

Journal of Scientific & Industrial Research



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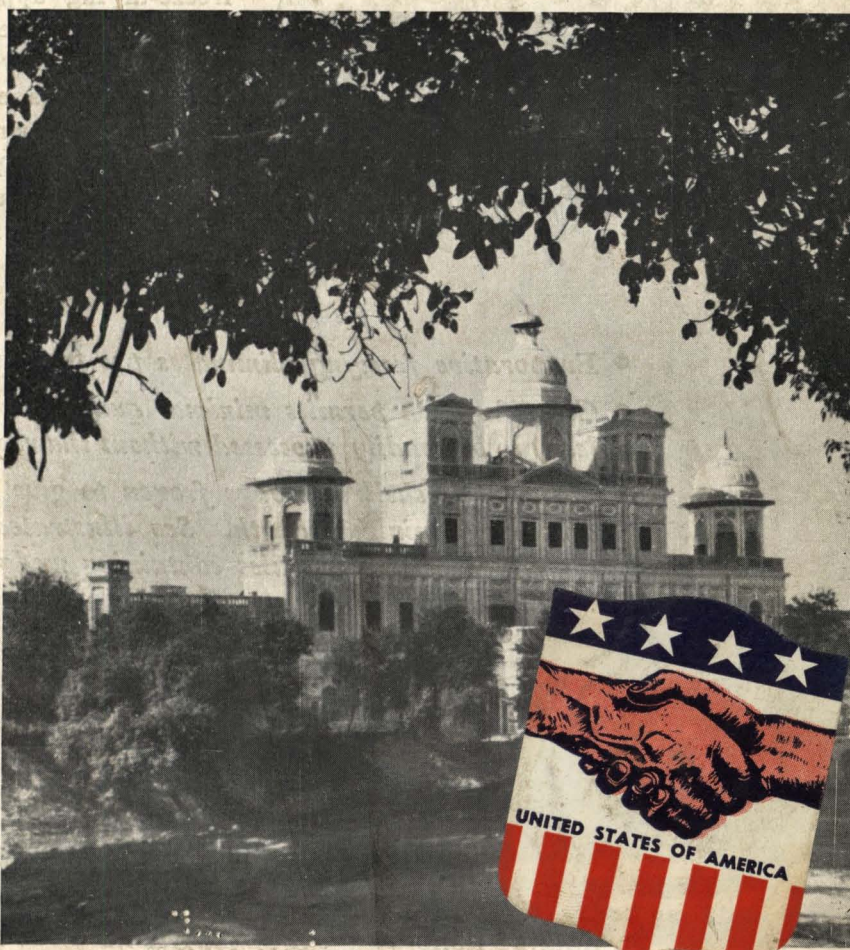
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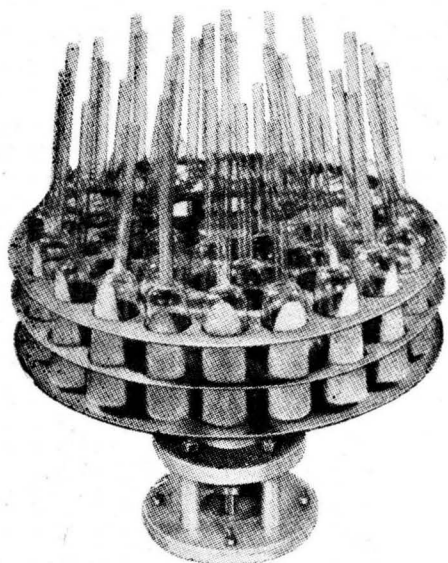
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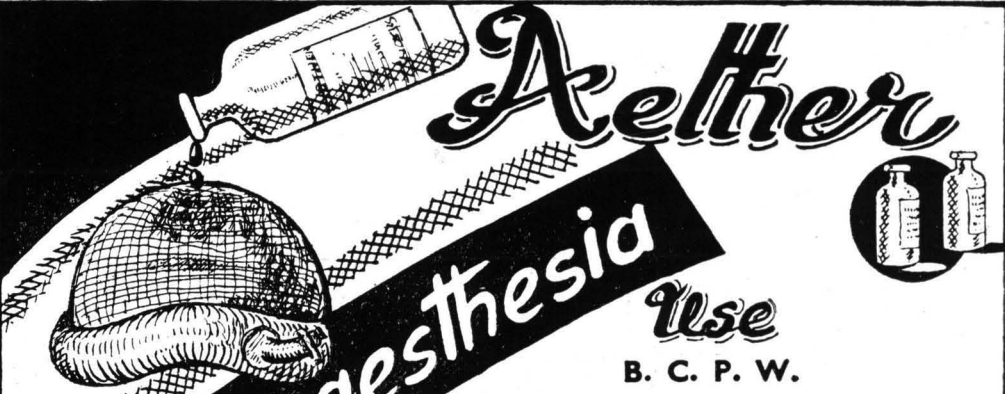
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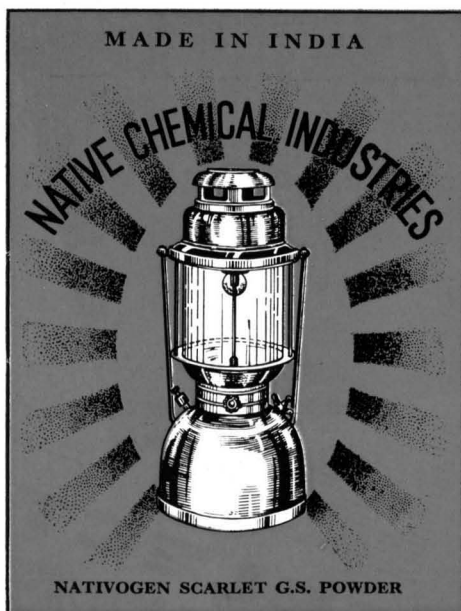
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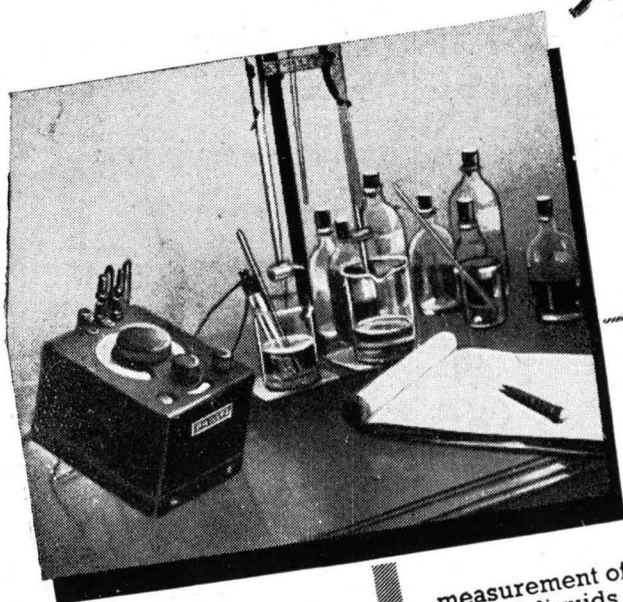
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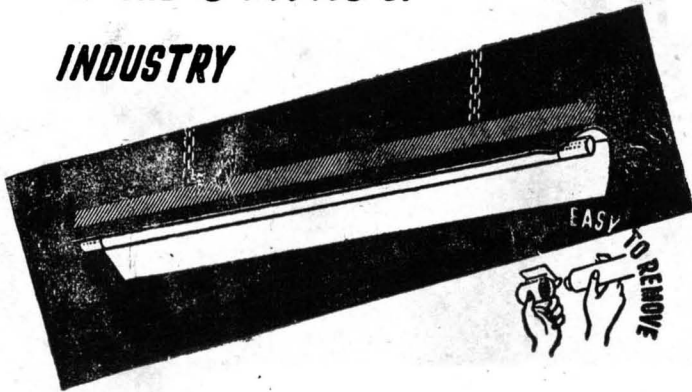
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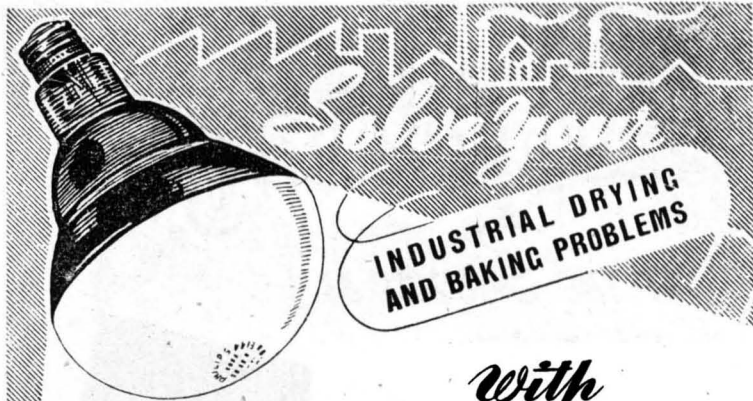
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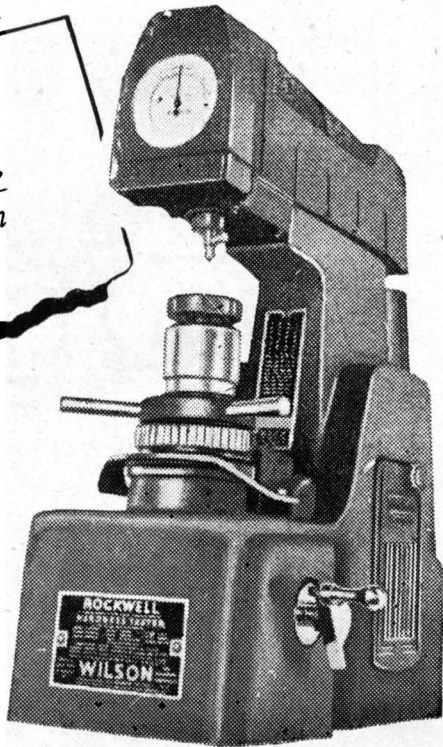
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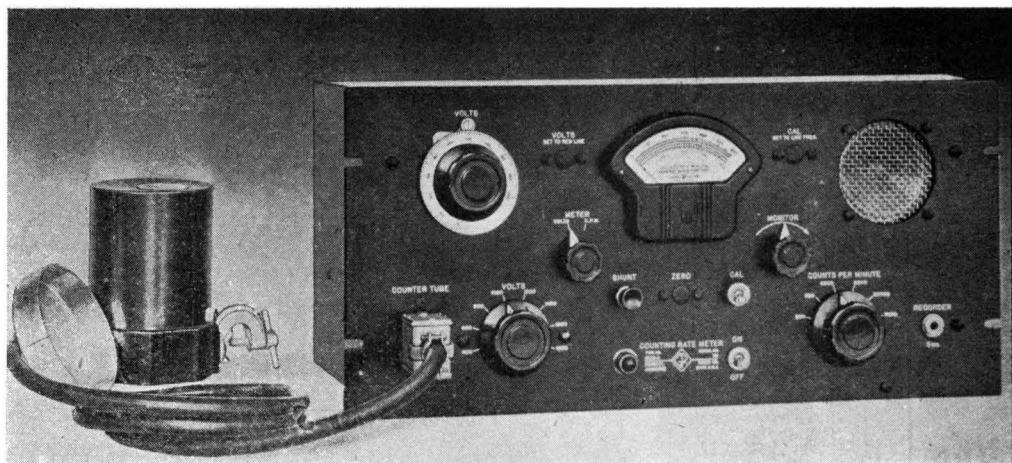
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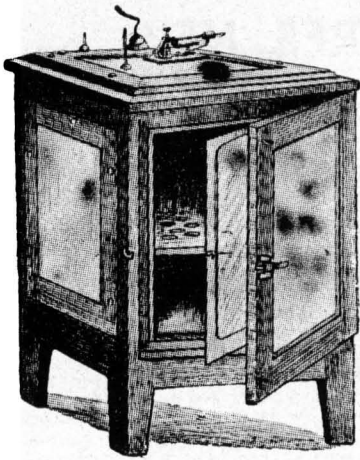
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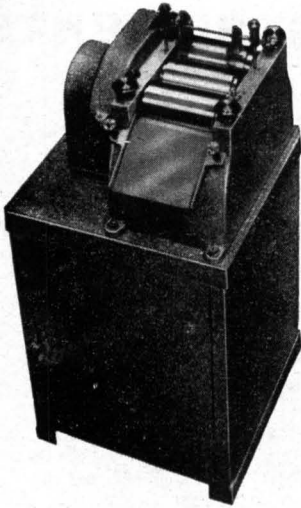
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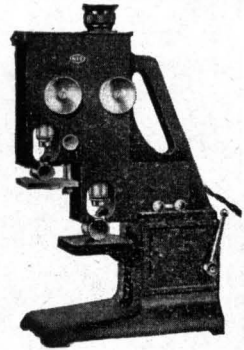
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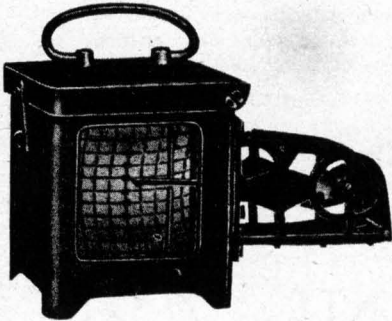
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Eclecticism in Medicine

THE report of the Committee on Indigenous Systems of Medicine, recently published by the Ministry of Health, Government of India, is a stimulating document based on the detailed inquiries into the potentialities and achievements of the systems of medicine serving the people of this country. In an article entitled *Rationalization of Medicine in India** appearing in these columns, we have referred to the practicability of bringing together the indigenous and modern systems of medicine, and the desirability — nay, imperative necessity — of utilizing the services of large numbers of practitioners of systems indigenous to this country for building up an adequate national health service. This was the thesis of the address of Col. Sir Ram Nath Chopra to the 1948 Session of the Indian Science Congress (Patna). The views expressed by him have been further emphasized and amplified by the Committee on Indigenous Systems of Medicine, over which Col. Sir Ram Nath Chopra presided.

The Committee was appointed in December 1946 to meet the public criticism which the Bhore Committee's failure to assess the value of indigenous systems of medicine evoked, and the Committee was charged specifically "to consider and recommend the steps to be taken to improve facilities for research in indigenous systems, and generally to increase their usefulness to the public". The Bhore Committee had reported: "We are unfortunately not in a position to assess the real value of these (indigenous) systems of medical treatment as practised today, as we have been unable with the time and opportunities at our disposal to conduct such an investigation into the problem as would justify clear-cut recommendations", and this after recognizing that the Western medicine, as practised in India, provided at

best only a part of the country's requirements for medical relief, and that doctors trained in allopathy were concentrated in urban areas "so much so the proportion of doctors to the rural population was such as to result in a total denial of effective medical aid to the people". The aid available to the masses in India is that provided by the *Ayurvedic* and *Unani* practitioners, and the people have shown a preference for these systems which have their roots in the soil. The indigenous systems are "time-tried and are based on sound observations, and many of their theories are capable of withstanding scientific investigation". Despite long and liberal State patronage, the Western system has failed to penetrate into the rural areas and minister to the needs of the vast majority of the people. This is not to minimize the value of the Western system, or to state that all is well with the practice of indigenous systems. The neglect from which the latter have suffered for centuries has resulted in the accumulation of a mass of superstition and dogma, and the incursion into them of a large number of ill-qualified and ill-equipped practitioners. In this respect their position is not different to what was obtaining in the Middle Ages in Europe when the classical system of medicine as taught by Hippocrates and Galen had degenerated into superstitious beliefs and quackery. In its development, allopathy has drawn freely from the knowledge available in the East, and thanks to the labours of a large body of savants working in well-equipped research laboratories all over the world, it has attained a position of pre-eminence. The indigenous systems, on the other hand, have stagnated and have fallen from their pristine glory, and their practice has suffered from the "human errors" which, according to Roger Bacon, arise from undue respect for authority, habit, prejudice and false conceit of knowledge. The first thing, then, is to rid

* March 1948, p. 101.

the systems of these "errors" and to vitalize them by the methods of experiment and observation. Research should be directed "(i) to clear these systems of accretions of centuries of doubtful value; and (ii) to give scientific meaning and significance to the fundamentals of these systems so that they may be accepted by science".

"Truth and science are one", said H.E. Sri C. Rajagopalachari in his reply to the address presented to him by the *Bangiya Ayurveda Mahasabha* in Calcutta (June 17, 1948). "There can be no competition between truth and truth, but only between truth and error. I would, therefore, entreat you to remember 3 things: The first is to demand, and not oppose, a high standard of education and equipment and careful selection in admitting students to your colleges for courses in medicine. The second is to include modern scientific knowledge alongside the traditional *Ayurveda* in your institutions so that truth may run in a single course and prejudice and ignorance vanish to the minimum point. Thirdly, scientific research should be encouraged and there should be no opposition but full co-operation in this between Western doctors and the learned *kavirajas*." This sage advice has been accepted by the Committee which "does not believe that there can be separate systems of Western or Indian medicine. Science is universal and medical science is no exception to this rule. Such multiplicity of systems is only believed and encouraged by people who have not clearly grasped the significance of the noble ideals as preached by the great *Acharyas* of Indian medicine and the savants of Western medicine. The so-called systems merely represent different aspects and approaches to medical science as practised in different ages and in different parts of the world. The aim of all systems is the maintenance of health and prevention and cure of disease. Anything of value emerging from them should be integrated and utilized for the benefit of humanity as a whole without any reservations." Starting from this incontrovertible premise, the Committee proceeds to collect and appraise evidence—oral and documentary—from leaders of Western and indigenous systems, and to examine critically the reports of Committees appointed by Provincial and State governments to inquire into, and report upon, the facilities for education and research in indigenous systems as obtaining in their respective regions. The result is a unanimous report embodying

valuable observations and recommendations for orienting medical education in the country with a view to integrating the different systems, and for rationalizing medical practice in the country as a whole. The Report observes "Indian medicine is defective in the field of surgery, obstetrics, structural physiology and pathology, and must look for help to Western medicine in these branches. On the other hand, great emphasis is laid on the prevention of disease by improving the 'soil' factor by adopting suitable dietary which has been elaborately worked out according to seasons and climates and the temperaments and avocations of individuals. Western system has yet to formulate a coherent system of principles of medicine and is in marked contrast to Indian medicine in this respect. Western and Indian medicines have thus much to give to each other, and a proper synthesis of the two will benefit humanity as a whole." The Report points out that such a synthesis "though not easy" is not only possible but practicable and essential. "It must, however, be admitted that its successful working will neither be easy nor quick. It will require the continued and painstaking research of many first-class experts for many years to evolve a complete integration that could be of real value to India in particular, and the world in general." How this research is to be organized and carried out has been fully dealt with and the steps which have to be taken immediately to make "a beginning in the right direction" have been indicated.

The emphasis placed on the "integration" of apparently different systems of medicine has given rise to the fear, in certain quarters, that the acceptance of the Committee's recommendations would be tantamount to bringing pressure on Western medicine to compromise with principles. It is feared that a "hybrid" system would result which may prove disastrous. A dispassionate study of the Report, however, reveals that the Committee does not call for any compromise with principles. What the Committee seeks to achieve is "a wise eclecticism with the main emphasis on the Indian system". This is sought to be achieved not by compulsion, but by the pooling of knowledge wherever it is found, by objective study and research. It is realized that the desired result cannot be obtained quickly or easily. Years of research by large numbers of scholars versed

in the many schools of medicine would be necessary, and the outcome of their labours would benefit not any one particular system but medicine as a whole.

The Report envisages the establishment of a Central Research Institute of the uni-purpose type. On the need for research on the subject listed in the Report there will be general agreement, but it is desirable, even necessary, to consider whether the Central Drug Research Institute, established by the *Council of Scientific & Industrial Research* at Lucknow, would not serve the purpose which the Committee has in view. An expert committee of the *Council of Scientific & Industrial Research* has thoroughly examined the problems of medical research in India, and the departments to be established at the Lucknow Institute are mostly those proposed for the new Central Research Institute. It would be prudent to consolidate, strengthen and

develop established institutions rather than to add to their number, an addition being justified only if it is established, as a result of a thorough inquiry, that one or the other of the existing institutions cannot be made to serve the needs in view.

The Committee's approach to the problems of medical education and research is objective and scientific, and discloses a width of outlook which is at once refreshing and stimulating. Its recommendations on the organization of medical relief in the country are characterized by realism. The Committee may well be proud of having found "a reasonable, practical and economical approach to the problem of medical relief to the people of this country in general and rural areas in particular without a violent disturbance of the existing state of affairs". We feel sure that their plea that the scheme may be given a fair trial would find wide support in this country.

World's First Dictionary of Colours

BRITAIN IS PRODUCING A UNIQUE AND UNUSUAL work of reference. It is a *Dictionary of Colours for Interior Decoration* and will be the first of its kind ever to be published. The dictionary will contain standard specimens of 378 different colours. These are illustrated on both plain and matt surfaces as well as on pile fabric. Each carries its own individual reference number.

This reference volume should prove invaluable not only to dyers and manufacturers of paints, fabrics and pottery, but also to the ordinary householder. It will eliminate all confusion and debate over the nomenclature of various shades of any one colour. Also, by the use of the standard reference numbers, it will make it possible to ensure perfect matching of shades either in interior decoration schemes or in dress fabric. (B.I.S.)

Dr. James William McBain, Director, National Chemical Laboratories, Poona

DR. James W. McBain, Professor of Chemistry emeritus at Stanford University in California, recently accepted appointment as Director of the National Chemical Laboratories in Poona. The appointment is for three years, beginning in October 1949.

Born in Chatham, New Brunswick, Canada on March 22, 1882, Dr. McBain received his Bachelor of Arts degree from the University of Toronto, Canada, in 1903, and his Master of Arts degree a year later. He was a student at the University of Leipzig for two years, and received a Doctor of Philosophy degree from Heidelberg University in 1906.

Known as a world authority on soap, McBain's interest in this particular field began when he was a student at Heidelberg. McBain's researches on soap have contributed a vast amount of knowledge in the field of physical chemistry and at the same time has had wide practical application.

