solution, the influence of various ions present, etc., he has come to the conclusion that the methylene blue absorption values give a direct measure of the carboxylic acid content, provided the measurement is carried out under strictly controlled conditions. His results show that the value of carboxylic acid groups as estimated by the methylene blue absorption are lower than

those obtained by the alkali titration method<sup>3</sup>, the divergence between the two being more marked for oxycelluloses having higher reducing properties. The present investigation was undertaken independently of the one referred to above and the results set out in the following indicate a good amount of agreement with the conclusions arrived at by Davidson<sup>7</sup>.

Oxycellulose samples were prepared using different oxidizing agents such as sodium hypochlorite, potassium permanganate, potassium dichromate, sodium hypobromite, hydrogen peroxide, nitrogen dioxide, etc. The oxidation experiments were carried out under different conditions such as the concentration of the active oxidant, the hydrogen ion concentration, the time of treatment of the material with the oxidizing agent and oxygen consumption. Other samples such as those prepared by the action of heat and light were also examined. The absorption of methylene blue by these oxycellulose samples was carried out by the method developed by the B.C.I.R.A.<sup>2</sup> and the amount of carboxylic acid was estimated by the method of Neale and Stringfellow<sup>3</sup>. As a result of these investigations the following conclusions have been arrived at: (a) the methylene blue absorption values and the carboxylic acid values do not bear any *direct* relationship with one another. However, they appear to depend on one another; (b) the variation in the ratio between these two quantities is more marked for oxycellulose samples having higher reducing property; and (c) the methylene blue absorption values are considerably affected by the ratio of the amount of cotton to the methylene blue solution and the concentration of the latter.

### Experimental

The cotton used for the preparation of oxycellulose samples was 14's single yarn manufactured from Indian cotton. The yarn was freed from impurities by the following treatment:

The yarn was steeped in water overnight and after squeezing was boiled under pressure (20 lb./sq. in.) for 4 hr. using 2 per cent caustic soda and 0.25 per cent Lissapol C on the weight of the material. At the end of the boil the material was washed and the boiling treatment was repeated. The boiled yarn was washed free from alkali and then treated for 1 hr. with a freshly prepared bleaching powder solution containing 0.5 gm.

of available chlorine per litre. The yarn was washed free from chlorine and then scoured in 0.25 per cent hydrochloric acid solution. It was finally washed free from acid, dried and carefully stored.

### Preparation of Oxycellulose Samples

*Hypochlorite Samples* — Purified cotton in the form of hanks was immersed in sodium hypochlorite solution of approximately 3 gm. available chlorine per litre with a material liquor ratio 1:20. The pH of the solution was varied using suitable buffer mixtures.

The treatment was carried out at 30°C. with solutions having different pH. In one set of experiments the time of treatment was kept constant; in another set, the oxidation was carried out in order to obtain approximately the same oxygen consumption. Influence of the variation of the strength of active chlorine was also examined. The active strength of the hypochlorite solution used for the experiments was estimated iodimetrically.

*Permanganate Samples* — Cotton hanks were treated with 0.04N potassium permanganate solution with a material liquor ratio 1:20 at different pH values for such period as to get the same oxygen consumption and also for a constant period so as to vary the oxygen consumption. The active strength of permanganate was measured as described above.

*Hypobromite Samples* — These samples were prepared exactly as described for hypochlorite using N/10 sodium hypobromite solution at different pH values. The sodium hypobromite solution was prepared by acidifying a mixture of potassium bromide and potassium bromate and neutralizing the same with caustic soda<sup>1</sup>.

*Acid Dichromate Samples* — The samples were prepared as described above using dichromate in sulphuric acid for different strengths of dichromate and sulphuric acid.

*Nitrogen Peroxide Samples* — The oxidation was carried out in a bottle (10 litre capacity). It was fitted with a ground-in stopper, carrying a glass hook from which the sample to be oxidized was suspended in the bottle. The bottle was first dried by blowing hot air. After cooling it was evacuated to 25" vacuum. The evacuated bottle was filled with nitrogen peroxide.

*Hydrogen Peroxide Samples* — The yarn was immersed in N/5 hydrogen peroxide solution adjusted to different pH values.

The reaction flasks were kept in water bath maintained at 80°C.

At the end of each oxidation treatment, the samples were removed from the reaction flask and freed from impurities by repeated washings with water. In the case of permanganate samples, the washed samples were treated with acidified sodium bisulphite solution so as to remove the deposited oxides of manganese, then washed thoroughly free from impurities. The dichromate samples, even after thorough washing, showed a bluish-green tint due to fixed chromium which could not be removed even after acid treatment.

All the oxycellulose samples were then treated with dilute hydrochloric acid in order to free the carboxylic acid groups. They were then washed free from acid with distilled water, dried, conditioned and stored away from dust.

*Buffer System Employed*<sup>8</sup> — Mixtures of M/15  $\text{KH}_2\text{PO}_4$  and M/15  $\text{Na}_2\text{HPO}_4$  for pH values from 5.2 to 8; mixtures of M/5 acetic acid and M/5 sodium acetate for pH values from 4 to 5; M/5 acetic acid for pH 2.7; N/10 sulphuric acid for pH 1; M/20 borax for pH 9.10; mixture of M/20 borax and M/10 sodium carbonate for pH 10; M/10 sodium carbonate solution for pH value of nearly 11; M/100 sodium hydroxide for pH 12 and M/10 sodium hydroxide for pH 13.

*Determination of Carboxyl Content in Cellulose by Alkali Titration Method*<sup>3</sup> — 1 gm. of the air-dried material (allowing for hygroscopic moisture) was weighed in a 250 c.c. stoppered conical pyrex flask and treated with 50 c.c. of N hydrochloric acid for half an hour. It was then washed with distilled water followed by washing with carbon dioxide-free distilled water until the wash waters were neutral to bromo-cresol purple (pH 5.2 to 6.8). The cotton was squeezed with a glass rod and 20 c.c. of pure sodium chloride solution (5 per cent) was added. This was followed by an addition of 6 drops of bromo-cresol purple and 20 c.c. of N/50 carbonate-free caustic soda solution and 20 c.c. of carbon dioxide-free distilled water. Carbon dioxide-free air was then bubbled through the mixture for 10 min. and the flask stoppered. At the end of 1 hr., the contents of the flask were titrated against N/50 hydrochloric acid. A blank in absence of cotton was carried out. From the difference in the readings the amount of alkali consumed by the carboxyl groups

of the cellulose material was calculated. Results are expressed as milli-equivalents of carboxyl groups per 100 gm. of cotton.

*Determination of Copper Number* — The copper number determinations were carried out by the Schwalbe-Braidy method<sup>9</sup> as modified to use smaller quantities of cotton by Hayes<sup>10</sup>. In place of N/25 permanganate, N/50 ceric sulphate was employed for titration of the ferrous iron formed, using *ortho*-ferrous-phenanthroline as internal indicator. As many of the samples had very high copper numbers, smaller quantities of cotton instead of those recommended had to be used.

*Determination of Methylene Blue Absorption* — The methylene blue employed in the experiments was the pure crystalline sample which was prepared by crystallizing commercial methylene blue hydrochloride from absolute alcohol. Its strength was estimated by the following methods: (1) by titrating against titanous chloride; (2) by determining the nitrogen and sulphur content; (3) gravimetrically estimating its perchlorate<sup>11</sup>; and (4) by titrating against standard Naphthol Yellow S solution. Naphthol Yellow S was purified by crystallizing it 3 times from aqueous alcohol and its purity estimated by determining the nitrogen content. The methylene blue absorption was carried out according to the standard method as given by Birtwell, Clibbens and Ridge<sup>2</sup>. It consists in treating 2.5 gm. of cotton in 15 c.c. M/250 methylene blue solution for 18 hr. at constant temperature and quantitatively determining the absorption of methylene blue from the difference in the titration readings before and after absorption against standard Naphthol Yellow S solution. As the pH of the methylene blue brings about variation in the absorption, the methylene blue solution was buffered at pH 7. It was found that using 15 c.c. of M/250 methylene blue solution for 2.5 gm. of cotton was not adequate for a number of oxycellulose samples under examination. Hence, the conditions of experiments were modified both by increasing the quantity of methylene blue solution and by decreasing the amount of cotton used for absorption. It was, however, found that by changing the ratio of the methylene blue solution to cotton, the methylene blue absorption values as obtained were found not comparable with one another. Hence, a study of the factors influencing the methylene blue absorption values was carried out using a fairly degraded cellulose sample.

In one set of experiments, 25 c.c. of M/250 methylene blue solution was taken and the amount of the cotton was varied. In the second set, 0.5 gm. of cotton was taken in each case and the volume of M/250 solution was varied. In the third set, the volume was kept constant at 15 c.c. and the amount of cotton also kept constant at 0.5 gm. but the concentration of the methylene blue solution was varied. Finally, in the fourth set of experiments, a large excess of methylene blue solution over the quantity likely to be absorbed by cotton was taken and varying amounts of cotton were immersed in this solution. In order that the yarn should properly mix with the solution, the

stoppered bottles containing the samples were tied to a cycle wheel which was kept rotating throughout the 18 hours at about 4 revolutions per minute. Though the oxycellulose sample in all the above sets of experiments was the same, the methylene blue absorption values were different. This is clearly seen from Table I.

### Results

The results of these investigations have been summarized in Tables I to VII and shown graphically in Figs. 1 and 2. Fig. 1 represents the relation between methylene blue absorption and carboxylic acid content of oxycellulose obtained with sodium hypochlorite and potassium permanganate. Fig. 2 represents similar relationship for oxycellulose samples obtained with sodium hypobromite, hydrogen peroxide, acid dichromate, heat and light and a few samples with permanganate solutions.

In Table II are given results obtained with hypochlorite oxidized samples. From a close examination of the copper number and the corresponding ratio ( $\text{COOH}/\text{M.B.}$ ) it is seen that the value of the ratio is very nearly unity for samples having copper numbers below 1.2. For copper numbers