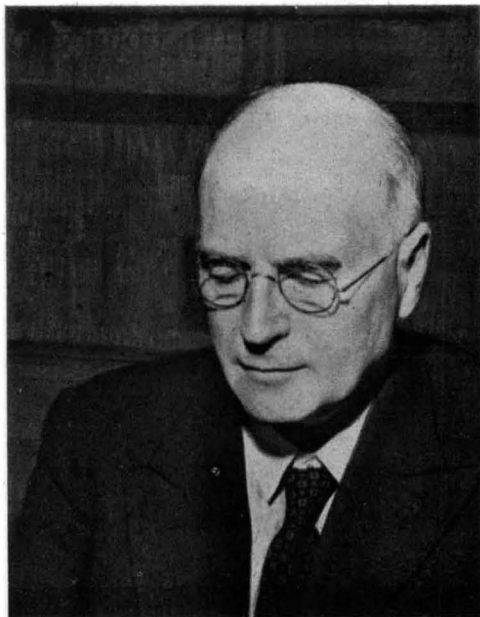
During World War II, Dr. McBain did much vital war work at Stanford and his specialization was particularly important in certain aspects of synthetic rubber manufacture, in the problem of foaming of aircraft oils, and in the thickening of fuel for incendiary bombs and flame-throwers. This was the second time Prof. McBain had been engaged in such war work; during World War I, when he was serving with the British Army in France, the British Munitions Ministry had called him back to England to do research in poison gases and explosives.

Since 1947, when Dr. McBain became Professor of Chemistry emeritus at Stanford, his "retirement" has consisted of continuing research and scientific writing. In addition, he has supervised two Office of Naval Research contracts at the Stanford Research Institute—one on the depth of a surface liquid which led to important new discoveries concerning the molecular make-

up of liquid surfaces, the other on the nature of bubbles in aqueous solutions.

Prof. McBain's publications in physical chemistry make a shelf of heavy volumes—he has published nearly 400 scientific papers on various topics in that field.

He has been a councillor and vice-president of the Faraday Society, is a Fellow of the Royal Society, and was awarded that organization's Davy Medal in 1939. He is a member of the National Institute of Social Sciences, the American Chemical Society, the Bunsen Gesellschaft, the British Chemical Society, the Society of Rheology, the American and British Associations for the Advancement of Science, and Colloid and Structure of Liquids Committees of the National Research Council (*USIS*).



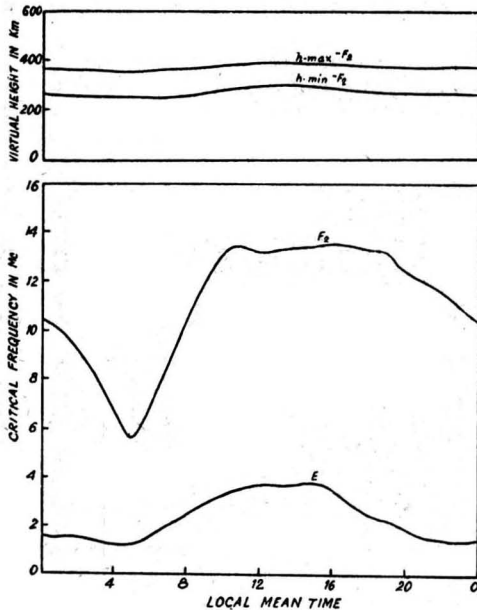
DR. J. W. MCBAIN

Characteristics of the Ionosphere over Calcutta (January 1949)

S. S. BARAL, S. N. GHOSH, R. K. MITRA,
D. C. CHOUDHURY & (Miss) T. K. PAL
Wireless Laboratory, University College of Science, Calcutta

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

Fig. 1 presents 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 the graphical form during the month of January 1949. Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmissions during April 1949 by reflection at the F region over Calcutta. Table I gives the list of occasions when E region ionization was found to be abnormal and the



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

FIG. 1 — JANUARY 1949.

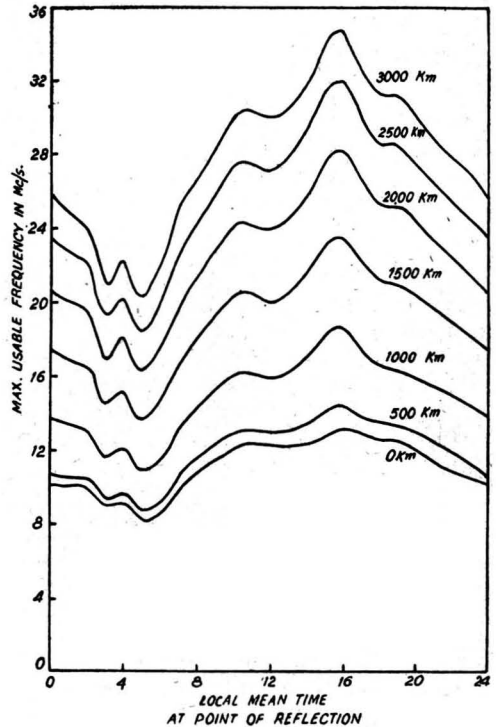


FIG. 2 — PREDICTED M.U.F. FOR TRANSMISSION VIA F_2 LAYER, APRIL 1949.

corresponding penetration frequencies and heights. The frequency of occurrence of abnormal E ionization was high during some nights and also during the afternoon on some days.

During the latter part of this month there was high sun-spot activity and day-to-day observations show that on some days the region F attained great heights and thicknesses during daylight hours and high ionization on the corresponding nights.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$	
			Mc.	Km.
Jan. 1949	3	17.00	3.30	120
		18.00	3.35	120
		21.00	3.40	120
		22.00	3.40	120
		23.00	2.85	120
	4	03.00	2.40	120
		04.00	4.05	120
		05.00	4.45	120
		06.00	3.05	120
		07.00	3.20	120
	5	08.00	3.20	120
		17.00	3.50	135
		19.00	2.15	120
		20.00	2.80	120
		21.00	2.75	120
	6	22.00	3.00	120
		23.00	3.00	120
		00.00	2.00	120
		07.00	2.00	120
		08.00	3.25	120
7	16.00	4.45	135	
	17.00	4.25	135	
	19.00	2.00	120	
	20.00	2.35	120	
	21.00	2.20	120	
	22.00	2.20	120	
	23.00	3.00	120	
	00.00	2.15	105	
	02.00	2.00	105	
	13.00	5.15	135	
8	14.00	5.20	135	
	15.00	5.20	135	
	16.00	5.20	135	
	17.00	5.00	135	
	18.00	2.60	120	
	19.00	2.10	120	
	20.00	2.80	120	
	22.00	2.10	120	
10	23.00	2.65	120	
	00.00	2.25	120	
10	04.00	2.20	105	
	17.00	3.10	120	
10	18.00	2.40	120	
	19.00	3.00	120	
	20.00	2.50	120	
	21.00	2.10	120	
	21.00	2.10	120	

TABLE I—contd.

MONTH & YEAR	DATE	HOURS	$f^{\circ}E_s$	
			Mc.	Km.
Jan. 1949	11	01.00	2.20	105
		02.00	2.20	105
		08.00	3.45	120
		09.00	3.35	120
		15.00	5.20	135
	17	16.00	4.20	135
		17.00	4.70	135
		18.00	2.30	120
		21.00	3.25	120
		23.00	6.00	190
	18	01.00	5.20	120
		02.00	3.50	120
		03.00	2.60	120
		04.00	2.35	120
		06.00	2.30	120
	20	10.00	3.95	120
		17.00	3.20	120
		18.00	2.50	120
		20.00	2.70	120
		21.00	3.30	120
21	22.00	2.70	105	
	23.00	2.70	105	
	00.00	2.30	105	
	03.00	2.60	105	
	04.00	2.50	105	
27	05.00	2.50	105	
	08.00	2.60	120	
	18.00	3.55	120	
	19.00	3.00	120	
	21.00	2.45	120	
28	22.00	2.25	120	
	23.00	2.20	120	
	00.00	2.05	105	
	07.00	2.20	120	
	08.00	3.05	120	
31	18.00	3.20	120	
	21.00	3.30	120	
	22.00	3.35	120	
	23.00	2.40	120	
	23.00	2.40	120	

Opium Alkaloids—Recent Development in India

B. K. MUKHOPADHYAY

Opium Agent, Ghazipur

OPPIUM provides India with a raw material for the manufacture of some of the most important alkaloids used in medicine. The drug has been produced in our country from time immemorial and has been used as a medicine and also to a considerable extent as an intoxicant.

The invaluable medicinal properties of opium were recognized early by modern medical science. It was found out later that the essential properties of the drug are attributable to various basic substances contained in it, the most important of which are morphine and codeine, the latter being

constitutionally the methyl ether of the former. The use of the opium alkaloids, in the form of their soluble salts, in place of the crude drug itself has been adopted largely in modern medical practice because of the accuracy attainable in adjusting the dosage, adaptability of administration through injections, ease of handling and storage and other advantages.

Morphine is the most valuable remedy for pain and consequent sleeplessness. Its use (in the form of injections of its salts in solution) is now firmly established for the treatment of internal haemorrhage. Morphine has a specific action on the alimentary canal, diminishing peristalsis, and is employed in the treatment of diarrhoea and colic. In the treatment of insomnia of cardiac disease, morphine is extremely valuable because it does not affect the heart and peripheral circulation.

Codeine is valuable in allaying cough, specially the cough of phthisis, and is useful also in insomnia, where the sleeplessness is due to incessant coughing. Equally with morphine, codeine decreases sugar in the urine of diabetic patients owing to its effect on sugar metabolism, but is not used in the treatment of diabetes. Habit formation from its continued use is almost unknown and it is used in the withdrawal treatment of morphine addiction.

The quantity of morphine and codeine used for medicinal purposes annually throughout the world has been estimated at about 1,48,000 lb¹. Medical treatment on modern lines, however, is as yet available to only a fraction of the world's population. The demand for the drugs, therefore, is sure to increase, as medical facilities are extended to hitherto undeveloped countries.

In India opium is produced solely under Government control (annual production C. 7,000 maunds). The major portion is used to prepare what is known as "Abkari Opium" or "Excise Opium", i.e. opium for sale to the public for use as a household remedy and also for consumption by habitual addicts. Opium smoking is now illegal in India. The drug is either made into pills for swallowing or drunk after being mixed with water. A portion of the raw opium is specially made into "medical" opium cake and powder corresponding to *B.P.* specification for opium and *opium pulvaratum* respectively for sale to licenced druggists and pharmacists. Opium unfit for use for these purposes and waste opium is used for recovery of alkaloids in the *Alkaloid Works* attached to the *Government Opium Factory* at Ghazipur. The quantity produced is restricted at present to what is necessary to meet the demand in India, viz. some 1,500 lb. of the principal alkaloids.

The principal alkaloids present in opium are morphine and codeine. In Indian opium the amounts are roughly 10 per cent of morphine and 2 per cent of codeine. Of the minor alkaloids, thebaine may be mentioned. This is present to the extent of some 0.5 per cent in Indian opium. Authoritative literature² mentions that Indian opium does not contain papaverine, another opium alkaloid found to the extent of up to 1 per cent in Turkish and Persian opiums. Recent work, however, carried out under the writer at the *Alkaloid Works* at Ghazipur has definitely established the presence of the alkaloid in Indian opium, though the quantity is only about 0.1 per cent. The details of the work are being prepared for publication.

AGENCY	SUB-DIVISION	M.S. max.	M.S. min.	PERCENTAGE OF challan					
				10% & above	M.S. 9-10%	M.S. 8-9%	M.S. below 8%		
Banaras	Fyzabad	12.15	8.33	52.4	33.3	14.0	nil		
	Ghazipur	11.79	8.39	46.7	26.7	26.7	nil		
	Budain	12.09	8.45	75.2	17.1	7.6	nil		
	Bareilly	12.09	7.88	71.7	19.6	7.6	1.1		
	Gonda	11.79	8.28	68.6	15.2	24.2	nil		
	Bara Banki	11.85	7.70	49.3	26.1	23.2	1.3		
	Azamgarh	12.09	8.39	80.6	11.1	8.3	nil		
	Malwa	Mandsaur	12.15	8.22	66.2	23.1	10.8	nil	
		Indore	11.85	8.17	61.3	23.8	15.0	nil	
Partabgarh		12.09	8.33	74.4	17.9	7.7	nil		
Jasor		11.74	8.56	63.3	30.6	6.1	nil		
Sitamau		11.85	8.75	75.8	18.2	6.1	nil		
Neemuch		11.74	8.39	58.8	27.5	13.8	nil		
Zhalawar		11.53	8.81	56.5	34.8	8.7	nil		
Ratlam		11.74	8.92	50.0	37.5	12.5	nil		
Sailana		10.11	8.56	25.0	50.0	25.0	nil		
Piplaudha		10.45	8.75	20.0	60.0	20.0	nil		
Dewas		11.91	8.75	50.0	nil	50.0	nil		
Nimbahira		11.69	8.28	27.3	36.4	36.4	nil		

Another alkaloid, narcotine, occurs in Indian opium to the extent of some 5 per cent. No use has yet been found for this product in quantities sufficient to justify regular production. The table above would give an idea of the amount of morphine present in Indian opiums.

For the recovery of morphine and codeine from opium the "Gregory" process³ is adopted in India. The opium is exhaustively extracted with cold water, the extracts combined and concentrated and then treated with calcium chloride which converts the alkaloids into their soluble hydrochlorides. The solution is filtered and then evaporated to crystallization. The crystals are centrifuged off the mother liquor and then treated with excess of caustic soda solution, which converts the morphine into the soluble sodium salt, and separates the codeine as the base. The latter is then separated by repeated extraction with benzene and finally recovered after distilling off the solvent and purified. It is also converted into codeine phosphate or any other salt required. From the alkaline solution, morphine is precipitated by addition of ammonium chloride. The crude morphine is filtered off, redissolved in hydrochloric acid, treated with activated charcoal and filtered, the process being repeated until the solution is sufficiently colourless. Afterwards, it is either evaporated to crystallization to produce morphine hydrochloride, or the pure base is reprecipitated with ammonia, filtered off and converted into the sulphate or any other salt. The method followed in Germany is to extract the raw opium with methylene chloride which removes the narcotine and papaverine. The residue is treated with lime water and filtered, whereupon the morphine and codeine pass on to the filtrate as the calcium compounds. They are separated as usual and made into the various salts and derivatives. The procedure is not suitable for adoption in India, because the solvent methylene chloride is not manufactured locally and is difficult to obtain in sustained and regular supply from overseas.

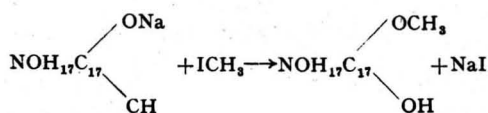
Owing to the greatly increased demands for morphine salts and codeine during the last war, the *Government Alkaloid Works* was equipped with modern machinery and fittings for recovery of the alkaloids from opium on a large scale. The plant, however, could be brought into use only after the cessation of hostilities. The capacity of the *Works* is now such that the present annual demand

of the Indian market, viz. about 1,500 lb., could be produced in less than two months.

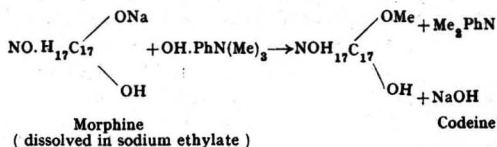
The world demand for codeine is considerable. Indian opium contains on the average about $\frac{1}{3}$ th as much codeine as morphine. The proportion is much lower in Turkish and Persian opiums. The consumption of codeine, however, is considerably more than that of morphine. Therefore, large quantities of codeine are also produced synthetically.

The synthetic production of codeine consists essentially in methylation of morphine. The latter contains two hydroxyl groups in the molecule. One of these is phenolic, while the other is alcoholic. The hydrogen of the former, upon replacement by a methyl group, produces codeine.

The classical method for methylation is analogous to the well-known Williamson's reaction, viz. by the action of methyl iodide on the sodium salt of the phenol:



The yield is, however, poor and the process has not proved to be economical enough for adoption on a commercial scale. A number of more suitable processes have been worked out of which the most successful method has been found to be the reaction employing trimethyl-phenyl ammonium hydroxide⁴:



The preparation of the methylating agent consists in treating dimethylaniline with methyl chloride in an autoclave:



The chloride is converted into the hydroxide in the presence of alkali. During the last war, the demand for codeine for the Defence Services in India was very urgent and synthetic production had to be attempted. The work was taken up and carried out under the writer at the Control Laboratory of the Central Board of Revenue at New Delhi. A tentative method was worked out and later developed using dimethyl sulphate⁵ as the methylating agent following the usual procedure adopted for the manufacture of dionine (ethyl morphine). Codeine as the

phosphate was regularly produced in small quantities and supplied to Defence Services thereafter up to the end of the war. With the cessation of hostilities the demand eased off and the production has been given up. The *Alkaloid Works* at Ghazipur is being fitted up with adequate equipment to adopt the latest process to supplement any demand for codeine beyond what is recovered from opium during the manufacture of morphine. In the meantime, manufacture of codeine from morphine on a small scale is being carried out by use of trimethyl-phenyl ammonium hydroxide (prepared in the laboratory) as the methylating agent⁶.

Planning for the Future

A market has to be found abroad for our surplus stocks of opium alkaloids. As mentioned earlier, the world demand for morphine and codeine is about 1,48,000 lb. annually. Out of this India could undertake to produce up to 10,000 lb. now and, if necessary, the forced production could be increased by providing additional units of equipment. The consumption of *Abkari* opium is bound to dwindle progressively, because the Government of India are committed to the policy of prohibition. International opinion also insists upon the prohibition of the use of opium as an intoxicant. It is, therefore, necessary to organize sale of Indian opium alkaloids in overseas markets as early as possible so that increased production of these drugs could be synchronized with the decrease in the production of excise opium. The Government of India are making attempts to secure the overseas markets accordingly. A chemical industry of international importance would emerge out with the establishment of suitable overseas markets for the drugs and would bring in considerable profits to the nation. The profits which may be expected would be apparent from the following figures :

	Price per oz. in India	Price per oz. in U.K.
Morphine	Rs. 20/-	Rs. 33/ 8/-
Morphine hydrochloride	18/-	27/14/-
Codeine	29/-	31/ 2/-

Besides this expected direct profit, the resulting employment at home and prestige abroad are also to be recorded on the credit side. Sale of raw opium to foreign manufacturers is an unsound proposition economically, politically and from the point of international prestige. The Government of India have now restricted the sale of raw opium to foreign countries, and it is likely that such sales would be totally prohibited hereafter.

As an illustration of the attempt that is being made by U.K. manufacturers to capture markets for opium alkaloids, it may be mentioned that the pre-war capacity of U.K. was 2.14 tons of these drugs corresponding to some 20 tons of raw opium. During the year 1946 and 1947, however, U.K. purchases from India amounted to 200 tons nearly.

Field for Research

As mentioned earlier, narcotine is a waste product of the opium alkaloid industry. The constitution of this base has now been established. It is a tertiary base containing three methoxy groups. Investigations would be worthwhile undertaking with the idea of manufacturing a chemical or intermediate for use, e.g. in the dye industry or in the manufacture of synthetic drugs.

Another peculiar constituent of opium is meconic acid, which is a cyclic compound containing two carboxyl groups and an alcoholic hydroxyl group in its molecule. Attention of organic research chemists is drawn to this compound which may be made available in considerable quantities. India's want of organic chemicals in general and cyclic compounds in particular should be remembered in this connection.

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Recent Developments in the Field of Highpolymers

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MACROMOLECULAR substances are equally interesting and important from the scientific and from the practical angle; their behaviour in solution and bulk can in most cases be explained by the existence of a net-work of entangled, long, flexible chain molecules, which attract each other by intermolecular forces and exhibit a certain tendency to form areas of higher geometrical order. The ultimate mechanical, thermal and electrical properties of such polymeric materials depend in a rather complicated manner on the various qualities of the individual macromolecules, and it will be the purpose of this paper to describe briefly our present knowledge of the connections between molecular structure and macroscopic properties of highpolymers. In order to obtain a vivid picture of the present state of development, I shall try to choose a number of actual problems in the field and explain how the application of certain rules has helped to improve the properties of polymers in a desired direction. The materials under consideration cover a wide range of mechanical behaviour, some of them are soft and rubbery, others are soft and plastic, others again are very hard and rigid and others finally are tough and flexible. From the practical point of view, highpolymers are used to make plastics, rubbers, films, fibres, or lacquers, etc., and it may be appropriate to discuss the modern trends in each of these fields and correlate the essential properties of representative materials with the structure of their molecules.

Trends in Plastic Research

One interesting line of development is, at present, to prepare organic plastics with very high softening points and with a great resistance to chemical changes at high temperatures. The normal organic plastics, polystyrene, polyethylene, polymethylmethacrylate, have softening ranges around 100°C. and start to deteriorate around 400°C. On the other hand, the inorganic polymers in the basis of silicates, borates, etc., which have

softening ranges around and above 300°C., exhibit distinct brittleness at lower temperatures and are stable up to 800° to 1,000°C. It would obviously be very interesting to bridge the gap between the softening range of organic (100°C.) and inorganic (400°C.) glasses and to have at our disposal transparent, colourless, hard but not brittle plastics which possess softening points between 100°C. and 400°C. and are chemically sufficiently stable to undergo reversible softening and hardening in the course of such processes as extruding, spinning, casting, injection moulding, etc. Let us consider the possible approaches to the solution of this problem. It will evidently be reasonable to start with the plastics already known and explore how their properties could be modified in the desired direction. This leads us to the question: why is the softening point of polystyrene so low? The answer is: the molecules are of the hydrocarbon type and, therefore, do not exert exceptionally strong attractive forces on one another. They are also rather flexible and can easily slip along each other under the influence of thermal motion. The *small attractive potential* between the chains and their relatively *high flexibility* are the two reasons for the low softening. As a consequence of this motion we have two possible ways to raise the softening range:

(1) to increase the mutual attraction between the chains; and

(2) to stiffen the individual chain molecules.

Both ways have been taken and in both directions improved products were obtained. To increase the intermolecular attraction between the macromolecules it is necessary to introduce polar groups or groups of high polarizability. As one wants to keep the material hydrophobic and electrically non-polar, one cannot use carboxyl, carbonyl or hydroxyl groups and is restricted essentially to such atoms and groups as the chlorine atom and the nitrile group.

In fact, great success was obtained with such simple styrene derivatives as *p*-chloro-

p-cyano-, 2,5-dichloro, 2,5-dicyano and 2,4,6-trichloro styrene. They cover a softening range from 100° to 150°C. and have almost the same excellent electrical properties as polystyrene itself. There exist certain difficulties in reaching sufficiently high degrees of polymerization with these materials and arriving at completely colourless samples; but there is little doubt that these difficulties will be overcome as one obtains monomers of high purity and works out appropriate catalysts and activators. The introduction of polar groups with a high attractive potential can also be effected by copolymerizing styrene with a monomer containing such a group. In this case one does not depart from the cheap and readily available basic monomer completely, but *modifies* it with a comparatively small amount of an auxiliary monomer. In view of water repellency and electrical properties it is again indicated to use the nitrile group or the chlorine atom. In fact, copolymers of styrene with acrylonitrile, fumaronitrile, vinylidene cyanide and pentachlorostyrene have been prepared. Their softening ranges extend from 100° to 200°C.; their electrical, mechanical and optical properties are perfectly acceptable. Equally successful was the other line of approach, namely the synthesis of chains with lesser internal flexibility. Macromolecules of the polystyrene type can be best stiffened by introducing larger side groups which impede the free rotation about the single carbon-carbon bonds in the backbone chain. Polymers of vinylnaphthalene, vinylphenanthrene, vinylfluorene, vinylcarbazole, acenaphthalene and vinyl dibenzofurane have been prepared, and it was, in fact, found that they cover the softening range between 150° and 250°C. and still have excellent optical, mechanical and electrical properties. It is evident that one also can combine the two principles enumerated above and can, at the same time, stiffen the chains and introduce groups of strong intermolecular attraction. Materials such as the copolymer of fumaronitrile and vinylfluorene or as the polyvinyltetrachloro naphthalene and the poly-dichloro-*p* dichlorodiphenyl ethylene are examples for such combinations.

It seems that one can arrive at hard, tough, transparent, colourless plastics which have softening points up to 300°C. and exhibit excellent electrical properties and remarkable chemical stability. In order to illustrate the principles involved, we have

started with polystyrene as original polymer and have introduced chemical changes in the monomer or applied copolymerization to arrive at higher softening plastics. We can, of course, also start with polymethyl-methacrylate and introduce analogous modifications. They have, in fact, been carried out in various ways and one has found that the introduction of polar groups in the chloro- and cyano-acrylates, the copolymerization of methacrylates with acrylonitrile, fumaronitrile, etc., and the introduction of bulky groups in phenacrylates, cyclohexyl methacrylates, etc., leads to a whole series of interesting polymers which combine excellent moulding properties with perfect optical clarity, remarkable ageing performance and with softening ranges up to 250°C.

Very interesting materials are obtained if one uses polyethylene as a starting material. Polyvinylchloride, polyvinylidene chloride and polyacrylonitrile are the simplest materials which one obtains if one starts introducing polar groups into the backbone chain of polyethylene. The softening range rises from 100° to about 250°C. and an almost incredible number of useful things can be made from these three polymers in the pure and plasticized state.

Copolymerization of ethylene with polar monomers also leads to higher softening materials; it meets, however, with the difficulty that ethylene, as an almost perfect gas, has to be polymerized and copolymerized under very high pressures which adds to the cost of the final product and makes copolymerization with a liquid monomer somewhat difficult. The third possibility, namely the stiffening of the principal chain by heavy substitution has led to a whole series of extremely interesting new plastics which have softening ranges as high as 350°C. and can be exposed to temperatures up to 400°C. for a prolonged period without decomposing. The first material of this type was the polytetra-fluoro-ethylene which represents a relatively soft, fatty, opaque material of excellent electrical properties, extreme toughness and a softening range near 350°C. It is very stable against any kind of chemical attack but has the disadvantage that it cannot be dissolved and not really be molten. Around 350°C. it sinters to a very viscous, opaque mass which can be brought into certain forms by high pressure. Much better moulding properties are exhibited by the poly-trifluoro-monochloro-ethylene which melts to a transparent mass around 300°C.

and has otherwise almost the same electrical, mechanical and chemical properties as the poly-tetra-fluoro-ethylene. A large number of other heavily substituted ethylenes have been, and are being, studied in connection with the syntheses of high-softening plastics; they all follow the principles outlined above either to distribute groups with strong molecular attraction along the chains or to decrease the internal flexibility of them. Summarizing, it can be said that the application of these working hypotheses has been extremely successful and that, as a consequence, we know today about 50 organic polymers, the softening point of which cover the whole range from 100° to 350°C.

Synthetic Rubbers with Exceptional Qualities

There exists a few exceedingly interesting problems in the field of synthetic rubbers. One of them is to produce substances which maintain a long-range low-modulus elasticity down to temperatures as low as -80°C. or -100°C. without being too soft at room temperature and above. The typical rubber-like elasticity of polymeric materials is connected with the presence of very long and very flexible chain molecules which are linked to each other by a few strong bonds. To comply with our requirements it is, therefore, necessary to build up an irregular molecular network of chains which have a very high internal flexibility, do not exhibit any large overall attraction in respect to each other but are firmly connected with each other by strong bonds being distributed in a random and infrequent manner over the whole system. A rubber can, in this sense, be considered to be a very fluid liquid with a superimposed system of widely scattered and irregularly arranged fix-points. These strong intermolecular bonds, which do not break during reversible extension and contraction of the samples, are responsible for the reversibility of the deformation. Their infrequent occurrence permits a long-range extension and rapid change in shape. Such fix-point systems can be produced in different ways, the most familiar being the vulcanization or curing of rubber with sulphur or sulphur compounds. One can also establish other strong chemical bonds between the macromolecules of the rubber material with the aid of polyfunctional monomers, di-*iso*-cyanates, peroxides, etc. It is not necessary that the permanent cross-links of the flexible molecular net-work must be established by primary valence forces;

they can also be caused by very strong adsorption of interfacial attraction.

This is probably the reason for the substantial improvement of the reversibility of contraction if one incorporates into an uncured rubber stock active fillers with large specific surfaces such as carbon black, zinc oxide and the like. Parts of the flexible rubber chains are strongly adsorbed and firmly held at the surfaces of the very small, colloiddally distributed carbon black particles, which assume in this manner the rôle of strong irreversible cross-links. Finally, it is possible to establish strong, if not permanent, bonds between the flexible chains by forcing small crystallized domains in the rubbery material. These minute areas of high lateral order of the macromolecules prevent them from slipping along each other in an irreversible manner and avoid the occurrence of a permanent set, even if the material is kept at a high elongation of a prolonged period. On releasing these crystals melt again and the contracted sample reassumes its soft and easily extensible character. One can also express this situation by saying that uncured rubber builds up its own cross-linking system of fix-points as it is more and more elongated by producing here and there a very small crystallized arc in which the chains are bonded together so firmly that they cannot slip along each other irreversibly and do not permit the development of a permanent set. As this process of crystallization progresses with increasing extension, the material becomes stiff and hard, as most rubbers do in the domain of high elongation and would stay so if the crystalline domains would not melt again as soon as the external forces cease to act. If one wants to have in a synthetic rubber the advantages of such a self-produced, well-controlled fix-point system and, at the same time, requires that the material should be soft and elastic at very low temperatures, it is apparently necessary that the crystals produced during stretching possess a very low melting point.

One structural requirement of a rubber which can be successfully used at very low temperatures will, therefore, be that its macromolecules should have a certain tendency to crystallize but that these crystals should have a melting point or melting range as low as -80° or -100°C. Let us now consider the significance of the fluid part of the structure, namely of the randomly entangled net-work of flexible chains which represent the bulk of the rubber material. This

liquid-like system is responsible for the tendency of a stretched piece of rubber to contract and to return to its original shape. In the normal, unstretched state these molecules represent a mass of randomly coiled and irregularly entangled chains, the individual segments of which can move past each other with a certain ease similar to the way the molecules of a viscous liquid can be displaced by the phenomenon of laminar flow. The higher the viscosity of a liquid, the slower is the displacement of its molecules under a given external force, and hence, the higher the internal viscosity of a rubbery material, the slower it will move if one attempts to deform it. That shows that if we want to get an easily deformable soft rubber, we must keep the internal viscosity as low as possible in order to make all reactions of our material as rapid and instantaneous as possible.

If one subjects a cluster of such randomly entangled, flexible polymer molecules to an external shearing force, their segments will flow as if they were individual molecules of a viscous liquid and, as a consequence, the long chain molecules will be disentangled, straightened out and oriented. This brings them into a state of decreased randomness which, according to the second law of thermodynamics, will have the tendency to go back to the original higher degree of randomness as soon as the external conditions such as temperature, pressure, acting mechanical forces, etc., permit. This is exactly what happens if we relax the stress at the extended rubber sample: the chains try to return to the state of maximum randomness (maximum entropy), coil up in an irregular manner and the sample contracts back into its original unoriented state. The degree with which this return is reversible depends upon whether any permanent displacements have occurred in the system and is connected with the strength of the fix-points which we have discussed above; the rate with which a given rubber material returns into the relaxed state depends upon the viscosity of the mass of irregularly entangled chain segments. If this viscosity is high, it will take a long time until an individual chain segment will diffuse back into its most probable position under the influence of the thermal motion, the average kinetic energy of which one degree of freedom is given by kT . As a consequence the change in shape of a stretched band of such a material is slow; it creeps back into the original, unstretched shape and will be a slow and

sluggish rubber. On the other hand, if the viscosity of the rubbery liquid is small, the individual segments will move very rapidly and we shall have a quick and snappy rubber. If we want this property to extend to very low temperatures, we must try to avoid that the internal viscosity increases with decreasing temperature, which is particularly important because the retracting tendency decreases with lowering of the temperature, as it is proportional to kT . This brief and, evidently, only qualitative analysis of our problem permits us to summarize the requirements for a material which remains rubbery even at very low temperatures as follows: we should synthesize long, flexible, chain molecules which have a very small internal viscosity in the randomly entangled (amorphous) state. This viscosity should not increase too much with a lowering of the temperature and should, if possible, still be fairly small at -80°C . or -100°C . The molecules should have a good facility to crystallize as soon as they are parallelized by our external force, but the crystals should have a melting point as low as indicated above. If a material of this character is to be used with success at higher temperatures, it will be necessary to superimpose over it a system of permanent chemical cross-bonds. Hence, our molecular chains should contain here and there, at irregular and infrequent points, certain atomic groups which permit the establishment of chemical cross-links. In this way we have now reduced our practical requirements of macroscopic behaviour to certain features of the molecular structure of the desired material and we shall now, as the next step, discuss the chances which exist to arrive actually at substances which combine the above molecular feature. Starting with the most essential requirement of a low internal viscosity and of a small temperature coefficient of it, one is evidently led to substances having a low overall intermolecular attraction, that is to hydrocarbons or fluorocarbons. The presence of polar groups along the chains will certainly be unwelcome, not only because of their strong intermolecular attraction, but also because they would impart to the material a certain degree of swelling in water, which is obviously not desirable in a material for our purpose. At first sight one could be inclined to think that polyethylene would be a suitable polymer for a low temperature rubber, because the material crystallizes easily and has no particularly attractive forces; but it

turns out that the chains of this polymer are so smooth and can be packed so easily in a crystal lattice that the melting point of the crystalline phase is too high. It is true that polyethylene remains soft and flexible even at very low temperatures but it does not exhibit any long-range, reversible elasticity because of the limited flexibility of the normal paraffin chain and of the too easy crystallizability of the material. It appears, therefore, reasonable to consider spacing the individual chain somewhat farther away to make the crystal lattice less stable. It is understood that we should not prevent crystallizability altogether, because we want crystals to be formed during extension in order to get a gradually increasing number of fix-points, but the crystallized domains should be so unstable that they will melt at very low temperatures. If we first consider using methyl groups to space the chains laterally farther apart, we are led to polypropylene. This polymer has received considerable attention, but for some reason it has not yet been possible to synthesize samples of sufficiently high degree of polymerization to produce a rubbery material of high strength. The next material is polyisobutylene which can be polymerized to very high polymerization degrees and is, in fact, an outstanding synthetic rubber with excellent properties at very low temperatures. It crystallizes remarkably easily upon extension but the crystals disappear again upon relaxation at very low temperatures. In order to facilitate vulcanization, isobutylene has been copolymerized with small amounts of isoprene or dimethyl butadiene, introducing widely spaced double bonds along the chains which are capable of reacting with sulphur and produce strong cross-links between the macromolecules. Considering polydienes it would appear that polybutadiene is a hopeful candidate. The conditions, however, are less favourable than one would expect because of the following facts:

(a) If the material is a pure 1-4-addition product, one has possibilities for *cis-trans* isomerization. A continuous *trans* chain is probably the actual configuration of this polymer; it is somewhat similar to polyethylene and forms too stable crystals to be good at low temperatures. If it were possible to synthesize a continuous *cis* chain, it would probably be a polymer which crystallizes easily and at the same time forms crystals having a low melting point. It has not yet been possible to prepare such

a material but its synthesis evidently would offer an interesting chance to arrive at a cheap superior low temperature elastomer. A polymer in which *cis* and *trans* configurations alternate randomly would be a good material from the point of view of general softness, but would probably not crystallize and, therefore, not be an ideal solution of our problem although it might have good properties at low temperatures, it hardly would satisfy expectations at normal and somewhat elevated temperatures because of lack of crystallization.

(b) The introduction of one methyl group in the 2 or 3 position leads to isoprene, the *cis* polymer of which is natural rubber, whereas the *trans* polymer is guttapercha. Looking at the molecular models of these two isomers it becomes evident that the smooth, paraffin-like chains of guttapercha can be much more easily arranged in a crystal lattice than the bulky and kinky chains of rubber. As a consequence the crystals of rubber have a much lower melting point than those of guttapercha. Both materials crystallize upon stretching, but rubber relaxes vigorously and rapidly while guttapercha remains in its stretched, and oriented state up to temperatures of about 70°C. Synthetic polyisoprene is probably a mixture of the two isomeric configurations and has, in fact, properties which are somewhere between rubber and guttapercha. The use of two methyl groups in the 2 and 3 position as spacers has first led to a polymer which was very soft but did not show sufficient strength to be regarded as a good synthetic rubber. However, it secures that the polymerization degree of these materials has not been high enough to permit the development of superior properties. Recent experiments have, in fact, shown that very interesting rubbers can be made from 2,3-dimethylbutadiene alone or in conjunction with other monomers such as isobutylene and styrene if the reaction is carried out under such conditions that a high degree of polymerization is reached. Another interesting case in which methyl groups are used as spacers is that of the poly-di-methyl-siloxane which contains backbone chains of $-\text{Si}-\text{O}-$ links flanked by methyl groups. It is not easy to produce very long chains of this type but several attempts have shown that if one succeeds in preparing them, the resulting products are rubbery and maintain their reversible elasticity down to very low temperatures.

(c) Chloroprene and fluoroprene can be polymerized to give rubbers with very attractive properties in which the chlorine and the fluorine atoms act as spacers. Both polymers show excellent reversible crystallization upon stretching. Polyfluoroprene maintains its rubberiness down to lower temperatures than polychloroprene and is also more stable against the attack of various chemical reagents. It means also that the ethyl, isopropyl, isobutyl and tertiarybutyl groups can be effectively used as spacers or internal plasticizers in polyvinyl and polybutadiene type compounds such as polyvinyl isobutylether or polyethyl butadiene which show surprisingly good behaviour at low temperatures and are also performing well at elevated temperatures as soon as the polymerization degree is high enough.

It seems, therefore, that the principle of internal plasticizers by spacing of the backbone chains offers interesting leads on how to achieve a decrease of the viscosity of the elastic liquid in a rubber and hence is a good working hypothesis for the synthesis of rubbers which remain soft and elastic even at very low temperatures.

New Developments in Fibres & Films

A very important field of practical application of macromolecules is the production of fibres and films, where many problems are still awaiting solution. The most outstanding property of a textile fibre is its ultimate tensile strength. In textile industry this is expressed in gm. per denier (where "denier" measures the cross-section of the filament under consideration). The tensile strengths of natural fibres are given briefly in the following table:

Wool	ranges from	0.8-2.2 gm. per denier
Cotton	"	1.5-4.0 "
Silk	"	3.5-5.0 "
Hemp, flax, ramie	"	4.0 to 8.0 "

The next important textile property is the elongation to break, which expresses how far a filament can be extended before suffering irreversible damage in form of a break. The protein fibres — wool and silk — have relatively high elongations to break, which range between 20 and 40 per cent, whereas cotton breaks at elongations around 10 to 15 per cent and the stronger cellulose fibres between 5 and 8 per cent; these fibres are strong but stiff. They are useful for many a practical application but leave

many other textile problems unsolved. It has, therefore, been a particularly attractive problem of fibre chemistry to prepare fibres which combine high tenacity with high elongation to break. Before we discuss the possible ways for the solution, let us first consider the conditions in the case of the natural fibres.

(a) Proteins are built up from polypeptide chains which carry many different substitutes and are characterized by the occurrence of a CONH (peptide) group in the backbone chain. These groups start strong intermolecular attraction between the long chains and, in general, favour the existence of crystalline domains. Whenever the substitutes are not too irregular such as in silk and in various synthetic polypeptides, one obtains relatively well-crystallized fibres which have a remarkable strength, but also a high elongation because there remains always a considerable part of the material in the disordered state. For the great internal flexibility of the polypeptide chain these amorphous domains exhibit rubber-like elasticity and are responsible for the relatively large reversible elongation of silk and other natural and synthetic protein fibres which exhibit crystallinity. If the substitutes at the polypeptide chains are of various nature and irregularly distributed, they impede crystallization and prevent the development of high strength. Fibres of this type, such as hair or wool, have, therefore, low tenacities but remarkably high reversible elongations.

Cellulose, on the other hand, consists of long chains of glucose which are intrinsically much stiffer than the polypeptide chains and carry a large number of hydroxyl groups which make strong intermolecular attraction possible. Cellulose fibres, as a consequence, are intrinsically stiffer than protein fibres but can have a very high tensile strength as soon as the chains are arranged so regularly that most of their hydroxyl groups contribute to intermolecular bonding. In such cases, however, the elongations to break become very small. If one wants to combine high tenacity with high elongation to break, it will obviously be necessary to build up long molecular chains of a high degree of internal flexibility which, at the same time, show a considerable tendency for crystallization. Evidently, it is necessary to strike a delicate balance; if the tendency for crystallization becomes too great, not enough of the material will be left

in the amorphous state and the fibres will be very strong but stiff. If, on the other hand, the flexibility is overwhelming and the intermolecular attraction too weak, there will result a material with a low stiffening point, which will not give the necessary strength to fibres made out of it. Most recent approaches to synthesize superior textile fibres are of different ways to arrive at a compromise between the flexibility of the backbone chain of synthetic macromolecules and the magnitude of intermolecular attraction between them. Probably the most successful solutions, at present, are the polyamides or nylons. There one maintains the peptide group CONH as the element which provides intermolecular attraction but eliminates the bulky side groups which space the individual chains apart, loosen the structure up and prevent the development of high strength. If one would stick to *d*-amino acids, however, one would arrive at very highly crystalline materials which are infusible, insoluble and give very strong but stiff fibres. Somehow one has to insert the element of flexibility without interfering too much with intermolecular attraction.

In the case of nylon this is done by introducing between the peptide groups along the macromolecules, relatively long, flexible paraffin chains of 6, 8 or 10 carbon atoms. These hydrocarbon segments do not contribute to the intermolecular attraction but they do not space the chains apart and hence do not lessen their lateral cohesion; they only introduce internal flexibility and, hence, facilitate the development of rubberiness in the structure. This leads to fibres which can be crystallized to a considerable extent by cold drawing and as a consequence possess tenacities as high as 8 and even 9 gm. per denier. The forces which bond the macromolecules laterally are essentially hydrogen bridges between the NH groups of one chain and the CO groups of the adjacent chains. They represent a rather strong lateral bonding and are responsible for the high melting point of the various nylons and for the strong resistance against swelling agents and solvents, all properties which are extremely valuable from the textile point of view. There are, however, even after cold drawing, enough disordered areas left to provide for a considerable amount of reversible elongation and for a high elongation to break. The longer the inter-spaced hydrocarbon chain pieces are, the polymers assume a more and more

plastic character and, in the limit, have the characteristic properties of polyethylene. They are very interesting products as far as injection moulding is concerned, but are not really outstanding fibre formers. Recently it has been found that high-withstanding and easily crystallizable polyesters can be obtained if one combines ethyleneglycol with aromatic dicarboxylic acids such as terphenylic acid, diphenyl-dicarboxylic acid or naphthalene dicarboxylic acid.

These acids introduce into the chain a certain element of stiffness which facilitates crystallization in spite of the fact that the lateral binding between the chains is not excessively high and makes this special group of polyesters, the so-called terylenes, to be particularly interesting polymers for the production of strong, high-binding fibres, which have somewhat less extensibility than the polyamides but compare favourably with them from the point of view of molecules and price. If one wants to make a fibre of rubber-like character, it will obviously be necessary to maintain a few irregularly spaced, relatively strong lateral bonds between the chains, but space them otherwise sufficiently apart and weaken their average intermolecular attraction so that they will form crystalline domains if parallelized, but crystalline domains of such low stability, that their lateral order collapses again as soon as the stress in the filament is released. One way of achieving this goal is to use a mixture of several dicarboxylic acids, glycols or diamines instead of only one type of each component. This blending produces an irregular arrangement of the points of strong lateral attraction along the length of the macromolecules; at those points, where CO and NH groups of adjacent chains are in favourable position, there will be considerable intermolecular attraction, whereas in between, where the CO and NH groups of neighbouring chains are out of phase, there will be only weak lateral attraction. Fibres of films made out of such polymers show, in fact, typical long-range, rubber-like elasticity. Another way to arrive at the same result is to space the chains apart by bulky, non-polar substances and to reduce the frequency of lateral hydrogen bonds by using alkyl-substituted diamines. It is evident that many combinations are possible to introduce the principles of rubberiness in a polyester, polyamides or polyurethanes and their sorts, in fact, a whole spectrum of polymers of this type ranging from low

melting, tacky rubbers to rigid, strong and high melting plastics and fibres.