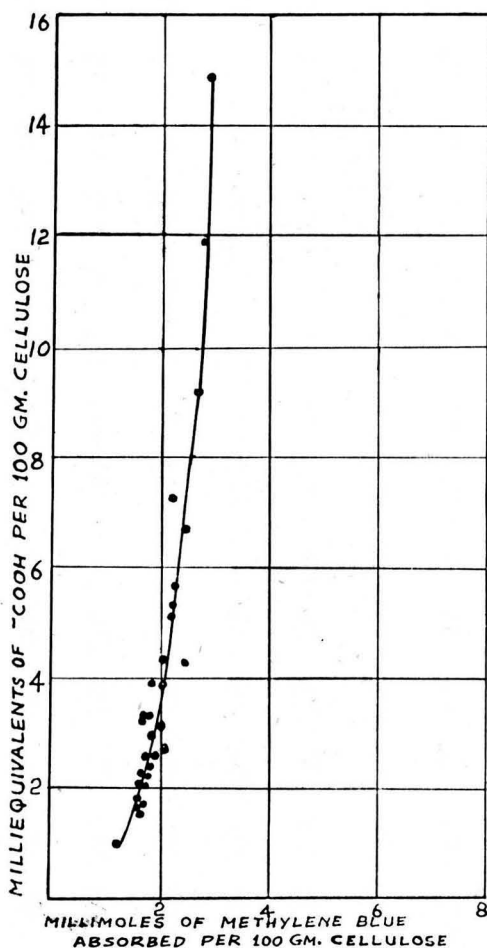


FIG. 1 — RELATION BETWEEN METHYLENE BLUE ABSORPTION AND  $-\text{COOH}$  CONTENT OF ACIDIC OXYCELLULOSE OBTAINED BY SOD. HYPOCHLORITE AND POT. PERMANGANATE TREATMENTS.

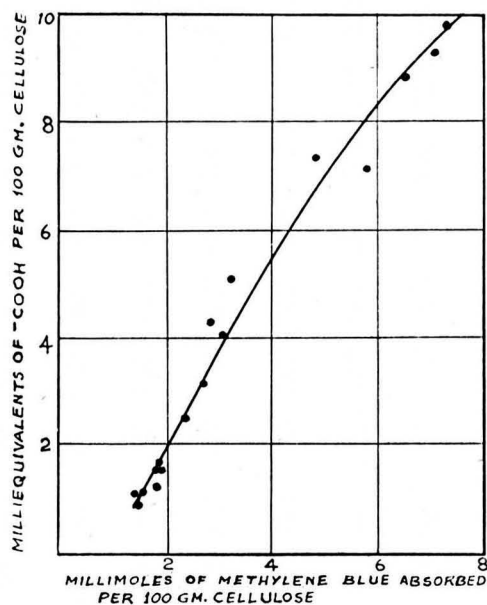


FIG. 2 — RELATION BETWEEN METHYLENE BLUE ABSORPTION AND  $-\text{COOH}$  CONTENT OF ACIDIC OXYCELLULOSES OBTAINED BY SOD. HYPOBROMITE, HYDROGEN PEROXIDE AND ACID DICHROMATE TREATMENTS.

TABLE I

STRENGTH OF METHYLENE BLUE SOLUTION	VOLUME OF METHYLENE BLUE SOLUTION C.C.	WEIGHT OF OXYCELLULOSE SAMPLE GM.	METHYLENE BLUE ABSORPTION, M. MOLES PER 100 GM. OF CELLULOSE
M/250	25	0.5	6.72
"	25	1.0	5.98
"	25	1.5	4.79
"	25	2.0	3.76
M/250	10	0.5	5.53
"	15	0.5	6.23
"	20	0.5	6.65
"	25	0.5	6.72
M/250	15	0.5	6.23
M/300	15	0.5	5.58
M/400	15	0.5	4.39
M/250	60	0.5	17.24
"	60	1.0	15.32
"	60	1.5	13.62

above this figure, this ratio increases rapidly, e.g. for a copper number of 12 this ratio has increased to 5. As suggested by Davidson<sup>7</sup>, this increase in the ratio may be attributed to the interference caused by the reducing groups during the alkali titration.

In Table III are given the results obtained with permanganate oxidized samples. The general remarks made above are equally applicable to the results recorded in this table. In view of the relatively low values of copper number of the various samples, the ratio COOH/M.B. has not deviated from unity to the same extent as was found to be the case with hypochlorite samples.

TABLE II—HYPOCHLORITE TREATMENT OF BLEACHED COTTON YARN

2 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution; temp., 30°C.

TREATMENT	OXYCELLULOSE No.	APPROXIMATE pH	METHYLENE BLUE ABSORPTION, M. MOLES PER 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
3 gm. of available Cl. per litre, for 4 hr.	1	4	1.69	1.56	0.86	0.98
	2	5	1.68	1.74	1.24	1.04
	3	6	1.75	3.29	2.81	1.80
	4	7	2.21	7.23	7.56	3.26
	5	8	2.47	6.68	4.98	2.70
	6	9	1.80	2.20	0.80	1.22
	7	10	1.96	2.61	0.84	1.30
	8	11	2.00	2.65	0.38	1.31
3 gm. of available Cl. per litre for approximately same oxygen consumption/100 gm. of cotton	9	4	1.68	2.01	1.54	1.20
	10	5	1.72	2.20	1.72	1.28
	11	6	1.76	3.29	2.74	1.80
	12	7	1.80	3.29	2.70	1.80
	13	8	2.07	3.84	2.03	1.90
	14	9	2.43	4.30	1.76	1.70
	15	10	2.08	3.11	1.07	1.50
	16	11	2.10	2.75	0.33	1.30
For 4 hr. with 2, 3, 4, 5 gm. available Cl. per litre	17	8	2.31	5.67	4.75	2.35
	18	8	2.70	9.15	7.88	3.38
	19	8	2.83	11.90	8.74	4.20
	20	8	2.95	14.82	12.01	5.20

TABLE III—PERMANGANATE TREATMENT OF BLEACHED COTTON YARN

Nos. 21-30: 2.5 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution  
Nos. 31-34: 0.5 gm. of oxycellulose in M/250 M.B. solution; temp., 30°C.

TREATMENT	OXYCELLULOSE No.	APPROXIMATE pH	METHYLENE BLUE ABSORPTION, M. MOLES PER 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
0.04 N. pot. permanganate for 2 hr.	21	2.70	1.68	1.78	0.50	1.06
	22	4.90	1.76	2.49	0.92	1.42
	23	7.00	1.69	2.05	0.84	1.29
	24	9.15	1.82	2.40	0.78	1.32
	25	12.00	2.29	5.25	0.75	2.28
0.04 N. pot. permanganate for approx. oxygen consumption equivalent to $\frac{1}{2}$ the strength of permanganate used	26	2.70	1.71	2.14	0.86	1.24
	27	4.90	1.87	2.89	1.40	1.55
	28	7.00	1.93	3.87	1.57	2.00
	29	9.15	2.07	4.32	1.39	2.00
	30	12.00	2.20	5.07	0.87	2.53
0.08 N. pot. permanganate for 1 hr.	31	12	5.80	7.12	1.38	1.23
	32	12	6.64	8.82	1.56	1.33
	33	12	7.11	9.32	1.67	1.31
	34	12	7.26	9.79	1.75	1.35

**TABLE IV—SODIUM HYPOBROMITE (N/10) TREATMENT OF BLEACHED COTTON YARN**

1.0 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution; temp., 30°C.; 5 hr.

OXYCEL- LULOSE No.	APPROXI- MATE pH	METHY- LENE BLUE ABSORPTION, M. MOLES PER 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
35	6	2.87	4.28	1.56	1.50
36	9	4.83	7.38	2.48	1.53
37	12	2.75	3.10	0.11	1.10

**TABLE V—POT. DICHROMATE + SULPHURIC ACID TREATMENT OF BLEACHED COTTON YARN**

0.5 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution; temp. 30°C.; 5 hr.

TREATMENT	OXYCEL- LULOSE No.	METHY- LENE BLUE ABSORP- TION, M. MOLES PER 100 GM. OF COTTON	CAR- BOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH B.M.
N/20 dichro- mate in N/10 sulphuric acid	38	2.39	2.43	2.65	1.0
N/20 dichro- mate in N/5 sulphuric acid	39	3.27	5.07	5.96	1.8
N/10 dichro- mate in N/10 sulphuric acid	40	3.11	4.05	5.00	1.3

**TABLE VI—TREATMENT OF BLEACHED COTTON YARN IN GASEOUS NITROGEN PEROXIDE**

0.5 gm. of oxycellulose in 50 c.c. of M/250 M.B. solution

TREATMENT	OXYCEL- LULOSE No.	METHYLENE BLUE ABSORPTION, M. MOLES PER 100 GM OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COOH M.B.
At atmospheric pressure for 1 hr.	41	39.63	48.50	1.21
For 2 hr.	42	35.64	40.42	1.13

**TABLE VII—TREATMENT OF BLEACHED COTTON YARN WITH N/5 HYDROGEN PEROXIDE AT 80°C. FOR 5 HR.**

1.0 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution

OXYCEL- LULOSE No.	APPROXI- MATE pH	METHY- LENE BLUE ABSORP- TION, M. MOLES PER 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
43	8	1.92	1.65	0.32	0.86
44	10	1.92	1.6	0.16	0.83
45	12	1.96	1.55	0.7	0.76

In Table IV are recorded the results obtained with hypobromite oxidized samples. It is seen that the ratio COOH/M.B. is not differing substantially since the copper number of the samples are not high.

The results obtained with dichromate oxidation are shown in Table V. In view of the fixed chromium in the samples, it is not possible to draw any conclusion from the methylene blue absorption values.

The results of nitrogen dioxide oxidized samples are given in Table VI. In spite of the very high carboxylic acid content, indicative of considerable degradation, the ratio of COOH/M.B. is very nearly one. The mechanism of this oxidation has been suggested to be a specific one, directed towards the conversion of the primary alcoholic groups in the gluco-pyranose residue to the carboxylic acid stage.

The results obtained with the hydrogen peroxide oxidized samples are summarized in Table VII. It is seen that the ratio COOH/M.B. is more or less constant and nearer to unity. The copper number of the oxidized samples are also low.

What has been said about the hydrogen peroxide samples is true of the properties of heat and light samples which have been given in Table VIII.

In Table IX are summarized the results of the calculations of aldehyde groups from copper number determinations described in Tables II to IV. The calculations were carried out on the assumption that the copper equivalent of maltose (10 cupric copper atoms to 10 cuprous copper atoms), as obtained by Richardson<sup>12</sup>, are equally applicable to cellulose. This means that the copper number values when multiplied by 1.58 give the amounts of milliequivalents of -CHO groups per 100 gm. of cellulose sample. From the calculated value of -CHO, it was possible to calculate the amounts of the carboxylic acid groups that might be formed due to Cannizaro reaction<sup>13,14</sup> ( $2\text{CHO} + \text{H}_2\text{O} \rightarrow -\text{COOH} + \text{CH}_2\text{OH}$ ) taking place in presence of the alkali. It was assumed that all the -CHO groups were affected in this manner.