If one applies our general principles to the question of fibre-forming polyvinyl derivatives, it becomes evident that the following factors have to be combined: high molecular weight, strong intermolecular attraction, sufficient internal flexibility of the chain, high stability against the action of heat, light and oxygen. Many polyvinyl derivatives have been studied from the point of view of their units as fibre formers and, in fact, relatively good filaments can be obtained from polyvinyl alcohol, polyvinyl chloride and polyvinylidene chloride. It means, however, that a real satisfactory economic compound of all of the above-mentioned qualities have only been obtained by the use of polyacrylonitrile. Fibres spun of solutions of this material are called "orlon" and combine high tenacity, soft handle, good resistance and pleasant lustre in a remarkable manner. They excel also most other textile materials in their resistance against the deteriorating influences of outdoor uses. It is very probable that other polymers, particularly copolymers consisting of two or more vinyl and acryl-type monomers, will perform equally well and, maybe, even better than "orlon" in certain special requirements.

Altogether it can be said that the modern full synthetic fibres are distinctly superior in most qualities to their natural counterparts, the only unsolved problem being at present a synthetic material of wool-like character with high tensile strength and good resistance against atmospheric influences and moulds.

Turning finally to the discussion of fibre-forming polymers, their main requirements are briefly enumerated and discussed. Qualities like clarity, transparency, good resistance against tear and high softening points are evident, but others such as flexibility at low temperatures, heat capacity and resistance against the permeation of water vapour are also of great importance.

Cellulose, in the form of cellophane, is a fairly satisfactory material for many purposes, but its tear strength is relatively poor; it has no heat-sealing capacity and offers no resistance against the diffusion of water vapour. The two last properties can be successfully improved by coating cellophane with a thin layer of a heat-sealing hydrophobic resin, a procedure which leads to an improved type of cellophane. It would, however, be more satisfactory and probably at the end also more economical if all essential properties were combined in one single polymer. As a consequence, many studies are carried out on exploring the fibre-forming properties of such materials as polyethylene, polyacrylonitrile, polyvinylidene chloride, polyesters, polyamides, etc., with the result that there is an almost unlimited number of polymers and copolymers which combine the above-mentioned natural properties of fibre-forming material in many different ways and lead to fibres of very attractive and interesting properties. It would probably be premature to say which of these polymers will finally win out as the best and cheapest solution of the problem, but it is evident that a rather rapid technological development is ahead of us in these fields.

German Dyestuff Industry

K. VENKATARAMAN

I— I. G. Farbenindustrie, Höchst

THE I. G. Farbenindustrie has already been reported upon by several British and American teams of workers. The object of the visit by the present team, consisting only of one person, was to make a few observations regarding the plant and processes on which information was not available at the time.

Höchst occupies an area of about 370 acres and normally employed 12,000 workers. The factory manufactured a very wide variety of dyes, intermediates, pharmaceuticals, heavy chemicals, etc. The factory generates its own power using boilers operating at 100 atm.

It was stated that much of the plant at Höchst was manufactured in their own engineering workshops. Höchst had 90 engineers and 2,000 skilled workmen in the engineering section. Rubber lining and plastic lining (the main plastics used being Igelit, polyvinyl chloride) were carried out locally, but enamelling was entrusted to Eisenwerke Kaiserslauten, Kaiserslauten, Palatinum.

The Höchst plant was characterized throughout by good lighting, ventilation, excellent layout and generous spacing for convenient working up and movement of materials, logical arrangement of plant, and availability of spare units enabling the factory to cope with sudden and varying demands.

The products on which reports are appended may be classified as follows:

Intermediates:

- (a) Aliphatic
- (b) Aromatic

Dyes

Miscellaneous chemicals

Intermediates

(a) ALIPHATIC

Dimethyl sulphate—Dimethyl ether was supplied by the I.G. Factory at Leuna in 12-ton tank wagons under 8 atm. pressure. Dimethyl ether was a by-product at Leuna.

Diethyl sulphate—This was made from 92 per cent ethanol. The process from ethylene was also employed.

Acetoacetic ester (Ethyl acetoacetate)—

The process was worked under licence from Alexander Wackerwerke, Munich. The normal capacity was 120 tons a month, but 40 tons a month are now being produced, the production having commenced in the third week of June 1946. The plant was constructed in 1935-36, and part of the equipment was supplied by Wacker.

Acetoacetic ester is prepared in the pharmaceutical section, but over 60 tons per month were used for dyes. The keten method was probably cheaper, but was not used at Höchst since the Wacker process was found to be more convenient to handle.

Detailed processes for diethyl sulphate and benzoyl acetic ester are appended (APP. 2, 3).*

Chloracetic acid—The monthly capacity was 300 tons, and the process employed is interesting. A solution of trichlorethylene is reacted at 130°-140°C. with water or aqueous sulphuric acid in a solution of about 50 per cent monochloroacetic acid in sulphuric acid of 97-98 per cent, while the monochloroacetic acid formed and dissolved in sulphuric acid is continuously run out of the reaction vessel. The hydrogen chloride evolved is used for esterifying methanol. The reaction vessels are of cast iron, jacketed for steam. The product is purified by continuous vacuum distillation.

(b) AROMATIC

Aniline—The conventional iron reduction process is adopted at Höchst and Uerdingen, and a catalytic reduction process at Ludwigschafen. Dr. Lanz, Head of the Intermediate Section, was of the impression that the catalytic plant at Ludwigschafen sometimes gave trouble, and aniline was often supplied to Ludwigschafen by Höchst. Dr. Lanz did not know the precise difficulties associated with the catalytic plant, but he personally preferred the ordinary iron process. At Uerdingen the iron oxide sludge was converted into pigments, but not at Höchst. Höchst's

*The appendices given here represent a selection of processes which have not been reported upon by other investigators.

capacity for aniline was 15 tons per day, using 5 kettles of 12 cu. m. There was no special specification for the iron employed, but this was necessary if the iron oxide sludge was to be ultimately employed for pigment manufacture. The iron used at *Hochst* came mainly from the *Opel* automobile plant in the neighbourhood.

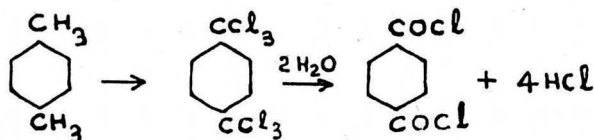
Chlorobenzene used to be supplied from *Bitterfeld*, where it was made by the ordinary batch process, but a continuous plant making 30 tons per month is now working at *Hochst* and 3 more units are under construction. The separation of chlorobenzene from dichlorobenzene is effected at *Griesheim*.

p-Nitraniline was made in 3 cu. m. autoclaves by amination of *p*-nitrochlorobenzene. It was stated that the process was not entirely free from danger, and a continuous process on a pilot-plant scale had been developed just before the war. No documents concerning the subject were available.

Phenylhydrazine was made by the sodium bisulphite, zinc and acetic acid reduction process. The hydrochloride was precipitated, filtered on a centrifuge, basified with aqueous caustic soda in presence of xylene, the xylene extract distilled *in vacuo* and the paste finally purified by vacuum distillation in a copper plant in an atmosphere of nitrogen.

m-Xylene & p-xylene hexachloride (*m- & p-bistrichlormethyl benzene*) — Complete side chain chlorination of *m*-xylene and *p*-xylene is carried out in a nickel reaction kettle with a glass ball installed in the cover for an "electric intensive lamp, 300 watts" (APP. 4).

Terephthaloyl chloride — The method was to hydrolyse *p*-xylene hexachloride with



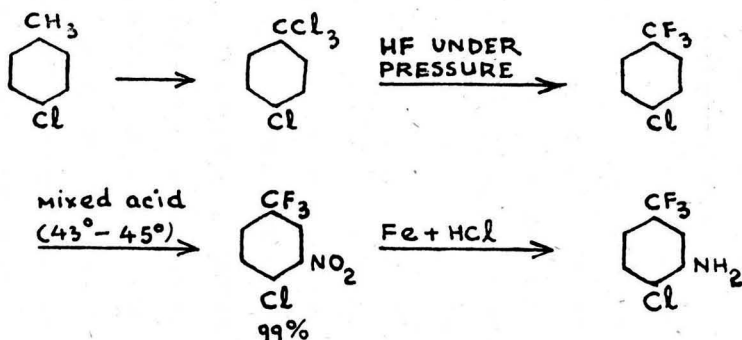
TEREPHTHALOYL bis ACETIC ESTER (APP. 3A).

water at about 130°C. in a lead-lined steam-jacketed kettle. The iron condenser was lined with bakelite, and hydrogen chloride was absorbed in an Igelit-lined tower.

Benzoyl chloride was similarly prepared from benzotrichloride.

Acetoacet-arylides — The acetoacetic ester-aromatic amine condensation was carried out in xylene solution in an aluminium kettle provided with an aluminium coil and mild steel jacket. The oil jacket was heated by means of a steam jacket. An aluminium column and an aluminium condenser were used for the removal of the ethanol formed in the reaction. The base was dissolved in xylene fed through a pressure filter in an atmosphere of nitrogen. The product was filtered in a horizontal centrifuge in a nitrogen atmosphere, the basket being lead lined and further lined with brass cloth. The arylide was dried in a steam-heated aluminium dryer; an aluminium worm moved the product alternately from one end of the dryer to the other, and when the drying was completed, the material was discharged from one end.

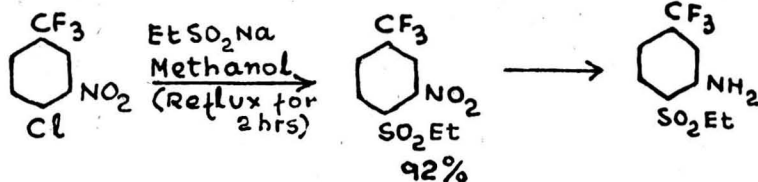
Fluorine Compounds — Dr. Scherer, Head of the Southern Section for Intermediates, had carried out considerable research on fluorine compounds as intermediates. Benzo-



FAST ORANGE RD BASE.

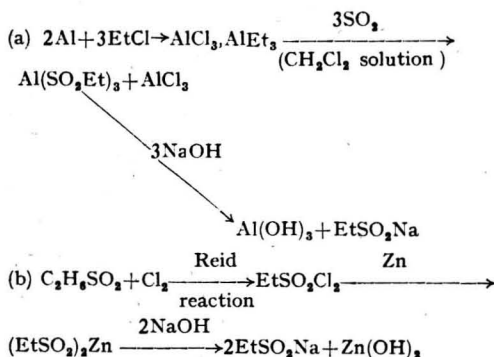
trifluoride was prepared by treatment of benzotrichloride with hydrofluoric acid under pressure, and other compounds containing the trifluoromethyl group were prepared by similar methods. Thus Fast Orange RD Base was prepared by the series of reactions given above.

Fast Golden Orange GR was prepared as follows:

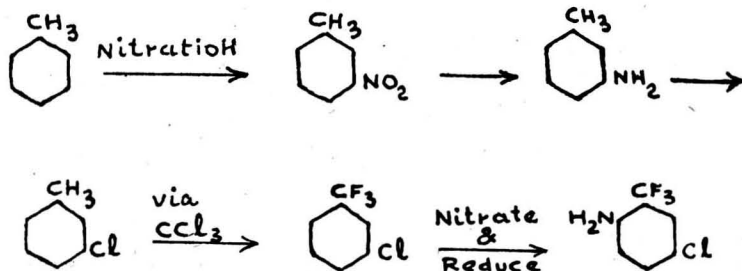


FAST GOLDEN ORANGE GR BASE.

The sodium ethyl sulphinate required in the above reaction was prepared by the following methods:



Fast Scarlet VD Base was prepared by the following series of reactions:



FAST SCARLET VD BASE.

When these bases were employed in azoic dyeing, the influence of the trifluoromethyl group was not merely to improve the clarity of the tone and the light fastness, but to reduce washing fastness. Several Indanthrene dyes containing the trifluoromethyl group were developed before the war, but only one was marketed commercially. This was Indanthrene Blue CLB, which was used for uniforms of the German Air Force, and was prepared as follows.

Variamine Blue B — The plant had a capacity of 30 tons per month. The sodium salt

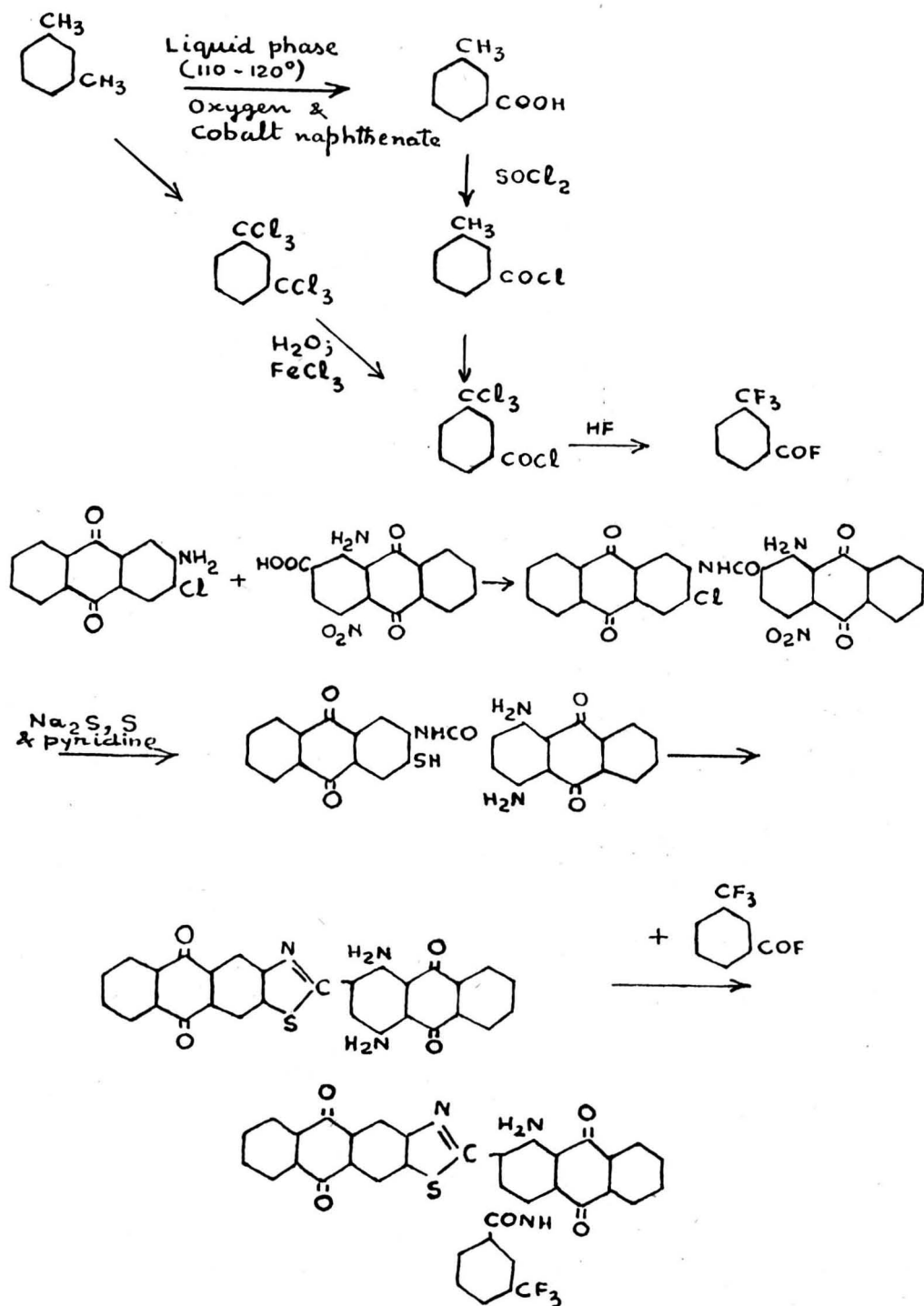
of 4-nitrochlorobenzene-2-sulphonic acid was obtained by salting out the sulphonation mixture. The sodium sulphonate was filtered off on a Nutsch, pasted up with water and heated in an autoclave at 110°-115°C. with

magnesium oxide and *p*-anisidine. The product was precipitated by sulphuric acid, filtered and reduced with iron and acid. The iron oxide was filter-pressed. The amine was salted out and then hydrolysed with 60 per cent sulphuric acid at 115°-123°C. Sulphuric acid was converted into sodium bisulphate by means of sodium sulphate and Variamine Blue filtered off on a rotary filter, and packed directly into drums for despatch to *Offenbach* for Fast Salt preparation.

Experiments on the preparation of Variamine Blue by condensation of *p*-nitrochlorobenzene with *p*-anisidine under pressure showed that better yields were obtainable by the *p*-nitrochlorobenzene sulphonic acid procedure.

Detailed processes for the following intermediates, fast bases, etc., are appended:

9. Blausulphochloride (*m*-dichlorobenzene-1-sulphochloride).
10. Blauthiosauric acid (2:4-dichlorophenyl-1-thioglycolic acid).
19. *p*'-Methoxyphenyl para-aminic acid (4-amino-4'-methoxydiphenyl-amine-2-sulphonic acid).
22. Amidol acid (1-amino-2-naphthol-4-sulphonic acid).
23. Diazoamidol acid (1-diazo-2-naphthol-4-sulphonic acid).
25. Nitroethylimide base (3-nitro-N-ethylcarbazole).
26. Carbazanil (diaminocarbazyl dichloroquinone).
27. Tetra' acid (naphthalene-1:4:5:8-tetracarboxylic acid).
29. Pyrenit S (nitropyrene).
30. Pyren chloride (hexachloropyrene).
31. Perchlorpyrene (tetrachloropyrene tetrachloride).



INDANTHRENE BLUE CLB.

32. Pyranil crude (dipyrenylaminodichloroquinone).
33. Pyranil pure.
34. Pyram (aminopyrene).
35. Leucopyranil.

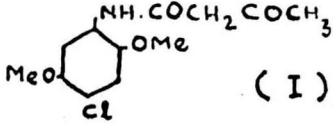
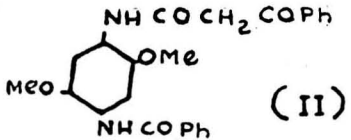
Alizarine was manufactured at *Hochst* 15 years ago, but the plant was later used for other purposes.

As Dyes—The azo shed at *Hochst*, which is modern and very well laid out, has been reported upon by a British team. There was no provision for the gravity flow of materials in the manufacture of *disazo* and *trisazo* dyes. Diazotization was carried out in wooden vats, and diazo solutions dropped into rubber-lined iron vessels from which the solutions could be blown out into a filter press. The coupling was carried out in wooden vats provided with rubber-lined iron agitators.

Pipe lines for the diazo solution were iron, rubber lined. For glacial acetic acid aluminium pipe lines were employed. The dyes were fed into the filter press by means of bronze pumps. "Paper cloth" was found to be suitable for the filter press. Pigments as well as dyes were filtered through filter presses.

Ice from the top floor was disintegrated in a simple type of disintegrator provided with spike rollers and dropped down chutes into rubber-tired hand carts of aluminium, from which they were discharged as required into vats directly or through chutes.

Rapid Fast Colours—The composition of various Rapid Fast colours was stated to be as follows; the base of which the anti-diazotate is used, and the Naphtol, are both indicated in the following table:

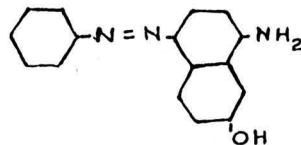
<i>Rapid Fast</i>		<i>Base</i>	<i>Naphtol</i>
Yellow	GGH GH	Red RC Scarlet GG	AS-G AS-G
	13 GH	Red TR	 <p>(I)</p>
Golden Yellow	IGH	Red RC	 <p>(II)</p>
Orange	GH IGH IRH RG RH	<i>m</i> -chloroaniline 4-chloro-3-amino-trifluoromethylbenzene do <i>o</i> -nitraniline Scarlet GG	AS-D AS AS-OL AS AS-PH (2-hydroxy-3-naphthoyl- <i>o</i> -phenetidine)
Gold Orange	IGG	2-amino-4-trifluoromethyl-phenyl ethyl sulphone	AS-D
Scarlet	ILH RH	Scarlet GG Red KB	AS-OL AS-PH
Red	B BB FGH GL G2H 13GL ILB IRH RH	Red B Red B Red KB Red GL Scarlet GG Scarlet 3GL Red RL Red TR Red RC	AS AS-D AS-D AS AS AS AS AS-RL AS-LT AS-OL

<i>Rapid Fast</i>		<i>Base</i>		<i>Naphtol</i>	
Bordeaux	IB	Red	B	AS-BOX (undissolved AS-BO)	
	RH	Red	RC	AS-BS	
Blue	B	Dianisidine		AS	
Brown	B	Mixture of dianisidine and <i>p</i> -nitraniline		AS	
	GGH	Scarlet	GG	AS-LB	
	IBH	Red	RC	AS-LB	
	IRH	Scarlet	GG	AS-BG	
Olive Brown	IGH	<i>m</i> -chloraniline		AS-LB	
<i>Rapidogen</i>		<i>Base</i>		<i>Amino acid used for stabilization</i>	<i>Naphtol</i>
Yellow	G	Red	KB	2-amino-4-sulphobenzoic acid	AS-G
	GS	Red	KB	Sarcosine	AS-G
	GG	Red	RC	do	AS-G
	14G	Red	TR	Methyl taurine	(I)
	PT			Diazoamino compound from <i>p</i> -toluidine	AS-G
Gold Yellow	IFC	Red	RC	Sarcosine	(II)
	IGL	Red	KB	do	AS-L ₄ G
	R	Red	KB	do	AS-G+AS-PH
Orange	G	Orange	GC	2-amino-4-sulphobenzoic acid	AS-D
	IGN		GCD	2-ethylamino-5-sulphobenzoic acid	AS-OL
	IRR		RD	do	AS-OL
Scarlet	R	Scarlet	GG	do	AS-PH
	IL	Scarlet	GG	do	AS-OL
	R	Red	KB	2-amino-4-sulphobenzoic acid	AS-PH
Red	RS		KB	Sarcosine	AS-PH
	GS		KB	do	AS-D
	G		KB	2-amino-4-sulphobenzoic acid	AS-D
	IGG	<i>o</i> -Toluidine-4-N-dimethyl sulphoramide		2-methylamino-5-sulphobenzoic acid	AS-LC
Bordeaux	ITR	Red	ITR	2-ethylamino-5-sulphobenzoic acid	AS-ITR
	R	Red	RC	Sarcosine	AS-OL
	IB	Red	B	2-methylamino-5-sulphobenzoic acid	AS-BO
	RN		RL	2-ethylamino-5-sulphobenzoic acid	AS-D
Corinth	IB	Corinth	LB	2-methylamino-4-sulphobenzoic acid	AS-LT
Violet	B	Violet	B	Sarcosine	AS
Red Violet	RR	2-nitro-5-amino-4-methoxy toluene		2-ethylamino-5-sulphobenzoic acid	AS-BO
Blue	B	Blue	BB	50% sarcosine, 50% methyl taurine	AS
	BN		BB	do	AS
Marine Blue	IB	Variamine		Sarcosine	AS
	D	Mixture of Blue Salts		do	AS-D
Blue	R	RR	Base	Methyl taurine	AS
Marine Blue do	B	Blue	B	do	AS
	R	Blue	RR	do	AS

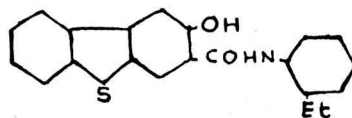
<i>Rapidogen</i>		<i>Base</i>		<i>Amino acid used for stabilization</i>	<i>Naphthol</i>
Green	B	Blue	BB	50% sarcosine, 50% methyl taurine	AS-BO
			BB	Sarcosine, N-methyl taurine	AS-CR
Brown	GN	Azored	Salt KBS		
			Azoblue		
	IB	Red	RC	Sarcosine	AS+AS-G
			RC	do	AS-LB
Olive Brown	IBR	<i>p</i> -toluidine	do	do	AS-LB
			do	do	AS-LB
Black Brown	ITR	Red	LTR	2-amino-4-sulphobenzoic acid	AS-LB
			LTR	do	AS-LB
	T	Red	RC	2-ethylamino-5-sulphobenzoic acid	AS-BT
			RC	Sarcosine	AS-BT
	IT	Azored	Salt RC		AS-BT+
			Salt RC		AS-ITR
Black	IT	Red	B	2-methylamino-5-sulphobenzoic acid	AS-SR+
			B	do	β -tetralol carboxylic acid- α - naphthyl- amide

MG Azoblue
Salt MG
and BD
(Blue B
and BB)

IG Red RL 2-ethylamino-5-sulphobenzoic acid



AS-SR+



Indigo White—Indigo production at *Hochst* was 400 tons per month of which 50 per cent was sold as Indigo White.