## Discussion

The results described in the foregoing will have to be considered from the following two aspects: (1) the mechanism of methylene blue absorption and the factors influencing the same and (2) the utility of the alkali titration method in



TABLE VIII—EXPOSURE OF BLEACHED COTTON CLOTH TO LIGHT &amp; HEAT

0.5 gm. of oxycellulose in 15 c.c. of M[250 M.B. solution

TREATMENT	OXYCEL- LULOSE No.	METHY- LENE BLUE ABSORP- TION, M. MOLES PER 100 GM. OF COTTON	CAR- BOXYL CONTENT M.EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
<i>Exposure to light</i>					
for 1 month	46	1.44	0.94	0.28	0.65
for 2 months	47	1.43	1.12	0.43	0.77
<i>Heat treatment</i>					
at 140°C. for 4 hr.	48	1.83	1.23	0.23	0.67
at 110°C. for 13 hr.	49	1.59	1.29	0.57	0.81

measuring the carboxylic acid content of oxycelluloses.

Birtwell, Clibbens and Ridge<sup>1</sup> have shown that the absorption of methylene blue by oxycelluloses or even by pure cotton is dependent to a considerable extent on the hydrogen ion concentration of the solution. In a recent publication this point has been dealt with in considerable detail by Davidson<sup>7</sup>. It is known that carefully washed hydrocellulose samples show higher methylene blue absorption than the original cotton

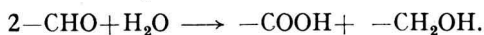
due to the presence of minute quantities of acid which are fixed and are thus extremely difficult to wash away. The presence of various salts also influence the methylene blue absorption, the cations of the salts competing for the carboxylic acid groups with the methylene blue. Reference to Table I clearly shows that the ratio of cotton to methylene blue solution and also the concentration of the methylene blue in the solution influence the methylene blue absorption. An increase in the concentration of methylene blue increases the absorption, while decreasing the ratio of methylene blue solution to cotton decreases the methylene blue absorption. This influence is more noticeable when the variation in the ratio of cotton to the methylene blue solution is substantial. For smaller differences in this ratio, the methylene blue absorption values are not seriously affected. The methylene blue absorption values, therefore, can only be compared with one another if they are determined under specified conditions. From Figs. 1 and 2 it appears that, provided the conditions for the determination of methylene blue absorption are kept constant, an approximate relationship between the two

TABLE IX

	OXYCEL- LULOSE No.	COOH— $\frac{1}{2}$ CHO	COOH M.B.	COOH— $\frac{1}{2}$ CHO M.B.	
Hypochlorite samples	1	0.88	0.98	0.49*	
	2	0.76	1.04	0.46*	
	3	1.08	1.80	0.60*	
	4	1.26	3.26	0.57*	
	5	2.74	2.70	1.10	
	6	1.57	1.22	0.87	
	7	1.95	1.30	1.00	
	8	2.36	1.31	1.16	
	9	0.80	1.20	0.47*	
	10	0.84	1.28	0.40*	
	11	1.13	1.80	0.64*	
	12	1.16	1.80	0.64*	
	13	2.24	1.90	1.08	
	14	2.91	1.70	1.12	
	15	2.27	1.50	1.00	
	16	2.49	1.30	1.07	
	17	1.91	2.35	0.80	
	18	2.93	3.38	1.10	
	19	4.99	4.20	1.76	
	20	5.33	5.02	1.80	Mean = 1.16
Permanganate samples	21	1.39	1.06	0.82	
	22	1.76	1.42	1.00	
	23	1.38	1.20	0.81	
	24	1.58	1.32	0.87	
	25	4.66	2.28	2.03*	
	26	1.46	1.24	0.83	
	27	1.79	1.55	0.90	
	28	2.64	2.00	1.36	
	29	3.21	2.08	1.55	
	30	4.39	2.53	2.00*	
	31	6.06	1.23	1.04	
	32	7.49	1.33	1.14	
	33	8.00	1.31	1.12	
	34	8.41	1.34	1.15	Mean = 1.11
Hypobromite samples	35	3.04	1.50	1.06	
	36	5.41	1.53	1.12	
	37	3.00	1.10	1.00	Mean = 1.09

In calculating the mean values for COOH— $\frac{1}{2}$ CHO, the results marked with an asterisk are omitted, since they differ very considerably from the average value of the ratio.

measurable quantities, viz. the methylene blue absorption and carboxyl content, may be obtained. In spite of the methylene blue absorption values being carried out under specified conditions, their relationship with the carboxyl content will depend on the correctness or otherwise of the method used for the determination of carboxylic acid values. The alkali titration method of Neale and Stringfellow<sup>3</sup> for the direct estimation of carboxyl acid groups has been criticized by Davidson<sup>7</sup>, who points out that the method is only applicable with acidic oxycellulose having low reducing power, and it is pointed out that the reducing groups interfere with the determination showing higher carboxylic acid values than those actually produced by oxidation. One of the possibilities in this connection is the conversion of the aldehydic groups (through the agency of water and alkali) to the carboxylic acid and primary alcoholic stage as follows<sup>13, 14</sup>:



The possibility of this type of reaction can only exist with oxycellulose containing aldehydic groups. In the case of specific oxidation mechanisms such as those with nitrogen peroxide, this reaction will not affect the determination of carboxylic acid values by the method of Neale and Stringfellow<sup>3</sup>. If the possibility of the reaction mentioned above is considered as interfering with the determination of carboxylic acid values, the higher values of carboxylic acid groups in the case of highly reducing oxycelluloses can be accounted for. It is assumed that all the available reducing groups are affected in this manner, so that the actual amount of carboxylic acid groups estimated in any one oxycellulose sample will be higher by a quantity equal to half the total number of reducing groups present in that sample. This is a big assumption. However, it was thought worthwhile to see how far this idea could be made applicable to explain the higher values of the carboxylic acid groups obtained with reducing oxycelluloses. It is assumed that the copper number values give more or less a correct measure of the total number of the reducing groups present in the oxycellulose samples. At present there is no direct evidence to relate the copper number of oxycellulose samples to the number of reducing groups present. But if the copper equivalent of 10 copper atoms found by Richardson<sup>12</sup> for reducing mono and disaccharides such as

glucose and maltose is made applicable to cellulose, it follows that for every aldehyde group present in cellulose, 10 copper atoms will be reduced from the cupric to the cuprous stage. This will, then, enable one to calculate the amount of carboxylic acid groups actually present and the amounts formed from the reducing groups. Such calculations were carried out with the various oxycelluloses described in Tables II to IV and are summarized in Table IX. The measured values of carboxylic acid groups have been corrected by that quantity which is calculated as having been formed from the aldehydic groups. The new ratio of corrected carboxyl content to methylene blue absorption is also given in the same table. This corrected ratio is found to be much more constant and nearer to unity than the uncorrected one for all samples except for those which are produced with hypochlorite solutions having pH 7 and below. However, it is of interest to mention that these also show an approximate constant ratio of 0.5 to 0.6. It is possible that, in the case of these oxycellulose samples, all the reducing groups are not affected by alkali, so that the corrections applied on this basis are higher and thus the ratio becomes lower than that for the rest of the samples.

From what has been said above, it is clear that both the methylene blue absorption method as well as the carboxylic acid determination by alkali titration method are dependent on several factors, and, therefore, they can be considered as semi-quantitative. The method for the carboxylic acid determination, however, gives more reliable results with acidic oxycelluloses with low reducing power.

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# Letters to the Editor

## SYNERGISM OF PTERYGOSPERMIN WITH OTHER ANTI-BACTERIAL AGENTS

"BACTERIA ARE KNOWN TO BECOME RESISTANT TO anti-bacterial agents, i.e. become drug resistant at a slow or fast rate, both *in vitro* and *in vivo*. This observed resistance has become a serious limiting factor in the therapeutic efficiency of anti-bacterial agents." It has been suggested that development of drug resistance is due to the rapid multiplication of a very few highly resistant organisms present in the parent strain. Since development of resistance to one drug does not make them resistant to others, combined action of more than one anti-bacterial agent has been found to be highly effective in inhibiting the growth of micro-organisms resistant to one of the drugs. Thus, the synergistic action of penicillin with many other anti-bacterial agents, *in vitro* and *in vivo*, has been demonstrated<sup>1-6</sup>. The synergism of streptomycin with several other anti-bacterial agents including penicillin against several Gram-positive and Gram-negative microbes has been studied and recorded<sup>7</sup>. Synergism in chemotherapy is thus not only a method of suppressing the induced drug fastness developed by microbes, but also of bringing under effective control several of the already resistant ones.

The purification and anti-bacterial properties of pterygospermin, the anti-bacterial principle of the drum-stick root has already been recorded by us<sup>8, 9</sup>. Since subsequent ex-

periments showed it to be highly non-toxic<sup>10</sup> also, the combined action of pterygospermin and penicillin, streptomycin or sulphapyridine against different microbes was studied. This is a preliminary communication in which we record a summary of the results of *in vitro* studies on the bacteriostatic action of pterygospermin in the presence and absence of small amounts of penicillin, streptomycin and sulphapyridine.

Crystalline sodium penicillin G, a standard sample of streptomycin (*Merck & Co.*), crystalline sulphapyridine and pterygospermin prepared by the method described by us<sup>9</sup> were used for these studies. The anti-bacterial activity of individual drugs and of mixtures of known composition were found out by the serial dilution method. Synergistic effects obtained for *S. aureus* and *E. coli* are presented in this communication.

The results clearly indicate that in the presence of non-inhibitory concentrations of penicillin and streptomycin, the anti-bacterial properties of pterygospermin are increased considerably. The minimum inhibitory concentrations of penicillin and streptomycin are also much lowered in presence of small quantities of pterygospermin. In view of the non-toxic nature of pterygospermin, the above findings are likely to assume great practical importance. The ability of pterygospermin to inhibit the growth of penicillin and streptomycin-resistant bacteria, the inability of -SH compounds to inactivate it, all seem to indicate that the mode of action of pterygospermin

TABLE I — SYNERGISM OF PTERYGOSPERMIN WITH STREPTOMYCIN

ORGANISM	GROWTH IN										CONTROL	
	PTERYGOSPERMIN ALONE IN PTERYGOSPERMIN UNITS PER C.C.			STREPTOMYCIN ALONE IN STREPTOMYCIN UNITS PER C.C.			MIXTURES					
	1.5	1.0	0.75	2.0	1.0	0.5	Streptomycin units per c.c.	Pterygospermin units per c.c.				
							...	0.75	0.5	0.25	0.1	
<i>S. aureus</i>	...	...	++	...	...	++	{	0.5	...	...	+	+++
								0.25	...	+	+	
	6	4	3.5	2.0	1.0	0.5		3	1.5	1.0		
<i>E. coli</i>	...	...	++	...	...	++	{	0.5	...	+	+	+++
								0.25	...	+	+	
								0.10	...	...	++	