The process for Indigo White was by nickel reduction of indigo. The catalyst was prepared by reduction of nickel carbonate mounted on Fuller's earth.

Indigo reduction was carried out in a horizontal boiler, jacketed for steam, using 20 per cent indigo paste, caustic soda, the catalyst and hydrogen. The leuco solution was siphoned off from the catalyst, and the procedure then varied according as whether the product was to be marketed in the form of paste or grains. For paste the leuco compound was precipitated from solution by

carbon dioxide and filtered off in a filter press using jute cloth. It was then mixed with molasses and evaporated in a Frederking at 50°C. *in vacuo*. This was necessary, since the viscous paste had to be agitated with a powerful 6" diameter scraper agitator. The product was thus evaporated to 50 per cent Indigo White content and dried finally in a vacuum tray dryer.

For grains the leuco solution containing about 17 per cent of Indigo White was mixed with molasses, concentrated in a Calandria ("Wetkowitz heat exchanger"), and dried in trays in vacuum. The addition of molasses was adjusted so as to obtain grains of 50-60-70-80 per cent indigo content.

Indanthrenes

While detailed processes for a few Indanthrenes are appended, a few general observations are made. Aluminium chloride was stored and handled in lead-lined drums. For reactions involving aluminium chloride and sodium chloride mixtures vigorous agitation was necessary, and a propeller agitator was employed. The reaction was carried out in cast iron enamelled vessels, and the hydrogen chloride evolved was removed by using steam ejector suction. Hydrochloric acid solutions were handled in brick-lined vats and stone Nutsches. For moist hydrochloric acid vapours, pipes were of polyvinyl plastic. Iron pipes were adequate for gaseous chlorine, but where iron contamination was involved, lead pipes were used. In preparing chlorinated Indanthrenes the chloride used was checked up by merely weighing the chlorine cylinders, and no rotameters were employed. In a few cases a modified Pitot tube was used. For chlorinations on a comparatively small scale chlorine in cylinders was used, but for bulk chlorinations (all these chlorinations being carried out in the same shed) such as chlorinated paraffins and chloranil, where more than 100 kg. per hour of chlorine were necessary, the chlorine was led directly from the neighbouring electrolyser house.

Detailed processes for the following Indanthrenes are appended:

41. Brilliant Orange GR and Bordeaux RR.
42. Brown GR.
43. Turquoise Blue GK Powder.
50. Printing Brown B.
51. Printing Brown 3R.

Indigosols (Anthrasols)

The British method for preparing solubilized vat colours was employed, but it was claimed that the process has been shortened and improved. Recovered pyridine was dehydrated by azeotropic distillation with benzene and fractionated in a suitable column. Iron was used for reduction in most cases — iron from the needle industry for Blue BC and electrolytic iron in other cases. Copper was used only in one or two cases such as Anthrasol Red IFBB.

Detailed processes for the following Indigo-sols are appended:

- 61a. Anthrasol Brown IRRD.
- 61b. Anthrasol Brilliant Pink I₃B.
- 61c. Anthrasol Red IFBB.

Miscellaneous Chemicals — A point of interest in the preparation of heavy chemicals

at *Hochst* was the use of mercury cells for caustic soda and chlorine, of which a detailed account is given in *CIOS XXVI-II*.

Pure sodium sulphide was prepared by an elegant process in which sodium-mercury amalgam flowing out of the cells was treated with the calculated amount of sodium tetrasulphide, leading to pure sodium sulphide.

Tetranitrocarbazole — This substance is claimed to be a valuable insecticide for specific purposes. Nitration of carbazole is carried out in the usual manner, but a safety device is used for stopping the flow of nitric acid and blowing a siren in the event of the failure of the agitator. The temperature during nitration is electrically recorded.

For immersing the nitration product containing sulphuric acid, the vessel is of mild steel, lined with polyvinyl chloride and then with bricks. Cooling coils are of lead, the coils being lined with Igelit above the level of the liquor; this is because large quantities of nitrous fumes are evolved and corrode the lead. The thermometer pocket and the agitator are lead lined and given a top lining of Igelit. The product is filtered on closed stone Nutsches. The piping is again lead lined with Igelit. The Nutsch capacity not being adequate, part of the filtration is carried out by a filter press using polyvinyl chloride fabric. For separating any liquid carried over by fumes, an Igelit cyclone separator is provided. The mother liquor, the sulphuric acid content of which is too impure to justify recovery, is sucked off through a pipe and barometric leg straight into the drain. The Nutsch cake is thixotropic and is snuffed up with a little water into a kettle. The paste is dried at a temperature of about 160°C. on a drum dryer provided with brass knives. The cast-iron drums are built for 15 atm. pressure and steam is supplied at 6.5 atm. The dry product is fed into barrels by means of a worm conveyer. (On one occasion, on account of the conveyer outlet choking up, there was a rise in temperature to above 200°C. resulting in a fire.) The vapours from the dryer are washed with hot water to remove tetranitrocarbazole dust, and the water is used for pasting up the next lot of Nutsch cake. The nitrous fumes are finally absorbed in an Igelit column packed with Raschig rings, and provided with an Igelit suction fan.

The tetranitrocarbazole thus prepared has m.p. 284°C. and is marketed in this form. If a pure sample is desired, the crude product (10 gm.) may be dissolved in water (3 l.) at

100°C.; on the addition of N. sodium hydroxide (65 c.c.) the substance dissolves. Sodium bicarbonate (10 gm.) in water is now added and the precipitate filtered off. The red product now melts at 295°C. A yellow product is obtained by using ammonium chloride in place of sodium bicarbonate or by recrystallizing the red compound from dioxane.

A detailed process for tetranitrocarbazole is appended (APP. 62).

In spite of many attempts a practicable method for the synthesis of carbazole had apparently not been developed.

The methods for the estimation of the purity of carbazole isolated from coal-tar being unsatisfactory, a new method was devised. The principle of the method, a detailed account of which is appended (APP. 63) (Ref. *I.G. Analyse* No. 400), is to separate carbazole as N-nitrosocarbazole, which is decomposed on heating with ferrous sulphate solution, and the nitrous gases evolved are measured. Phenanthrene, anthracene and acridine do not interfere.

Difluorodichloromethane (Frigen) — A detailed process for Frigen (Freon) is appended (APP. 64).

Octadecyl isocyanate — A detailed process for this intermediate used in the preparation of Persistol VS, which is a useful finishing agent for textiles for the production of water-repellent and waterproofed effects, is enclosed (APP. 65).

II. — I. G. Farbenindustrie, Ludwigschafen

The object of the visit was to inspect the plant for *o*-benzoyl benzoic acid and its derivatives, β -aminoanthraquinone and Indanthrene Blue R, and to obtain information regarding a few intermediates and dyes.

Dr. Pflaumer, Director of the Works, mentioned that the entire factory had already been surveyed by several British and American teams, and one of the British teams, which stayed for four weeks, collected complete data on all vat dyes. A complete set of photographic copies of all the processes for intermediates and dyes employed at *Ludwigschafen* had been supplied to the American F.I.A.T.

m-Hydroxydiphenylamine — This is an intermediate in the preparation of Variamine Blue FG. A mixture of resorcinol (1,550 parts), aniline (1 850 parts) and sulphanilic acid (16 parts) is heated at 185°-190°C. for 45 hr., the temperature then being raised to 230°C. in 10 hr. Sulphanilic acid has now

taken the place of iodine which was originally used and which was not available during the war. The product is fractionated, *m*-hydroxydiphenylamine coming over at 220°-265°C. at 7 mm. pressure. The yield is 2,000 kg.

BOB Plant — The plant for condensing phthalic anhydride with benzene, chlorobenzene, etc., and cyclizing the resulting *o*-benzoylbenzoic acids is of a very modern design. A series of four 5 cu. m. vessels and one 3 cu. m. vessel are provided, and the same type of kettle is used for sulphonation of anthraquinone, the preparation of chloro- and methyl benzoylbenzoic acid, and the preparation of butyl phenol. The special features of the plant are large porcelain pipes, glass water-sprinklers and a continuous method for decomposing the Friedel-Crafts reaction product with water and for subsequent stages. For separation of aqueous and non-aqueous layers, horizontal separators are preferred, clearer separation being obtainable from vertical separators.

The aluminium chloride used in the plant is also made at *Ludwigschafen*.

The benzoylbenzoic acids are filtered on rotary filters and dried in a Buetner continuous dryer consisting essentially of revolving plates with large perforations, through which the material slowly travels down the series of plates until ultimately discharged.

β -aminoanthraquinone is made from β -chloroanthraquinone in an autoclave heated by internal steam coils (30 atm. steam pressure). Arsenic pentoxide is used in the amination. The product is blown over and filtered in a closed Nutsch, and dried on a Venuleth dryer.

2-amino-3-chloroanthraquinone is prepared by partial amination of 2:3-dichloroanthraquinone.

2:6-diaminoanthraquinone is made from 2:6-disulphonic acid, 25 per cent ammonia and arsenic pentoxide at a temperature of 200°C. and a pressure of 45 atm.

1:4-diaminoanthraquinone is made from quinazarin.

Indanthrene Blue RSN — Nickel-iron (0.5 per cent nickel) kettles are used for fusion. There is a screw conveyor for the introduction of β -aminoanthraquinone and an automatic arrangement for the addition of sodium nitrate. A mixture of caustic soda and caustic potash in molten form is fed in. For filtering the leuco compound, a closed-type imperial rotary filter is employed. The leuco compound is then mechanically

fed in through a worm into the oxidation vessel.

Chlorinated Indanthrenes — These are made in iron reaction kettles provided with Hoesch stirrers with a speed of 700-1,000 r.p.m. The progress of the chlorination is followed by loss in weight of the chlorine cylinders and by estimating chlorine in samples of the product.

Indanthrene Yellow G is prepared from β -aminoanthraquinone in 3 stages. In the first stage, 1-chloro-2-aminoanthraquinone is heated with phthalic anhydride in presence of ferric chloride to produce 1-chloro-2-phthalimidoanthraquinone. In the second stage, 2 mols of 1-chloro-2-phthalimidoanthraquinone are heated with copper powder to produce 2:2'-diphthalimido-1:1'-dianthraquinonyl. After removing the copper by means of sodium chlorate and hydrochloric acid, hydrolysis and cyclization are effected by heating with caustic soda solution.

Indanthrene Brilliant Green B — 4:4'-dibenzanthronyl is prepared by heating benzanthrone with caustic potash and sodium acetate in isobutanol solution. Oxidation of the dibenzanthronyl to 16:17-dihydroxydibenzanthrone is effected by means of manganese dioxide and sulphuric acid, and the final stage of methylation by methyl benzenesulphonate.

Violanthrone (Indanthrene Dark Blue BO) — In the fusion of benzanthrone with caustic alkali and sodium acetate, carbazole-free anthracene residue is used as flux.

Indanthrene Direct Black RB is prepared by the condensation of 1 mol of tetrabromopyranthrone with 2 mols of amino-violanthrone and 2 mols of α -aminoanthraquinone.

Detailed processes for the following are appended:

67. β -aminoanthraquinone.
68. 6-amino-1:9-pyrimidanthrone (Pyrimidin AFS).
69. Indanthrene Yellow 7GK.
72. Nitrocarbonsaure (1-nitroanthraquinone-2-carboxylic acid).
74. Indanthrene Blue RS.
- 74a. Indanthrene Blue BC.
- 75a. 4, 4'-dibenzanthronyl.
- 75b. 2, 2'-dihydroxydibenzanthrone.
- 75c. Indanthrene Brilliant Green FFB.
76. Benzanthronsulfid.
77. Isothren.
79. Dichinyl C.
82. Indanthren Red RK.
83. Indanthren Olive Green B.

III. — I. G. Farbenindustrie, Offenbach

The object of the visit was to inspect the plant for β -hydroxynaphthoic acid (BON acid).

The plant is very old and, except the carbonators for BON acid, nothing of special interest was noticed. The only hydroxycarboxylic acids made at *Offenbach* were BON, salicylic (on occasions) and *p*-hydroxybenzoic acid. The other acids used in the preparation of Naphtols came from *Leverkusen* and other I.G. plants. Yellow-producing Naphtols (G, LG, L₃G, L₄G) were not made at *Offenbach*.

The BON acid plant, though fabricated over 20 years ago, proved to be of great interest. The carbonators, of which there are 6, are heated by cast-in steam coils using steam at 25 atm. pressure, and are provided with powerful heavy duty agitators (1.5 r.p.m.). On account of the great resistance offered by the material to be stirred the agitators were liable to break down from time to time, and there was provision for immediate stoppage of the agitator by means of an electrical resistance indicator connected to the motor driving the stirrer. A feature of the process was that β -naphthol was continuously distilled off during carbonation. The process was stated to yield excellent results, but the plant was costly to construct and maintain.

2 small boilers (2 tons of steam per hour at 60 atm.) were reserved for process work, as the BON acid plant was a complete self-contained unit which included the production of carbon dioxide.

Stabilized Diazonium Salts — The drying procedure appeared to be unsatisfactory and the equipment crude in design. Tray dryers operated at 50°C., and provided with safety flaps in case of explosion, were employed. During grinding of Fast Salts with Glauber salt, aluminium sulphate, etc., dry ice was added. The initial size reduction of Fast Salts was carried out in disintegrators made of phosphor bronze to minimize the danger of self-ignition and explosion. After dilution with dry inorganic salts, Fast Salts could be ground in iron mixers consisting of revolving drums, provided with scraper blades working in opposite directions. Each mixer was 1 to 3 tons in capacity. The plant was provided with a very good dust-extracting system, the dust entering a tower sprayed with water before being discharged into the atmosphere.

For making Rapid Fast printing colours, special mixers with flat top, flush with the

floor level, and provided with spiral agitators were employed.

Detailed processes for the following are appended :

91. Nitrazol CF extra.
95. Echtschwarzsatz G (Fast Block Salt G).
96. Naphtol AS-SW.
103. Naphtol AS-LC.

IV.—I. G. Farbenindustrie, Mainkur

The main object was to see the Hydron Blue plant.

Hydron Blue — The plant was constructed in 1937. With a production capacity of 1,000 tons a year, 714 tons were actually produced in 1937.

p-Nitrosophenol — This was prepared in the known manner. A lead-lined horizontal centrifuge was employed for filtration; the basket was lined with copper fabric fastened with copper screws. *p*-Nitrosophenol was safe to handle in paste form, but was inflammable when dried.

For acidification at 0°C. a brick-lined vessel with wooden agitator was used. The solution was introduced in the sulphuric acid by means of a short pipe.

Carbazole-indophenol — The critical factor was the temperature. Cooling was effected by calcium chloride solution (d. 1.3) from an ammonia plant with a capacity of 60,000 k. cal. per hour at -40°C., supplied by *Gesellschaft f. Lindle's Eismaschinen, A.G., Hildastr., 6, Wiesbaden*. The indophenol was filtered in a wooden filter press using nitrated cloth or polyvinyl cloth.

The best tests for the quality of carbazole-indophenol were solubility in acetone and thionation. For dissolving carbazole and *p*-nitrosophenol and for the condensation of the two to form carbazole-indophenol, 6 reaction vessels are available of which 2 are provided with jacket and coils for cooling, while 4 have cooling coils only. One of the reaction vessels, reserved for condensation, has 4 banks of cooling coils reaching to the top of the vessel. The others have 3 banks of coils in the lower half of the vessel. Agitation is effected by means of propeller stirrers.

For the reduction of the indophenol, lead-lined iron kettles with wooden agitators, chutes for the addition of ice and large vent pipes are available.

The indophenol is washed in brick-lined vats and filtered on 2 filter presses.

Thionation is effected in butanol at a temperature of 108°-110°C. The iron thionation pot is steam heated and provided with steam pipes for quenching fires and for a little pressure for the discharging pump.

The solvent is distilled off after thionation, steam entering through 2 pipes, one inside the liquid, and the other touching the surface so as to break the foam. The distillation pot is also steam jacketed.

The drying of the dye is carried out in a circular chamber with glazed tiles provided with a disc spray dryer revolving at 7,000 r.p.m. A scraper plough working on the floor of the chamber discharges the material. The hot air outlet passes through filter bags to catch any dyestuff dust. The dryer is capable of evaporating 250 l. of water per hour, and yields 50-60 kg. of the dyestuff.

Suprafix dyes for printing are manufactured at *Mainkur*. They consist essentially of mixtures of a vat dye (sometimes as a leuco compound) and of Solution Salt B (sodium salt of N-benzyl sulphanic acid); a booster (such as anthraflavin or ferric sulphate) is also added, groups of dyes requiring specific boosters. The whole is then diluted with glycerin and water as required and colloidalized in a suitable mill. A typical procedure was to mix in a lead-lined vessel 30 per cent glycerin, 10 per cent Solution Salt B, 2.5 per cent ferric sulphate, 10 per cent tragacanth solution (6.5 per cent), 0.3 per cent Preventol and 10 per cent leuco compound of Indanthrene Pink R (or Alcol Orange RF) made up with water. The mixture is then worked in a colloid mill and sieved.

Detailed processes for the following are appended :

106. *p*-Nitrosophenol.
107. R-base.
114. Ethyl carbazole.
126. Nitrocarbazole.
129. Anthronaphthylen.
133. Dinasaure (1:1'-dinaphthyl-8:8'-dicarboxylic acid).
137. Pyrazolgelbkali.
138. Indanthrene Yellow GF.
140. Indanthrene Scarlet B.
142. Kupenbraun IVD (Vat Brown IVD).
147. Kupenscharlach RM (Vat Scarlet RM).
148. Indanthrene Brilliant Orange RK.
153. Sirius Light Yellow RT.
154. Diethyl Red.

V — Kalle & Co., Biebrich-Wiesbaden

Cellulose Glycollic Acid—Cellulose (wood pulp; 38 per cent cellulose, 6 per cent moisture) (1,000 kg.) in the form of sheets is treated with 18 per cent caustic soda solution in a rectangular iron box between perforated plates at room temperature for 1½ hr., and submitted (hydraulic press) to horizontal pressure for 1 hr. The weight of the sheets is 2,600 kg. The cage containing the sheets is then lifted and the sheets are dropped into a shredder. The alkali-cellulose consists of 14 per cent sodium hydroxide, 1 per cent sodium carbonate and 32-34 per cent cellulose, and is transferred by means of a worm into a Werner-Pfleiderer shredder mill. The shredded soda cellulose is blown by compressed air through pipes into 2 larger boxes from which it is discharged into wagons, then weighed and dropped into a Werner-Pfleiderer kneading machine together with sodium chloracetate. Each charge in the shredder consists of alkali-cellulose, corresponding to 500 kg. of cellulose, together with 375 kg. of sodium chloracetate. The temperature rises during the reaction and after 2 hr. the material is discharged into a box and therefrom into a rotary dryer, where it is worked for 4 hr. This crude product is Tylose HBR containing 33 per cent sodium cellulose glycollate, 33 per cent water. The material now passes down into a box on to a belt conveyer and is fed into a disintegrator. After passage through a cyclone separator to remove air, and through pipes, where it is blown by compressed air into a higher floor from which it drops through a hopper, the Tylose is finally weighed, and packed in paper bags which move forward mechanically.

Methyl Cellulose—Soda cellulose is prepared in the same type of plant as for cellulose glycollic acid. Soda cellulose is then either shredded, or the sheets are placed on a wire and rolled on a wheel into a package consisting alternately of cellulose sheets and wire.

Methyl chloride from tank wagons is pressed into a feeder autoclave where it is heated by steam coils and fed into a reaction autoclave by means of a centrifugal pump. The iron autoclave is nickel-lined, and the pump and pipes are of iron-nickel alloy. The charge per autoclave corresponds to 600 kg. cellulose and 550 kg. of methyl chloride, a large excess of methyl chloride being necessary on account of the formation of methanol of which 16-18 kg. are recovered per 100 kg.

of methyl cellulose. The autoclave is heated externally by steam first to 70°C. and then to 90°-100°C., the pressure being 5 atm. The reaction is carried out for 5-6 hr., the total cycle in the autoclave from loading to unloading taking 8 hr., so that each autoclave could handle 3 batches a day. Methyl chloride is circulated through the alkali cellulose, methanol is condensed, and the residual methyl chloride re-heated and circulated. Methyl cellulose is purified by agitation with hot water and filtration on a horizontal centrifuge. The material is dried in a rotary dryer. When the soda cellulose is used in the form of sheets mounted on wheels, a different type of autoclave is used.

APPENDIX 2

DIETHYL SULPHATE (made from ethyl sulphuric acid)

Principal Uses—Ethylating agent for pharmaceuticals.

Equipment—(1) 1 enamelled vessel, 3 cu. m., with jacket and enamelled stirrer; (2) 2 aluminium receivers, 1 cu. m. each, with a 5-stage steam jet pump; (3) 1 aluminium stock vessel, 4 cu. m.

Process: Diethyl sulphate—In (1), 1,100 kg. of ethyl sulphuric acid and 1,950 kg. of dry sodium sulphate are mixed and distilled into (2) within 12-15 hr. under a reduced pressure of 5 mm. Hg. The distillate is stored in (3). Yield: 580 kg.

Process Control Tests—Determination of the sp. gr. and strength of ethyl sulphuric acid and diethyl sulphate.

Raw Materials, Intermediates & Finished Product Specifications—Sp. gr. of ethyl sulphuric acid: 1.41 at 15°C.; sp. gr. of diethyl sulphate: 1.18 at 15°C.; content: 99.8 per cent of diethyl sulphate and 0.1 per cent of ethyl sulphuric acid.

DIETHYL SULPHATE (prepared from ethylene and sulphuric acid monohydrate)

Equipment—(1) 1 cast-steel autoclave of 1,000 l. with an admissible maximum pressure of 50 atm. gauge pressure; (2) 1 iron measuring vessel of 1,000 l.; (3) 1 iron measuring vessel of 1,000 l.; (4) 1 iron vacuum distilling vessel of 2.5 cu. m. provided with jacket heating; (5) 1 5-stage steam jet water pump; (6) 2 aluminium receivers each of 1 cu. m.; and (7) 1 aluminium stock vessel.

Process—527 kg. of sulphuric acid monohydrate (representing 307 l.) are filled into the vessel and 328 kg. of ethylene from a steel

bottle are pressed in. The vessel is then heated to about 70°-75°C. by means of warm water, the ethylene being absorbed rapidly. After about 12-16 hr. the main quantity of ethylene is consumed. The remainder is absorbed with difficulty. After about 20 hr. the pressure (5 atm.) is released. When the product has assumed the right density and acidity, the contents of the autoclave are pressed into another vessel. From here the reaction mass is brought in 2 equal portions into a vessel which contains 140-150 kg. of dry sodium sulphate and is distilled at 5-8 mm. reduced pressure in the course of 12-15 hr. The yield amounts to 630 kg.

Process Control Tests — The strength of diethyl sulphate is determined by saponifying it with a measured quantity of hydrochloric acid.

Raw Materials, Intermediates & Finished Product Specifications: Contents of the Autoclave — The sp. gr. at 20°C. amounts to 1.22-1.25; acidity: 10 c.c. of it correspond to 28-35 c.c. of N. sodium hydroxide.

Diethyl sulphate — The sp. gr. at 20°C. amounts to 1.18. Strength: 99.8 per cent of diethyl sulphate and about 0.1 per cent of monoethylsulphuric acid.

APPENDIX 3

BENZOYLACETIC ESTER

Principal Uses — Intermediate for making dyestuffs.

Equipment — (1) Iron condensing vessel, 7 cu. m., provided with coil and iron stirrer; (2) iron separating vessel, 5 cu. m.; (3) iron stirring vessel, 2.5 cu. m., provided with coil and cooler with continuous aluminium separator; (4) clarifying press; and (5) rubber-lined precipitating vessel, 2.5 cu. m., provided with rubber-lined anchor stirrer and cooling jacket and continuous aluminium separator.

Process — Into the condensing vessel are introduced 1,000 l. of water, 500 l. of Naphtha C, about 130 l. of caustic soda solution (33 per cent = 180 kg.), 390 kg. of acetoacetic ester and 500 kg. of ice, the temperature being adjusted to about 5°-8°C.

Into this mixture are run in 422 kg. of benzoylchloride and 540 l. of caustic soda solution (33 per cent) with such a speed that the temperature does not surpass 15°C. before half the quantity has run in. At the end of the running in the temperature may rise to 20°C. The reaction must be distinctly alkaline to thiazol paper. The whole is stirred for another hour and then heated to

35°C. Now the whole is pressed into the vessel (2). After 1½ hr. the aqueous alkaline solution is run into the vessel (3), stirred together with 10 kg. of kieselguhr and filtered through (4) into vessel (5). The clear alkaline filtrate is mixed with 160 kg. of ammonium chloride and stirred in the closed vessel during the night at room temperature. Then about 80-100 kg. of common salt are added in order to increase the sp. gr. of the aqueous layer to 1.13. The benzoylacetic ester is allowed to deposit in the course of 2 hr. and slowly separated using the separating vessel.

The yield amounts to 415 kg.

Recovery of the Naphtha — The naphtha remaining in the vessel (2) is introduced into the vessel (3), distilled off with steam through a cooler, separated continuously and can be used again in the vessel (1) without drying.

The yield amounts to 475 l.

Process Control Tests — The sp. gr. of the aqueous layer in the vessel (5) is tested. The strength of the benzoylacetic ester is determined by ketonic cleavage by means of alkali and weighing the carbon dioxide separated from the acidified mixture.

Raw Materials, Intermediates & Finished Product Specifications —

Benzoyl chloride :

sp. gr.	1.211
b.p.	198°C.
m.p.	-1°C.

Acetoacetic ester : commercial product.

Naphtha C :

b.p.	130°-190°C.
sp. gr.	0.88

Benzoylacetic ester :

sp. gr.	1.118/20°C.
b.p.	149°C./12 mm.

1,000 kg. Naphtha C is obtained as by-product per ton of finished product and is used again in the plant.

APPENDIX 3a

TEREPHTHALOYL bis ACETIC ESTER

Principal Uses — For dyestuffs.

Equipment — (1) Condensing vessel made of iron, iron stirrer, cooling jacket for brine, (3 cu. m.); (2) separating vessel made of iron, (5 cu. m.); (3) stirring vessel made of iron with coil and connected cooler with continuously operating separator, contents 7.5 cu. m.; (4) clarifying press; (5) rubber-lined precipitating vessel made of iron, with rubber-coated anchor stirrer and cooling jacket with connected cooler with continuously operating

separator made of aluminium (2.5 cu. m.); and (6) stone-ware suction filter with receiver.

Process — Into (1), containing 1,000 l. of water, 500 l. of Naphtha C and 340 l. = 348 kg. of aceto-acetic ester while stirring, 25 l. of caustic soda solution (33 per cent) are run in at a temperature of 10°C. Within 1½-2 hr., simultaneously 214 kg. of ground terephthaloyl chloride *tel. quel.* = 203 kg. of 100 per cent strength and about 355 l. of caustic soda solution (33 per cent) are introduced. The reaction must always be strongly alkaline to phenolphthalein, but not yet to Thiazol Yellow. The temperature is kept at 15°-20°C. by cooling with refrigerating brine. After stirring for further 2 hr. the mixture is separated in (2). The aqueous solution is mixed with 20 kg. of kieselguhr in (3) and filtered through (4) into (5). 68 kg. of aqueous ammonia (25 per cent) and 110 kg. of ammonium chloride are then added. Stirring is performed overnight and after further standing for 1 day the crystallized T-acetic ester is filtered off on (6).

Yield — 225 kg. of 100 per cent strength.

Recovery of the Naphtha — The naphtha remaining in (2) is distilled off in (3) by means of steam through a cooler, separated continuously and can again be introduced in (1) without being dried.

Process Control Tests — Terephthaloyl chloride: determination of the degree of purity by vacuum distillation.

Acetoacetic ester: commercial.

T-acetic ester: determination of the degree of purity by coupling with diazotized aniline-2.5-disulphonic acid in an aqueous pyridine solution at a pH 7 (double coupling).

Raw Materials, Intermediates & Finished Product Specifications —

Terephthaloyl chloride:

m.p.	75°-77°C.
b.p.	142°C./12 mm.

T-acetic ester:

m.p.	70°-71°C.
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1,900 kg. of Naphtha C is obtained as by-product per ton of finished product and is used again in the plant.

APPENDIX 9

BLAUSULPHOCHLORIDE

Principal Uses — For Blauthiosaure.

Equipment — Enamelled stirring vessel with jacket, contents 2 cu. m. rubber-lined, and brick-lined vessel with rubber-lined fittings and decomposing vessel, 9 cu. m. Suction filter built in masonry with wooden grate and cloth filter of 3 sq. m.

Process — Within 2 hr. 150 kg. of *m*-dichlorobenzene are run into the enamelled vessel containing 750 kg. of chlorosulphonic acid at 0°-10°C. Within ½ hr. the vessel is heated to 40°C., then maintained at this temperature for 3 hr. and cooled down again to 5°-10°C. Then the contents of the vessel are pumped within 3-4 hr. into the decomposing vessel which contains 1.5 cu. m. of water and 3 cu. m. of ice. After stirring for 10 hr. the sulphochloride is filtered on the suction filter and washed with ice water until neutral. The resulting product has a dry content of 80-90 per cent.

Process Control Tests — After finishing the sulphochlorination, a sample when placed on ice should not be oily; when adding rock salt to the filtrate, no separation of flakes should take place.

Raw Materials, Intermediates & Finished Product Specifications — *m*-dichlorobenzene: analysis by fractional distillation. Blausulphochloride is a slightly greyish crystalline product with a melting point of 52°-53°C. (pure: 55°-56°C.). For calculating the yield the dry content at 40°C. has to be determined.

(To be continued)

REVIEWS

Principles of Biological Assay, by C. W. Emmens (Chapman & Hall Ltd., London), 1948, pp. xv+206. Price 21s. net.

THIS NOTICE OF "PRINCIPLES OF BIOLOGICAL Assay" by Dr. Emmens following the reviews I have had the honour of giving in recent issues of this *Journal* of two books on statistical methods, namely "Industrial Experimentation" (by Dr. Brownlee)¹ issued by the U.K. Ministry of Supply, and "Statistical Methods in Research and Production with special reference to the Chemical Industry" (edited by Dr. Owen Davis)² issued on behalf of the *Imperial Chemical Industries Ltd.*, will perhaps serve to indicate the increasing scope and speed with which publications on the use of statistical methods are now being released. For long, these methods had been used for ascertaining economic magnitudes of the resources of a country; now they are also proving their worth for improving the efficiency of agricultural and animal husbandry practices, for increasing the quantity and quality of industrial production, and for planning strategy and greater security in national defence.

In his foreword to the book, Sir Perceival Hartley, formerly of the (British) National Institute for Medical Research, where the author of this book is working, quotes with evident satisfaction a continental appreciation describing England, the home of Fisher and Yates, Finney and Irwin among several other contemporaries, as "the home of modern biometry". Rightly so. But the United States of America, too, where Chester Bliss is now working, and wherefrom come also L. J. Reed, W. R. Thompson, C. E. Rice and others, have not been lagging. After the discovery, by Professor R. A. Fisher, of the statistical techniques specially suitable for extracting *information* from *small* samples, and for testing *significance* in variations among identifiable sub-divisions (the italicized words being used in the technical sense now associated with each of them), it was only a question of time for biologists, pharmacutists and statisticians to come together in the solution of

problems arising in the production of therapeutic substances, of fungicides, insecticides and many other products in order that there may be guarantee that official requirements are complied with (up to a measurable degree at least). This has happened, sooner and more effectively perhaps than expected, because of the official efforts of the Permanent Commission on Biological Standardization of the Health Organization of the League of Nations.

The establishment by this Organization of International Standards marked a turning-point in biological assaying, and the readers of this book can follow the stages of this progress from the earliest attempts at precise measurement of potency to the varying designs for assays and in the subsequent treatment of potency, to the varying designs for assays, and in the subsequent treatment of the results. In a brief space of about 200 pages, divided into 20 practically non-overlapping chapters, suitably illustrated with worked examples, the author has admirably succeeded in writing one single book which will "enable the research worker or routine analyst who is not already an amateur statistician to plan his own tests and analyse them without reference to other manuals". The foundations are of course the methods of analysis of variance, curve fitting and regression relationship between log (dose) and response, and the first 5 chapters are occupied with these. Randomization of test objects (Latin square design) is dealt with in chapter 6, the short-cut method of using polynomial coefficients is treated in chapter 7, while chapter 8 is given to the use of co-variance analysis (rather the relative ineffectiveness of multiple co-variance analysis for increasing precision), and chapter 9 draws attention to missing terms and Bartlett's test for homogeneity. Chapters 10 to 14 deal with estimation of relative potency, standard error of M and quantal response (discontinuous variation), while "combined estimate of potency" in groups of tests and fiducial limits of error are again dealt with in chapters 17 to 20. Probit analysis is particularly dealt with in chapters 15 and 16, while the author's general appraisal to "transformations" of the response methods is probably that what he states in

1. 1948, 7, 196.
2. 1948, 7.

para 19.5 (pages 194 and 195) — “transformations of the response may be employed to equalize variances, but are not always likely to be successful . . . (we) must bear in mind that it is rarely justifiable to attempt too much by way of statistical refinements . . . much better to repeat an assay than to spend hours trying out various transformations or tests for the rejection of aberrant responses and so forth”.

Such summary disposal of transformation methods in an otherwise admirable book is somewhat surprising seeing that much commendable work has been done using the angular transformation in biological assays. Without going too much into details (an illustration of this method appears in a paper by Knudsen and Curtis of the U.S. Federal Security Food and Drug Administration in the June 1947 issue of the *Journal of the American Statistical Association*, Vol. 42, No. 238, pp. 282-297), it may be said that the chief trouble in numerical work arises on account of the weighting factor, particularly if it involves an appeal to a normal probability curve term. In the method of angular (or square root) transformation, the weighting depends only on the number of animals used on each dose, and “a comparison of the calculation time involved shows that the probit method requires about 12 times as long as the angular transformation method involving graph and monograph” (Knudsen and Curtis). Again, in a recent paper published in September 1948 (that is after the publication of Emmens' book) in *Biometrics* (*A.S.A.*, Vol. 4, No. 3, pp. 197-210), W. R. Thompson “assays” several methods in use — the fundamental log (dose) — response curve, logistic curve, the integrated normal curve, etc., — and describes a situation in which, the active agent and the inhibiting agent do not react completely but some of each remains dissociated. In his own preface, too, this author (Dr. Emmens) has stated that he confines himself only to some of the basic principles which the design of tests and the treatment of results should follow.

To be self-contained in laboratory practice, this book has reprinted Tables I to V and Table IX of Fisher and Yates' *Statistical Tables for Biological, Agricultural and Medical Research*, and, in addition, a table due to Bliss (reproduced from *Quarterly Journal of Pharm. Pharmacol.*) giving for stated values of the expected probit, the maximum and minimum corrected probits, the range, and the weighting coefficients. These tables

are, however, incorporated in the text of each chapter with numbers following table numbers of worked examples. It may have been probably more useful if they had been collected at the end of the book and marked with exclusive numbers or letters. Sometimes, as in para 4.5 (dealing with the change of base of logarithms), there is too much of (unnecessary) detail. On page 115 the limits $0.124 \pm 0.0304 t$ is a misprint, though an obvious one, for $0.1214 \pm 0.0304 t$.

These minor features aside, I think this very stimulating and very effectively written book would be very welcome not only to pharmaceutical biologists, but to the orthodox statisticians also, although the author's preface opens with unnecessary modesty, with the sentence, that “this is not a text-book of statistics, or even of statistical methods of general application to biology”.

K. B. MADHAVA

Thermodynamics, by Edward F. Obert
(McGraw-Hill Book Co. Inc., New York),
1948, pp. ix+571. Price \$5.50.

THIS BOOK IS INTENDED TO SERVE, IN THE words of the author, “as a fundamental text in the fields of thermodynamics and heat power. The presentation is believed to be sufficiently rigorous to satisfy the students and to serve as an introduction to more advanced topics that may be given in the later course.” The main objectives in writing the book, according to the author, are: (1) “to stress those essential aspects of the subject that every practising engineer must thoroughly understand”; (2) “to emphasize the continuity of thought leading to the Second Law”; and (3) “to show that for many real situations the perfect-gas equation provides an adequate basis for study and analysis at least for engineers who use slide rules for computations”. The text covers a wide range of subjects. It deals with numerous problems concerned with the flow of fluids, mixtures of gases and vapours, thermo-chemical calculations, air-conditioning, psychometry, and also the gas-turbine. Each chapter contains, at the end, a useful list of suggested references and also numerical problems. A list of relevant symbols is also provided at the end of each chapter.

The book undoubtedly covers a very wide field and covers it well, but it is unlikely to satisfy all the demands of a serious-minded reader interested more in “understanding the subject”, than in merely acquiring

information about it. The presentation in the text in some places gets rather heavy and involved. It has not, speaking generally, that lucidity of exposition and crispness of style which a young engineer-student would expect of a text meant to make him understand and provide insight into the fundamentals of a subject which is so important to him. Witness, for instance, the definition of *dimension* given in the opening chapter of the book. It says: "A dimension can be defined as a name describing certain qualities or characteristics of an entity. In this sense all names are dimensions and an infinite number of dimensions is possible. To reduce this number, certain descriptions may be expressed in terms of other more basic descriptions (dimensions)". An unsophisticated reader can hardly be expected to derive much satisfaction or illumination from the above definition. (One would, perhaps, have expected the treatment of dimensions to run on lines somewhat similar to that in Guggenheim's instructive article on "Units and Dimensions" in a recent issue of the *Philosophical Magazine*.) Again, though the chapter describes Reynold's number, there is no mention either in the chapter or elsewhere in the text of those important groups of dimensionless numbers that find such a wide application in the theory of natural and forced convection.

As another instance of a statement likely to confuse the student, consider the following (page 110 of the text): "The Second Law is not restricted to interchanges of heat and work, but it is rather a broad philosophy on the behaviour of energy and energy transformations. Many statements of the Second Law have been proposed; one of the most famous is that of Clausius: 'Heat cannot, of itself, pass from a colder to a hotter system'. Here, the obvious is recognized: Heat cannot flow 'uphill'; that is, the potential gradient of temperature must be in the direction of the flow of heat. The thought of Clausius can be expanded to include not only temperature, but all intensive properties (potentials): 'Energy cannot, of itself, pass from a lower to a higher potential.'" The above statement, at any rate, as it stands, is unfortunate. It often happens in science that things which, on a superficial analogy, appear to be obvious, may not really be so. In discussing the Second Law, in fact, the opportunity should have been taken to inculcate in the reader that, in spite of any feelings he may have to the contrary, the statement that heat cannot, of itself, pass from a colder to a hotter system,

is far from being obvious. If there was only one method of transferring heat, the statement of Clausius would reduce to a tautology — it would merely become a criterion for determining as to which system was the hotter and which the colder of the two. However, — and herein lies the significance of the Second Law — as there are various methods of transferring heat, the Law asserts that no matter what method one employs, heat, of itself, will never flow from a colder to a hotter body. Again, the student should have been warned in assigning any significance to the superficial analogy between temperature and potential. The concept of temperature is not analysed adequately enough in the text.

Frankly, the book is not likely to satisfy a reader who is interested in the study of the fundamentals of the subject. The book abounds in applications of the subject, provides valuable information, but for the fundamentals the reader will have to go to books by Planck, Ewing and Fermi, to mention a few names. Apart from this, the book under review will prove a useful addition to the existing large literature on thermodynamics. The charts at the end of the book, which cover a wide range of topics (for instance, the values of the total heat content of saturated air given in chart 5), will be of special interest to students of engineering and applied physics.

D. S. K.

The Chemistry of Acetylene and Related Compounds by Ernst David Bergmann (Interscience Publishers Inc., New York), 1948, pp. 108. Price \$ 3.00.

THE BOOK REPRESENTS A COURSE OF 3 LECTURES delivered at the Polytechnic Institute of Brooklyn in 1946. Although the author regards the book only as an introduction to a vast field, he has in fact succeeded in giving a remarkably clear and comprehensive picture of the reactions and transformations of acetylene and its derivatives. Much work has been carried out in recent years on the chemistry and technology of acetylene, which has immense possibilities as a starting material for the synthesis of a vast range of organic compounds. The applications of acetylene to the synthesis of organic intermediates, mainly due to the work of Reppe, have been described in several of the British and Combined Intelligence Sub-Committee Reports on the *I. G. Farbenindustrie*. The large-scale utilization of acetylene for

chemical synthesis has become possible by the methods developed by the *I.G.* for handling acetylene at high pressures and temperatures. The present book is an invaluable supplement to the technical reports, being an able and stimulating account of the mechanism by which acetylene functions in a variety of reactions. While a notable feature of the book is the very complete documentation, it is not a review, but a critical discussion which includes a considerable amount of unpublished work carried out by Dr. Weizmann, Dr. Bergmann and their collaborators.

The first lecture begins with a thermodynamical consideration of the basis for attempting to make acetylene by high temperature cracking of hydrocarbons such as methane, and proceeds to discuss the formation of diacetylene as a by-product in the German electric arc process for the manufacture of acetylene from natural gas. The behaviour of acetylene and its derivatives towards the halogens, hydrating agents, and in vinylation of alcohols, phenols and amines are then discussed. Among other reactions, an unpublished observation that acetylenic alcohols add to diene hydrocarbons to give alcohols of the dihydrobenzene series is mentioned. The metal derivatives of acetylene are finally reviewed.

The addition of nitrogen compounds to acetylene is further elaborated in the second lecture. Charts of Reppe's acetylene-formaldehyde and acetylene-acetaldehyde condensations are accompanied by a treatment of the reactions and products. Weizmann's studies of the reaction of acetylene with ketones, using as condensing agent potassium hydroxide dispersed in an acetal or a dialkyl ether of an ethylene glycol, and of the transformations of the acetylenic carbinols and diols are described. The lecture concludes with a reference to the fact that acetylene provides us with all the substances, except for isobutylene, required for producing the known synthetic rubbers.

The third lecture commences with a further discussion of the properties of the system potassium hydroxide plus solvents such as acetal or glycol dialkyl ethers. The polymerization of acetylene is then considered, and the mechanism of the formation of vinylacetylene, divinylacetylene and the higher polymers is explained. From the reactions of various acetylenic compounds it is shown that the triple bond can participate in a conjugated system with a double bond to cause resonance phenomena. The polymerization

of acetylene to cyclo-octatetraene and the properties and reactions of the latter are discussed. There is a brief account at the end of acetylene derivatives occurring in nature.
K. V.