TABLE II — SYNERGISM OF PTERYGOSPERMIN WITH PENICILLIN

ORGANISM	GROWTH IN										CONTROL PER C.C.	
	PTERYGOSPERMIN ALONE, UNITS PER C.C.			PENICILLIN ALONE, UNITS PER C.C.				MIXTURES				
	1.5	1.0	0.75	0.35	0.03	0.02	0.01	Penicillin units per c.c.	Pterygospermin units per c.c.			
<i>S. aureus</i>	...	...	++	...	+	+	+++	{ 0.03 0.02 0.01	...	...	...	+++
	6.0	4.0	3.5	5.0	4.0	3.0	1.85		0.75			
<i>E. coli</i>	...	...	++	...	++	++	{ 5.0 4.0 3.0 2.0	...	...	...	+++	
	...	...	++	...	++	++		+	+	+		

TABLE III — COMBINED ANTI-BACTERIAL ACTION OF PTERYGOSPERMIN & SULPHAPYRIDINE AGAINST *S. AUREUS*

PTERYGOSPERMIN UNITS PER C.C.	SULPHAPYRIDINE DILUTION × 10	GROWTH
Nil	Nil	+++
Nil	0.2	+
0.75	Nil	+
0.75	0.2	++
0.75	0.1	+
0.75	0.02	++
0.375	0.2	++
0.375	0.1	++
0.375	0.02	++

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is widely different from either that of penicillin or streptomycin.

Synergic effects observed in mixtures of sulphapyridine and pterygospermin are of a very low degree and may not be of much practical utility. Bacteria resistant to pterygospermin appear to be not affected by sulphapyridine as well. It is not, however, possible to infer anything regarding the similarity or otherwise of the mode of action of the two compounds. Detailed investigations on the synergic action of pterygospermin with other anti-bacterial agents, against other microbes, and the efficiency of the synergic mixtures *in vivo* are in progress.

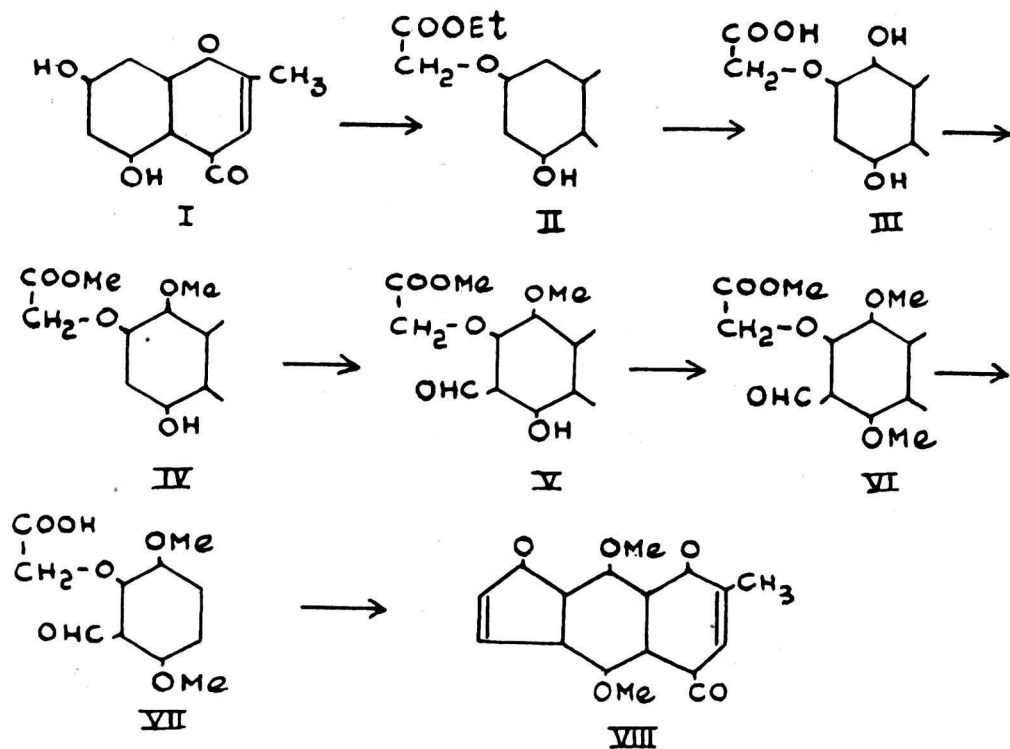
Our thanks are due to Dr. K. M. Pandalai, Dr. N. N. De and Prof. V. Subrahmanyam for their interest and helpful suggestions, to Merck & Co., Rahway, N.J. for the supply of streptomycin used, and to the Council of Scientific & Industrial Research for financial assistance.

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September 13, 1948

## A SYNTHESIS OF KELLIN

AMONG THE NATURALLY OCCURRING COMPOUNDS having a fused chromone and furan ring systems, kellin has recently assumed great importance because of its use in the treatment not only of leucoderma but also of spasmodic conditions such as asthma and intestinal colic and of certain diseased conditions of the heart. Its constitution was established by Spath and Gruber<sup>1</sup> but its synthesis has not so far been published. Based on our work of the past two years on nuclear oxidation in flavones and related compounds, it has been possible for us to effect this synthesis. The starting material is 5 : 7-dihydroxy-2-methyl chromone (I) whose preparation has been made in improved yields. It is next condensed with 1 mole of bromacetic ester in acetone solution in the presence of anhydrous potassium carbonate, the product being (II). Nuclear oxidation of it with alkaline persulphate gives rise to the quinol (III) which is partially methylated to form (IV). Condensation with hexamine in glacial acetic acid solution yields the aldehyde (V) which is subsequently subjected to complete methylation (VI),



and gentle hydrolysis with dilute alkali, yielding the aldehydo-acid (VII). Boiling with sodium acetate and acetic anhydride finally produces synthetic kellingin (VIII) which is found to be identical with natural kellingin from *Ammi visnaga*.