Paramagnetic Relaxation, by C. J. Gorter (Elsevier Publishing Co. Inc.; Cleaver-Hume Press Ltd., London), 1947, pp. vii+127. Price 12s. net.

THE BEHAVIOUR OF PARAMAGNETICS IN STATIC magnetic fields has been the subject of extensive study since the days of Faraday. Both theoretical and experimental work has laid, well and truly, the foundations of our knowledge of this branch of magnetism.

The study of the properties of paramagnetic substances in a magnetic field which varies harmonically (with frequencies of the order of 10^6 Hz.) has been comparatively of recent origin. Investigations lead to 2 terms. The first is the high frequency magnetic susceptibility whose dependence on the applied frequency is called paramagnetic dispersion. The second term characterizes the paramagnetic absorption of energy from the high frequency magnetic field. Waller's paper in 1932 first gave a detailed theory of paramagnetic relaxation. He discussed in this paper the dependence of relaxation time on the strength of the magnetic field and temperature.

Extensive experiments have been carried out on paramagnetic absorption and dispersion by 3 independent methods. The first is the heterodyne beat method developed mostly in Leyden; the second method is a calorimetric one used extensively at Amsterdam. Finally, detailed investigations have been carried out at Leyden by the bridge method for the simultaneous determination of paramagnetic dispersion and absorption.

In the book under review, these problems have been treated in detail. The first chapter gives a historical account of the subject and makes clear the fundamentals involved in the study of paramagnetic relaxation. Thermodynamical considerations lead to a relation between adiabatic magnetic susceptibility and ordinary or static susceptibility. At low frequencies the susceptibility tends to the static value while at high frequencies the adiabatic value is obtained.

The second chapter gives full details of the 3 experimental methods stated above. The advantages and disadvantages of each of these methods are fully discussed. A review

of the results obtained by these methods with several substances is given in chapter III.

A résumé of the theory in the light of experimental evidence is presented in chapter IV. The theories of spin and lattice relaxations are considered in detail. The exchange of energy between the magnetic ions and the lattice is considered either as a lattice vibration quantum absorbed or emitted by the ions or alternatively as a vibration quantum being scattered non-elastically by the magnetic ions — a sort of a "Raman" process. The first order process preponderates at liquid helium temperatures while "Raman" processes are more frequent at liquid air temperatures. These theoretical considerations constitute the basis of the theory of paramagnetic relaxation.

Chapter V presents a discussion of the results outlined in chapter III on the basis of the theory given in chapter IV. It is well recognized that a qualitative understanding of the phenomena involved has been reached in certain respects. Several problems which need further study and elucidation have been briefly stated at the end of the book.

The book under review presents, in a concise and lucid form, full theoretical and experimental details of this new and rapidly growing branch of paramagnetism. Investigators in the field of magnetism and allied subjects are sure to find much in the book that would be of special interest and importance.

S. R. R.

Colorimetric Methods of Analysis, Vol. I, 3rd edition, by F. D. Snell & C. T. Snell (D. Van Nostrand Co. Inc., New York; Macmillan & Co. Ltd., London), 1948, pp. xii+229. Price \$ 4.50 or 25s. net.

THE BOOK UNDER REVIEW IS THE THIRD edition of the first volume of the authors' comprehensive treatise on *Colorimetric Methods of Analysis* and includes some important nephelometric, turbidimetric and fluorometric methods. The latest edition of the entire treatise is divided into three volumes. The present volume, the first of the series, covers treatment of theory, instruments and pH . The second and third volumes deal respectively with inorganic and organic applications of the methods.

The treatment of the subject-matter in this volume is rather of a condensed nature, as it aims at a complete treatment of all instruments and methods. As such, it will

not be easy for those, who are not already more or less familiar with these methods and instruments, to follow their description for practical work. But as more or less complete and up-to-date references to original literature have been cited in all cases, those interested in the subject will undeniably find the book to be of great help.

Colorimetric methods, together with its newer development in the form of nephelometry, turbidimetry and fluorometry, are acquiring ever-increasing usefulness and importance as powerful and speedy means for the estimation of minute quantities of substances not only in the field of chemistry, but also in various other allied branches of science, particularly in biology, physiology, soil science, etc. In the assay of vitamins and certain other biological materials, colorimetric methods are the only available means of investigation. They are also being extensively used as controls in the manufacture and standardization of many industrial products related particularly to foods and drugs. The present volume will, therefore, not fail to receive a wider welcome than what might have otherwise been restricted to the circle of analytical chemists only.

After a general but brief account of the various types of colorimetric methods, a short discussion of the essential theory behind these methods, dealing with the physical laws of transmission and absorption of light, has been added. This is followed by a somewhat detailed account of the various methods including spectrophotometry, filter photometry, and photo-electric methods together with a short description of the various types of instruments employed in each case. An entire chapter has been devoted to the description and use of glass filters. Then an account of nephelometry, turbidimetry and fluorometry with a description of the different instruments employed for the purpose has been added. A chapter hereafter deals with the factors limiting the accuracy of colorimetric methods and the typical sources of errors involved. Then follows a short chapter on calculations for different types of methods. A considerable part of the book has, however, been reserved at the end for the discussion of colorimetric methods for determining hydrogen ion concentration or pH values of solutions with the help of indicators. This includes an account of the theory underlying the methods and deals with the sources of errors and accuracy in each case; preparation of various buffer

solutions and indicators have also been included, followed by a somewhat detailed description of the preparation of samples for pH determination; the book then closes with an account of the different methods of pH measurement describing the procedure to be followed under each.

Author and subject indices at the end have been carefully prepared. The printing and get-up leave nothing to be desired.

There are a few misprints or errors which have come to the notice of the reviewer. Of these, the following may particularly be mentioned :

On page 65, "transmittance= I_1/I_2 " should be inverted and written as I_2/I_1 .

On page 143, 1.14, "0.1000" should be 1.0.

On page 159 in eq. (11) at the bottom, " $(1-\alpha)K_m$ " should be replaced by αK_i .

On page 210 in the last line over Table 44, the words "sample" and "standard" should better be exchanged; the same holds good also for the words "initial" and "final" on page 217, in line 8 from the bottom.

The deduction of eq. (13) on page 161 is also not quite clear.

P. RAY

Handbook of Textile Industry, by Sir Padampat Singhanian (Bureau of Economic Research, Kanpur), 1948, pp. xxxii+542+iii. Price Rs. 15.

INDIA'S PREMIER INDUSTRY — THE TEXTILE industry, is by far the single largest industry of the country with a block capital of Rs. 100 crores. The industry employs nearly a million people in its different stages. The total value of the annual production by the industry exceeds Rs. 400 crores. All the above facts are exhaustively annotated in this handy publication.

This collective publication compiled by a group of economists, technicians and industrialists and edited by the Director, *Bureau of Economic Research*, Kanpur, is comprehensive and up to date from every point of view — statistical, financial and technical.

In addition to cotton textiles, the book contains an authoritative account of other textile materials: woollen, silk, jute, flax, rayon, etc. Auxiliary requirements for the industry such as mill stores, machinery, spare parts, chemicals are dealt with in the publication.

The chapter on "Post-war Planning" is a lengthy one, and includes summaries of Industrial Panel Reports, both published and unpublished. The statistical section is an elaborate one and gives several useful tables.

The financial aspects of the industry have received special attention. The balance-sheets and working results of over 500 leading textile concerns (for periods extending from 4 to 7 years) are critically analysed. Other topics discussed under this section are cotton consumption, labour employed, capital structure, profit earning, etc. This chapter will be of special interest to the investing public.

The "Who is Who" section with specially prepared write-ups and photographs gives biographical sketches of all those personalities who have contributed to the growth and stability of the textile industry. The proceedings of the last four sessions of the *All-India Textile Conference* with special emphasis on some of the important pronouncements made during the conference by leading textile technicians and industrialists are included.

The Handbook is an indispensable reference volume to all connected with the textile industry.

A. K.

NOTES & NEWS

Estimation of Lactose

THE EXISTENCE OF UNCERTAINTIES in the tables employed in the determination of lactose, alone and in the presence of sucrose, by the method of Munson and Walker has led to the redetermination of the sugar-reducing values from which a set of tables have been computed (*J. Res.*, 1948, 41, 211). The copper equivalents of lactose determined range from 7.7 mg. up to 342.0 mg. alone and in the presence of 6.6 mg. to 355.8 mg. of sucrose. The method employed is that of Munson and Walker (*J. Amer. Chem. Soc.*, 1906, 28, 663).

The lactose used was prepared from U.S.P. lactose. A hot 50 per cent solution was treated with vegetable char and filtered. Aluminium hydroxide was then added, solution filtered and lactose crystallized. The material was re-crystallized, washed with water at 5°C. and dried at room temperature in a desiccator. Analysis showed: moisture, 5.05 per cent; ash, 0.003 per cent; and $[\alpha]_D^{20} = 52.54^\circ$. The data indicate that the material is lactose monohydrate, $C_{12}H_{22}O_{11} \cdot H_2O$. The sucrose used was *National Bureau of Standards*, sample 17.

The basic conditions for the determination, as given by Munson and Walker, were followed. The Soxhlet reagent contained 34.639 gm. of copper sulphate, $CuSO_4 \cdot 5H_2O$, in 500 c.c., and 173 gm. of potassium sodium tartarate ($KNaC_4H_4O_6 \cdot 4H_2O$) and 50 gm. of sodium hydroxide in 500 c.c. All solutions were measured at 20°C.

In the original method the solution was heated over a gas flame, but in this work electrical heating was substituted. The heater was such that the solution in the beaker was practically surrounded by the heating element and the current was controlled by a constant voltage regulator and a variable ratio transformer. The solution could be brought to boil in the required time within ± 5 sec.

The determinations were made as follows: 50 c.c. of the solution containing lactose were transferred to a 400 c.c. beaker containing 50 c.c. of the mixed Soxhlet

reagent. The solution was heated to the boiling point in 4 min. and then allowed to boil 2 min. longer. The cuprous oxide was transferred immediately to a Gooch crucible and washed with water at 60°C. It was then dissolved by adding 5 c.c. of 1:1 nitric acid, and the crucible was quickly covered. Before the addition of acid a small quantity of water was added to the crucible to minimize the evolution of nitric oxides. The copper nitrate and washings were received in a 250 c.c. beaker to which 10 c.c. of 1:1 nitric acid and about 5 gm. of ammonium sulphate had been added. Sufficient water was added to cover the cylindrical platinum-gauze electrodes, the total volume of electrolyte being about 180 c.c. The electrolysis was conducted overnight at room temperature and at an approximate current density of 0.10 amp./dm². Upon completion of the deposition, the electrolyte was replaced by distilled water before the circuit was broken. The copper was washed with alcohol, dried for 15 min. at 100°C., cooled in a desiccator and weighed. All deposits were bright and showed no evidence of oxidation.

Controls for Testing Textile Colour Fastness

A SIMPLE METHOD OF CONTROLLING the integrated exposure of fluctuating arc lamps employed in testing light fastness of textile dyeings is described (*J. Res.*, 1948, 41, 109). The procedure is to expose a piece of light-sensitive test paper, placed in front of an arc lamp along with materials to be tested, and terminating the exposure when a match with a standard, observable with the unaided eye, is obtained. The standard is a strip of the same batch of paper that has been exposed in a master lamp to a definite light dosage.

Carbon arcs are commonly employed for testing colour fastness of dyed fabrics and of stability of materials in general to light. The lamps usually employed are "Fade-O-meter" type. Since uniform performance of arc lamps is a practical impossibility, a method of exposure control by

estimating the radiant output with a light-sensitive material is a necessity. The method, based on standards produced with reproducible light dosages rather than reproducible batches of the light-sensitive media, has been found satisfactory. Freedom from difficulties of batch reproducibility, ease of recognizing the extent of exposure, reasonable assurance of uniformity within each batch, wide range of exposure periods available, and general standardization of procedure, constitute the main advantages of the method.

A special kind of paper was coated with a light-sensitive dye. A portion was standardized in a master lamp by exposing it to a convenient measured light dosage defined in terms of "standard arc hours". This standardized paper becomes a secondary standard to gauge light dosage in other lamps. In a typical application, a piece of light-sensitive paper is simultaneously exposed with materials to be tested, and when a match is observed with the unaided eye, the materials are assumed to have received corresponding light dosages. Estimating the degree of exposure is facilitated by auxiliary standard strips representing 80, 90, 110 and 120 per cent of the light dosage.

The actual time of exposure is immaterial unless it differs consistently by 30 per cent or more from the number of standard arc hours identified with the exposure, indicating that an adjustment of the lamp may be desirable.

The requisites of a light-sensitive paper are: colorimetrically stable in the dark, both exposed and unexposed samples producible in large uniform batches, normally responsive to temperature, arc characteristics and relative humidity, and visually sensitive.

2 samples produced at the *National Bureau of Standards* were found suitable. The papers were neutral (pH 6.8) and unsized. Paper 1444 was made entirely of wood pulp and paper 1370 entirely of cotton. The papers made from cotton fibres were considerably more uniform in fading rate than those made from wood pulp. These 2 papers were tested with 47 representative dyes and it was found that *National Aniline Niagara Blue G. Conc.*, lot 64704, colour index 502, and, to a lesser extent, *Du Pont Victoria Blue Conc.*, lot 22, colour index 729, gave the most useful products. By varying the dye, fibre and method of

application, papers of different fading rates were obtained. The samples had the following treatment and exposure characteristics:

(a) Paper C: Exposure time, 3 to 10 hr.; paper 1370, tub-dyed with 0.09 per cent Victoria Blue in 1:3, 95 per cent ethanol tap water solution.

(b) Paper E: Exposure time, 10 to 40 hr. Paper 1370, tub-dyed with 0.54 per cent Niagara Blue in tap water

(c) Paper F (or A): Exposure time, 40 to 100 hr. Paper 1444, tub-dyed with 0.65 per cent Niagara Blue in tap water. A somewhat lighter modification of paper F has been referred to as paper A.

(d) Paper S: Exposure time, 20 to 40 hr. This paper was beater-dyed by adding a solution of Niagara Blue to give a 0.5 per cent ratio dye: fibre, using the same kind of fibre as in paper 1444. Melamine resin (3 per cent on fibre weight) was added to the beater to give high wet strength for use in lamps with water spray.

These 4 types serve to indicate the methods of producing papers of various desired characteristics. The tub-dyed papers, which are finished papers run through a dye bath, must be used dry; the beater-dyed paper is non-leaching and may be used for "weathering" lamps, but is decidedly inferior to the tub-dyed papers in sensitivity. The latter have the disadvantage of tending to exhaust the dye bath which thus requires replenishing from time to time.

All the samples were uncalendered after dyeing and were found unchanged during storage for 8 to 18 months.

The standardized strips are mounted in a comparison booklet having flexible black cellulose acetate covers to protect the strips from soiling and light during non-use.

Although temperature has no effect on the fading rate of papers in the dark, it has considerable influence during exposure to light. Temperature specifications for papers should ensure uniformity of their performance. The light-sensitive papers showed excessive fading whenever exposure was interrupted through failure of arc in overnight runs. Other factors requiring standardization are: relative humidity, type of arc, and voltage.

With the master lamp it is not possible to obtain a precise fraction of a light dosage for auxiliary

standard strips because of variation of electrode resistance during the burning of carbon electrodes. For this reason light dosage is measured by means of an "exposure meter".

For reproducible results the carbon electrodes must be of uniform quality and size. The arc is enclosed in a 18 cm. Corex-D tube, and the arc chosen is one which burns in a current of air without depositing any residue. The arc lamp is housed in a special room at $25^{\circ} \pm 0.5^{\circ}\text{C}$. and 50 ± 1 per cent relative humidity in order to avoid seasonal effects. Regulation of arc current is necessary for reproducible performance of the arc. A device, which is essentially a contact-making ammeter, regulates the arc current at 30 amp. by controlling a reversible motor that regulates the arc gap. The line voltage is kept within a tolerance of ± 0.5 per cent by means of an induction voltage regulator, mechanical type. The line voltage is applied to a fixed transformer that operates the arc near 40 volts.

By limiting the tests to a definite portion of the electrodes, it is possible to obtain a measure of the reproducibility of the arc by relating radiant energy which was measured in turn by an exposure meter.

Synthetic Methanol

RECENT DEVELOPMENTS IN SYNTHETIC methanol production in the United States have been reviewed (*Ind. Eng. Chem.*, 1948, 40, 2230). The phenomenal growth of the industry is attributed to the accelerated demand for formaldehyde required in the plastics industry. 44 per cent of the output (total output 138 million gal. in 1948) is utilized in formaldehyde production and 38 per cent in the automobile anti-freeze industry.

The more important synthetic methanol plants are the *Du Pont* plant located at Belle, W. Va., and the 2 plants of the *Commercial Solvents Corporation* at Peoria and at Sterlington. The *Du Pont* unit, a by-product unit attached to the ammonia synthesis plant, was the first to go into operation. The carbon monoxide impurity was removed by methanation. This practice of running a single synthesis gas and steam through both methanol and ammonia converters is considered to be a major American contribution to the technology of methanol syn-

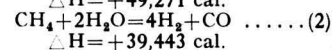
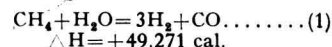
thesis. This practice is widely adopted in Europe.

The *Commercial Solvents Corp.*'s plant, also a by-product unit, uses fermentation gas from butyl alcohol-acetone units as raw material. The gas consists of carbon dioxide (60 per cent) and hydrogen (40 per cent). This installation was the first to use high pressure synthesis employing a specially developed catalyst. The Sterlington plant also operates on the same lines.

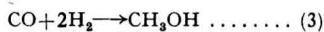
The raw gas for the Sterlington plant is obtained from the nearby Monroe gas-fields (about 50 miles) at a pipe-line pressure of 35-50 lb./sq. in. The gas is made up of methane (93.18 per cent) and unsaturated hydrocarbon gases. The gas is fed to the reformer furnace at a pressure of 27 lb./sq. in. The process gas is pre-heated in a gas-fired spiral tube ($225' \times 5'$ dia.) and passed through sulphur removing drums ($6' 6'' \text{ dia.} \times 28'$ deep) filled with extruded zinc pellets ($0.1875'' \times 0.5''$). The purified gas is mixed with steam at 30 lb./sq. in., which has been heated to 940°F . in a shell and tube heat exchanger. The ratio of steam to gas is mechanically adjusted, and mixing is accomplished by a simple T-joint. The combined gas and steam passes downward through a bank of 132 catalyst-filled tubes arranged in 6 sections in the reformer furnace. The catalyst consists of 0.75% ceramic cubes impregnated with finely divided metallic nickel. The furnace is gas-fired from the top and held at about $1,750^{\circ}\text{F}$. The flow of gas to the furnace is regulated by the temperature reading of a thermo-couple located in the reformed gas outlet where the temperature is held at $1,250^{\circ}\text{F}$.

The reformed gases pass immediately through a steam superheater where the temperature drops to about 950°F ., then into a boiler feed water-heater where it is cooled to 335°F . and finally in a bubble-cap tower cooler. The water stream in the cooler is controlled to hold the temperature below 120°F . to avoid corrosion.

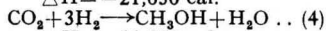
The reaction of steam and methane in the reformer follows the equations:



From these reformed gases methanol is formed according to the following equations:



$$\Delta H = -21,630 \text{ cal.}$$



$$\Delta H = -11,850 \text{ cal.}$$

For the products of equation (1) and (2) to react according to equations (3) and (4), excess carbon oxides have to be provided. At the Sterlington plant excess carbon dioxide is introduced along with the reformer gas. This procedure is wasteful as a third of the hydrogen generated is converted to water and methanol gets diluted. The recent practice is to exclude carbon dioxide and induce the reaction to proceed according to equation (3), the only drawback being excessive temperature in the catalyst mass which, if not properly regulated, results in methane reformation. Addition of carbon dioxide in the converter feed has a quenching action and affords a simple means of controlling the speed and temperature of the methanol synthesis reaction. Synthesis from monoxide gives about 95 per cent methanol, and the mixture of dioxide and monoxides gives about 80 per cent methanol.

The methanol converters contain catalyst-filled baskets of a special design patented by the *Commercial Solvents Corp.* They are so designed that the baskets can be lifted from the top of the converter shell by an overhead crane for convenient examination and replacement. The catalyst is a specially patented product by the *Commercial Solvents Corp.* and was developed first at the Peoria Works (French Patents: 635,023 and 636,337, 1927). Since the converters are estimated to have about 12 to 15 per cent conversion per pass, new make-up gas comprises only about 15 per cent of the feed.

The Sterlington plant, operating at about 5,000 lb./sq. in. and 400°C., is a relatively high temperature and low pressure operated unit. Current practice in methanol conversion makes use of 7,200 to 14,700 lb./sq. in. pressure and 250° to 400°C. temperature. The percentage conversion varies inversely with the temperature and directly with pressure. Optimum conditions are reported in the temperature range of 300° to 400°C.

Condensed methanol and unreacted gases from the after-cooler flow to a methanol separator where separation of the gases and methanol is accomplished and liquid methanol flows by mechanical liquid level control to the let-down drums from where it is

pumped through positive displacement meters to intermediate storage tanks.

An external refrigeration system, having a capacity of 130,000 B.T.U. per hr., is provided to condense the vapours vented from the let-down drums.

The crude methanol is distilled in a continuous refining unit consisting of 3 columns, the 1st one being a 45-tray column. Crude methanol, pre-heated, is introduced between the 17th and 24th tray in the 1st column. The column is heated by 35 lb. steam through a closed calandria cut in below the last tray. The 3rd column is the largest consisting of 75 trays; the feed is introduced at the 14th and 19th tray, and methanol is taken off between 67th and 73rd tray.

Extensive instrumentation and automatic controls are a feature of the plant. Most gas flow meters operate by measuring the pressure drop through a fixed orifice. Automatic valves are of the air-actuated diaphragm type. Temperature measurement is by thermo-couples. Pressures are measured by Bourdon tubes. All compressors are equipped with automatic cut-outs operated by lubricating oil pressure as well as emergency switches located at strategic points in the compressor house. Any or all of the compressors can be stopped instantaneously. A safety device also is installed on the reforming furnace which actuates an alarm if the furnace temperature exceeds 1,500°C. Automatic liquid level indicators are of the mechanical float type. Bubbler type direct reading indicators are used to indicate still reservoir levels.