Our thanks are due to Professors A. Schonberg and R. B. Fahmy of Cairo for the supply of samples of natural kellingin.

V. V. S. MURTI  
T. R. SESHADRI

Department of Chemistry  
Andhra University, Waltair  
April 25, 1949

#### REFERENCE

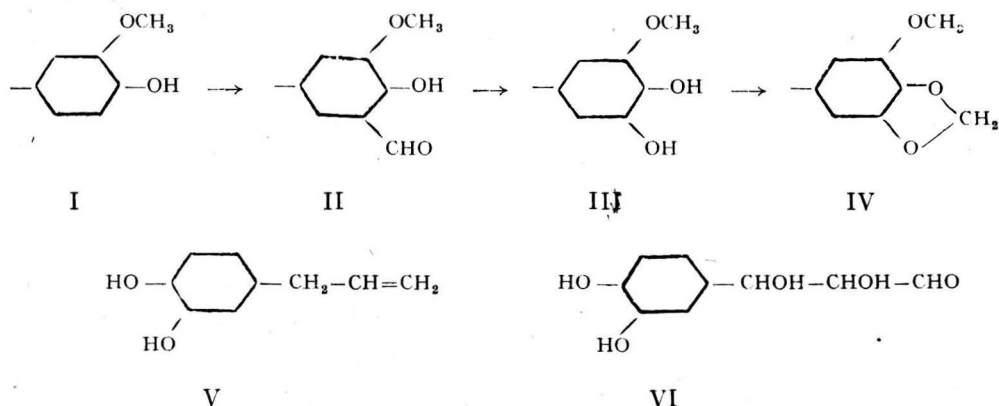
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### A SYNTHESIS OF MYRISTICIN & ELEMICIN

IN THE STUDY OF NUCLEAR OXIDATION IN the flavone series, it has been shown that *para*-oxidation takes place readily with alkaline persulphate and it could be used

in a number of ways<sup>1</sup>. On the other hand, *ortho*-oxidation does not proceed satisfactorily by this method. A two-stage process formulated below (I to III) was, therefore, adopted and its suitability proved by the synthesis of myricetin<sup>2</sup> and robinetin<sup>3</sup> involving *ortho*-oxidation in the side phenyl nucleus. One of the synthetic advantages of this method is in the preparation of methylene ethers (IV) and as a typical example the synthesis of kanugin<sup>3</sup> has been carried out.

This work has now been extended to the components of essential oils which are classified as allyl benzene derivatives. Robinson<sup>4</sup> has drawn attention to the biogenetic similarity between the nine carbon system present in these (V) and in the non-phloroglucinol part of the anthocyanins and anthoxanthins arising from component B of the biogenetic precursor (VI). The derivatives of allyl pyrogallol like myristicin (X) and elemicin (XI) may, therefore, be considered to arise from those of allyl catechol by nuclear oxidation. In support of this view, the two-stage *ortho*-oxidation process has now been carried out with

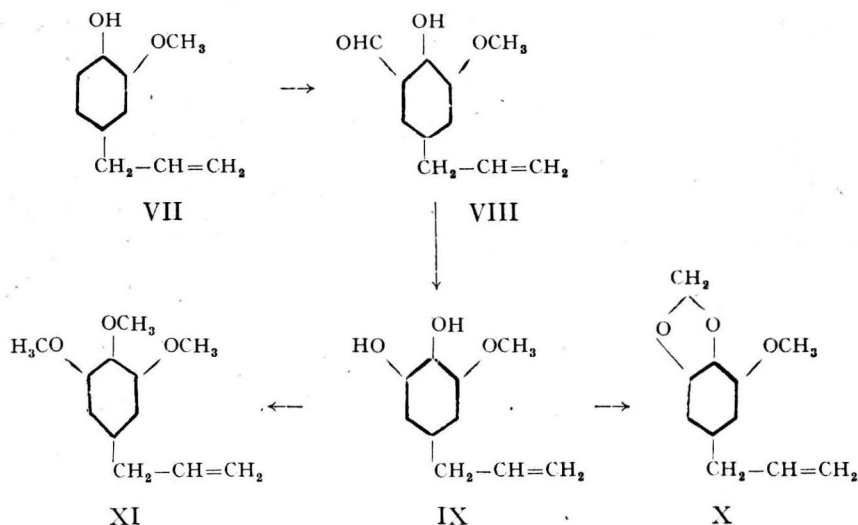


eugenol (VII), probably the most important member of the allyl-catechol type. By the action of hexamine it readily forms aldehydo-eugenol (VIII) which undergoes oxidation with hydrogen peroxide to hydroxy-eugenol (IX). Methylation of (IX) with methylene dibromide yields myristicin (X) and methylation with dimethyl and

elemicin isomerized to *iso*-elemicin and oxidized to trimethyl gallic acid.

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sulphate yields elemicin (XI). The products have been characterized by a study of the physical properties (boiling point and refractive index), by analysis and also by conversion into derivatives. Myristicin has been converted into dibromomyristicin dibromide

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