Industrial Chromatography

CHROMATOGRAPHY HAS NOW BECOME a commercially feasible unit operation (*Chem. Met. Eng.*, 1948, 55, 133). In the separation of rare earth elements by this method, it is possible to separate the individual elements with high purity so that they contain less than one part per million of either of the adjacent rare earth neighbours.

The possible uses for chromatography are: (1) testing the homogeneity of a substance; (2) establishing the identity or non-identity of 2 substances; (3) concentration of a product occurring in natural sources at great dilutions; (4) separation, identification, estimation and isolation of the constituents of a

mixture; (5) purification, e.g. of a technical product; and (6) identification and control of commercial products.

The commercial adsorbents commonly employed are: activated carbon, alumina, silica gel, sugars, aluminium oxy-hydrate, magnesium oxide, calcium hydroxide, calcium sulphate, calcium carbonate, soda ash, Floridin, Fuller's earth, and the zeolites. The ion-exchange resins exhibit the same chromatographic separability that the physical adsorbents do. Moreover, these can be tailor-made for an individual job.

The design of chromatographic columns for batch operations is determined by the flow rate, particle size, column diameter and column height. The main calculable factor is pressure drop. The adsorption columns used by the *Valley Vitamins Inc.*, Tex., are 26" in dia. and 7' in height. Adsorbent supports consist of: first, a steel plate with ½" holes drilled on ¾" centres, then a 100-mesh stainless or monel screen, and finally a ¼" galvanized screen. Columns used by *Merck & Co.*, at Elkton, Va., are 36" in dia. by 12' of packed height. Bed supports for these columns consist of sieve plates and layers of fine mesh wire cloth. Pressure drop through these columns is 60 psig.

β-carotene, xanthophyll and chlorophyll are produced from alfalfa by *Valley Vitamins Inc.* The ground alfalfa is first extracted with hexane and the 3 pigments separated by chromatography. The resulting separate solutions are concentrated, and the products recovered for sale. 6 adsorption towers are in service. Normal practice is to charge the towers in pairs, each pair being charged in parallel. At a given time, one pair is being charged with the crude solvent mixture, the second pair is eluting xanthophyll and the third pair is eluting chlorophyll. Carbon black is the adsorbent employed. Hexane is charged at the bottom at a rate of 3 gal. per min. at first, and then the crude extract at the rate of 1 gal. per min. Operation is continued till lipoids appear in the discharge. The time taken is 126-155 min. Sight glasses are provided at the top of the tower. The charging of extract is stopped when the yellow colour of the carotene appears in the discharge. Hexane is pumped next for about 110 min. till the yellow colour of the discharge begins to fade.

The towers are next allowed to drain to avoid contamination by hexane of the hexane-isopropanol solvent used for the elution of xanthophyll. The solvent is pumped from the bottom at the rate of 2 gal. per min. The darkening of the sight glasses shows that xanthophyll has started eluting. The time taken is about 4 hr. for the xanthophyll to elute, which is indicated by the fading of yellow colour.

Benzene-isopropanol mixture is employed for eluting chlorophyll which is pumped from the top. The discharge is for about 8 hr., the bottom sight glasses showing a light green discharge. The tower is washed for another 8 hr. with fresh benzene-isopropanol thus completing the cycle.

Carotene extract which is contaminated with lipoids is subjected to a second adsorption step for the removal of lipoids. 4 more towers are employed for this purpose. Fresh hexane is pumped into the bottom of 2 towers at about 3 gal. per min. and when the solvent is flowing out through the sight glasses, the carotene extract is pumped in at the same rate until the lipoids appear in the discharge (tested manually by the greasy character of the discharge fluid). The feed rate is then reduced to 2 gal. per min. until a yellow colour appears in the discharge which takes about 45 min. Weak carotene eluate is pumped through until the yellow colour fades. The towers are washed with benzene-isopropanol solvent till the discharge shows no yellow colour and finally with hexane for about 90 min. to complete the cycle.

Merck & Co. uses chromatographic adsorption on alumina to produce highly active streptomycin. When a solution of crude streptomycin is passed through the column and then eluted with pure methanol, the activity of the effluent rises rapidly from 0 to a peak, and then tails off slowly. A solution of streptomycin hydrochloride in methanol is pumped through the columns at 60 psig. pressure into the top of the column, which is fitted with 100-200 mesh alumina. At the end of a given amount of feed solution, the pumping is stopped and the column is fed with pure methanol to elute the crude streptomycin at the top of the column. The effluent from the column is tested chemically by spot tests and when active material appears, the effluent is directed to

a receiver directly under the column. Spot chemical analysis is permissible despite the time it takes, because the entire cycle takes 24 hr. The column is re-vivified with distilled water. Channelling and faulty column operation is avoided by maintaining the column full of liquid at all times.

At present 2 major fields of the process industry — biochemical and petroleum refining — are actively employing chromatography. The biggest advance in the petroleum field would be the development of continuous chromatography. The up-grading of lubricating oil fractions by removal of aromatic constituents, and the production of pure organic chemicals from natural, cracked hydrocarbons, are 2 fields in which chromatography is likely to be employed on a large scale in the future.

Improved Methods of Sulphuric Acid Manufacture

A NEW THEORY OF SULPHUR dioxide oxidation has been recently developed and embodied in several patents by the Italian chemists: Guareschi, G. Maragliano-Buseti and Lucia Pettinati (*Chem. Age*, 1948, 59, 849).

An improved method has been envisaged for the production of oleum by catalysis, whereby plant and operation are simplified and considerable economy in cost and space is ensured. In this invention, the mixture of gases from the pyrites furnace, after purification, is liquefied. The mixture is reduced to $\text{SO}_2 + \frac{1}{2}\text{O}_2$ ratio and led into a heat recuperator attached to the pyrites furnace where the temperature is raised to 450°-480°C. Owing to the high concentration of gas, catalytic reaction takes place giving a high yield.

In another patent on oleum manufacture, instead of the SO_3 being absorbed by the acid in a gaseous state, it is solidified and dissolved in this form in a monohydrated sulphuric acid (98 per cent) solution. By this all bulky and costly plant parts, such as absorption towers, are eliminated. In the patent by Maragliano-Buseti and Pettinati for the intensive manufacture of sulphuric acid, nitrogen oxides are used as reaction catalysts and accelerators. Sulphuric acid is formed simultaneously with the recovery of liberated nitrogen oxides. The SO_2 is in intimate contact with nitro-sulphuric acid (nitrosyl sul-

phate) dissolved in sulphuric acid of over 60°Be. The reaction is at first vigorous and nitrogen oxides are set free in bulk and are oxidized immediately. By suitably varying the amount of nitrosyl sulphate, the composition of sulphurous gas mixture and the temperature, the two phases involved in the cycle become practically simultaneous.

Germanium Electronic Appliances

A GERMANIUM CRYSTAL RECTIFIER, which has been recently developed, is less liable to overload than the usual silicon rectifier (*Nature*, 1948, 162, 982). It can withstand a peak inverse voltage of 80 volts and deliver a maximum continuous current of 50 milliamperes; although its frequency response is inferior to that of silicon, it is very much superior to that of the metal oxide rectifier. As a detector or rectifier, it can replace the vacuum diode with considerable advantage being a non-heating device. A.C. hum, noise and thermal delay, from which all hot-cathode devices of the vacuum type suffer, are absent. The germanium crystals used in these high back-voltage crystal rectifiers are obtained from ingots formed *in vacuo* and slowly cooled.

A new 3-element germanium electronic device, termed "transistor" (transfer resistor) or semiconductor triode, has been developed at the *Bell Telephone Laboratories*. The device consists of a metal cylinder about an inch long and of the thickness of a pencil containing germanium and its 3 electrodes. The transistor is used to amplify telephone and television signals, and a standard model radio receiver with transistors in place of the ordinary valves, operates quite satisfactorily. A standard frequency note generated by an oscillatory circuit employing transistors has also been produced. The transistor uses less power than a vacuum tube, has an output of 25 milliwatts and can operate at frequencies up to 10 megacycles per sec. Owing to their simplicity, small size, performance, long life and probably low cost when mass-produced, these transistors should find many applications in all forms of electronic equipment.

The preparation of germanium of rather unique properties, of which the most striking is the absence of surface rectification at the germanium-metal contacts,

has been recently announced from the *G.E.C. Research Laboratories*. The germanium ingots were prepared by melting germanium powder under a pressure of less than 10^{-4} mm. of mercury. Its high magneto-resistance and variation of Hall Coefficient with magnetic field, in spite of the high degree of homogeneity, is a point of great interest and value in the designing of electronic equipment.

Spectral-transmissive Properties & Use of Eye-protective Glasses

CONSERVATION OF EYESIGHT IS one of the foremost problems confronting safety engineers today. They have found that protection of the eyes from excessive exposure to radiant energy is as important as protection from mechanical injury. Information on the transmissive properties of most of the widely distributed makes of tinted lenses is now available in a new circular, *Spectral-Transmissive Properties & Use of Eye-Protective Glasses*, recently issued by the *National Bureau of Standards*, U.S.A.

This publication is the result of a study of the spectral-transmissive properties of about 200 glasses intended for use either as sun-glasses or in special industrial operations, where injurious amounts of radiant energy are present. Extensive consideration is given to elimination of glare, colour distortion, use of glasses for night driving, standardization of glasses for outdoor and industrial purposes, and the spectral-transmissive properties of glasses for use under various types and intensities of ultra-violet, luminous, and infra-red radiation. It is proposed that sun-glasses be grouped into three or four shades having luminous transmittances of 60, 35, 25, and about 10 per cent respectively, values that correspond roughly to those for the industrial shades numbered 1.5, 2.0, 2.5 and 3.0.

A Measuring Device for Radioactivity

THE DEVELOPMENT OF A NEW AND compact device for measuring radioactivity was described by Dr. Benjamin Schloss of the *Nucleonic Sales Co.* at a symposium of the *American Chemical Society* at New York. The new instrument excels the Geiger counter and is expected to facili-

tate the use of tracer isotopes in medical research (*Chem. Age*, 1948, 59, 820). Described as a "crystal counter", the instrument utilizes a small crystal to trap and detect radiation in place of the comparatively bulky gas chamber of the Geiger counter. It efficiently records γ -rays.

Process Development Laboratories

THE 4 REGIONAL RESEARCH Laboratories operated by the U.S. Department of Agriculture, specially designed for process research, are in operation since 1940-41. The experiences of the chemical engineering staff in these institutions and their opinions regarding the design and operation of process development areas are described in a recent number of *Industrial & Engineering Chemistry* (1948, 40, 2014).

These laboratories were established with the specific purpose of applying the results of scientific and technological research to the utilization of farm commodities. Research is not restricted to the discovery of new uses for farm products, and considerable research on the improvement of established means of utilization is also carried on. A sum of \$4,000,000 is expended on these laboratories yearly. Each laboratory maintains a total staff of 300 persons, approximately 200 of whom are scientists and engineers of professional grade. The buildings are designed to house a research staff of this size.

As large-scale commercial application of research results is regarded as the criterion of success, emphasis has been laid, during the planning of buildings, on facilities for process development on a pilot-plant scale. From the first it was evident that no one plan for the pilot-plant space would serve equally well for all the 4 laboratories.

The laboratories are all 4-storey buildings constructed on the U-plan. The base of the U contains the administrative offices. One of the long wings consists of typical research laboratories, and the other the pilot-plant area. The length of the wings vary from 256' to 304' but the width is maintained constant at 64'. Because of special differences in research programmes, no 2 industrial wings are developed alike. All the laboratories, however, contain a large open area, 4 storeys in height, measuring 45' from floor to roof beams.

The pilot-plant area proper measures 112' to 208' in the different laboratories.

In the Northern Laboratory, space is set apart and partitioned for housing an alcohol pilot plant and this has a floor area of 96' x 64'. In addition, a separate specialized pilot-plant building has recently been erected to house large-scale experimentation units on production of motor fuels and other products by hydrolysis of agricultural residues such as corn cobs.

In the Southern Laboratory, experimental textile equipment is installed on 3 one-storey floors, each 96' x 64', and cotton treating equipment including kier boiler, calender, dryers, etc., are housed in the chemical wing (96' x 26').

After 7 years of varied operational experience, the engineering staff of these laboratories are of the opinion that 45' head room in the pilot-plant room is excessive and 22' are ample for the majority of operations. Another lesson is that distinctly different types of large-scale development cannot be successfully carried on together in the same room. In the Western Laboratory, large-scale food processing and chemical process developments were for some years located in the general pilot-plant area. This has been far from satisfactory. The design requirements for a chemical process room and food processing room are different. The latter must be built so that scrupulous cleanliness is not only possible but relatively easy to attain. Foreign odours, dust and fumes must be excluded.

The general pilot-plant areas are constructed at ground level with heavily reinforced concrete floors and direct truck access through a roll-up door for which a width of 15' is recommended. The solvent extraction plant is treated as an explosion hazard. In all the laboratories the 35' span between rows of columns is bridged by a 5-ton electrically operated travelling crane which has proved very useful for moving heavy equipment.

Provision for the supply of utility services to these areas was given most careful thought. High pressure steam (120 lb.) in a 2.5" line, city water in a 4" line, gas in 4" line are carried most of the way round the area. Shut-off valves in the loops permit isolation of a section during installation of a new connection without shutting down the entire area. Plugged tees at 16' intervals along the

loops make it easy for new connections.

The primary electric supply to the area is a heavy 208-v., 3-phase bus of 2,500 amp. capacity carried all the way around the walls in a grounded metal box below the second floor level. Heavy connections are made direct from the bus. Load centre boxes fed from the bus are mounted at intervals on the walls and conduit lines are run from the nearest load centre to lighter equipment. In addition, heavy duty single-phase and 3-phase plug receptacles are installed on each column. The general opinion is that the cost of the heavy bus loop has not been justified by experience. Electrical fixtures in solvent operation areas are of the type suitable for Class I, Group D, hazardous locations.

The entire floor area is pitched 0.125" to the foot towards floor drains which are located in the centres of each 16' x 12' rectangle except down the centres of the room. A slightly greater pitch has been found advisable to provide more convenience and comfort to the staff.

Steam condensate lines are located in pitched trenches which run along both side walls below the floor level. The trenches are covered flat with the floor level by flat steel plates with non-skid surface pattern.

The pilot-plant area is heated by means of low-pressure steam space heaters suspended below the second floor along the sides of the rooms. Ventilation is by means of 4 motor-driven exhausters to give one air change in 7 min. in the Southern Laboratory and 2 such are used in the Western Laboratory to give one air change in 15 min.

To provide maximum structural flexibility steel-grating balconies were included at the 3 elevated floor levels at the end of the pilot-plant area. In most of the laboratories these have been replaced by drained and curbed concrete balcony floors. This change has transformed the balconies into useful working spaces.

Various special service facilities have been installed in the pilot-plant areas. Though special provision is made for plants dealing with flammable solvents, it is generally agreed that a satisfactory location for such operation is a separate structure or out-of-doors. Safety equipment such as fire extinguishers, fire hose with fog nozzles, masks, fire blankets,

showers and protective clothing for workers are stressed as necessary, and proper location of these items deserve careful thought. The Eastern Laboratory has made extensive use of colour schemes for identifying pipe-lines making control valves and switches conspicuous, and marking out pedestrian aisles on the floor.

Although 120 lb. house steam supply is ample for most needs, 2 special installations have been found desirable. A small compressor is used to furnish high temperature steam to plattens of moulding presses. Odourless steam for food processing is supplied from a reboiler heated by house steam and fed with city water that is pre-heated to vigorous boiling in an open vessel.

Facilities for clean up include: portable vacuum cleaners with long handle attachments; water outlets provided with standard hose thread located at numerous places throughout the room; a booster pump supplying 16 gal. of water per min. at a pressure of 500 lb., permanently connected to a small line serving 2 or 3 places where particularly tough cleaning jobs are to be done, e.g. spray dryer; and steam-water mixing fixtures installed at a large number of places to provide hot water for washing. Safety of these fixtures for operators is assured by standard devices.

The pilot-plant areas have been used for several general types of activity and not merely for pilot-plant operations. They have been employed extensively for large-scale preparation work and for various kinds of engineering and technological research. The latter is particularly characterized, in part, by its dependence on the use of industrial types of equipment. Pilot-plant developments are undertaken only after careful analysis of the commercial prospects of a new process.

In each of the regional laboratories, from 20 to 30 employees of a professional grade, most of them trained chemical engineers, are concerned with experimental operations in the pilot-plant area. A locker and a shower room have been provided for clean up and change of clothes. It has been also found necessary to provide them with office space, outside the pilot-plant area, where they can keep their books and records and study or write without distraction.

Maintenance and emergency repairs are facilitated by keeping

small stocks of gaskets, belts, packing, nuts, screws and bolts and spare parts in chests or racks along the walls. The tools for assembling or cleaning a special piece of equipment should, if possible, be kept in a cabinet adjacent to the equipment.

A few standard chemical laboratories capable of accommodating 2 to 8 workers are located near the pilot-plant area.

It has been found that in the original design not enough general storage space was provided in these laboratories. Inactive equipment in the pilot-plant area obstructs working space, and leads to undue crowding of active equipment. It is recommended that at least 0.5 sq. ft. of proper storage area, under cover, should be provided for every sq. ft. of active pilot-plant area.

New Oxygen Isotope

THE SIXTH OXYGEN ISOTOPE to be isolated is Oxygen 14 which has a brief life-period of 76 sec., after which it turns into an excited nitrogen isotope. Both positrons and γ -radiations are involved in its decay. The other known isotopes of oxygen are those having atomic weights of 15, 16, 17, 18 and 19 (*Sci. News Letter*, Dec. 11, 1948).

Vitamin B₁₂ by Fermentation

THE ISOLATION OF VITAMIN B₁₂, the new vitamin used in the treatment of pernicious anaemia, has been reported from the laboratories of *Merck & Co.*, Rahway, N.J. Red crystals of the vitamin were obtained in the fermenting media using the mould *Streptomyces griseus*. This discovery may soon make the production of the vitamin in commercial quantities possible (*Sci. News Letter*, Dec. 11, 1948).

Use of Liquid Fuels

A CONFERENCE ON "MODERN Applications of Liquid Fuels" was recently organized at Birmingham by the *Institute of Petroleum* and the *Institute of Fuel*, at which modern trends in the design, operation and performance of burners, agricultural dryers, Diesel rail traction engines, the use of oil in the gas industry and the principles of combustion chamber design for gas turbines were discussed (*Nature*, 1948, 162, 951).

The suggested uses for liquid fuel in domestic heating are largely

based on the work of a special committee of the *Institute of Petroleum*. The fuels employed are liquefied butane and other gases, kerosene and Diesel oil. The development of modern domestic appliances burning kerosene was described and attention was directed to their high thermal efficiencies. Such appliances have many advantages in rural areas.

The conference was primarily concerned with the industrial application of oil fuels for which coal and petroleum may be used alternatively. In Britain coal is employed only because of its low cost, and the opinion is held that but for this advantage oil would be used in preference to coal. Oil has a higher calorific value, is easier to handle and store than coal, and is cleaner in use—qualities which account, for example, for the replacement of coal by oil for use at sea.

The oil flame has a higher radiation efficiency than the gas flame—a property which has contributed to the efficiency obtaining in the manufacture of steel in the open hearth furnace by conversion to oil-firing. The combined use of oil and coke-oven gas has given good results, and it was suggested that other combinations, including such fuels as coke breeze and powdered coal, might be used with advantage.

Another factor in the achievement of improved results with oil-firing is the greater ease of control which attends the use of oil. The use of oil in glass furnaces also gives the advantage of greater ease of control and increased rate of glass-melting, but the intense radiation from the oil flame results in increased wear on refractories and, at present, is not economical in comparison with efficient producer-gas system. The assessment of the relative merits of coal and oil-firing involves many technical and economic factors.

New Phosphating Compositions

TWO RECENT PHOSPHATING PATENTS relate to the use of double fluorides for coating metallic surfaces which have been found difficult to coat hitherto, especially surfaces which are predominantly of zinc or zinc alloy and iron (*Chem. Age*, 1949, 60, 20). These ensure better paint adhesion to coated surfaces.

To an aqueous solution of zinc or manganese dihydrogen phosphate (0.3 per cent metal) are

added a soluble salt of nickel (nickel sulphate, 0.3 per cent nickel), a fluoborate and preferably a nitrate or nitrite (NO_3 , 0.3 per cent). Immersion for about 1 min. at 150°F. or even 15 sec. is sufficient for electroplated and galvanized metal and high aluminium-zinc alloys. The same solution may be sprayed or run over the surface. The addition of fluoborate increases the etching tendency and is of special value in securing good paint adhesion on surfaces containing aluminium.

A second formula relates to phosphating of surfaces predominantly of aluminium or its alloys. Solutions containing an acid phosphate, fluoborate and an oxidizing agent as principal reagents are sprayed on to the surfaces to be coated. A replenishing solution is prepared by dissolving in water 1,295 lb. of phosphoric acid (75 per cent), 700 lb. of nitric acid (42°Be), 830 lb. of zinc oxide, 730 lb. of boric acid, 1,018 lb. of hydrofluoric acid (60 per cent) and 307 lb. of sodium fluoride.

Coconut Oil-base Detergent

A NEW COCONUT OIL-BASE SYNTHETIC detergent called Hi-10 is made by *Eureka Chemical Corp.* at Los Angeles. The product is odourless and non-soluble. It is obtained by combining coconut oil with amines. The result is a penta-valent nitrogen compound in which coconut fatty acids are combined with amines and amino-methyl compounds.

The new detergent has been employed to remove odour from railroad refrigerator cars in which fruits and vegetables are shipped. It can also be employed for general cleaning (*Chem. Met. Eng.*, 1948, 55, 196).

New Anti-oxidants

TWO NEW ANTI-OXIDANTS, TENOX HQ and Tenox BHA, have been developed by the *Tennessee Eastman Corp.* Added to fats and oils during processing, small percentages of the two compounds are reported to retard effectively the development of rancidity. Indications are that these compounds, used singly or in combination, will stabilize fats and oils throughout the normal time of processing, distribution and consumption. Toxicity tests have shown that the products are safe for human consumption when used in prescribed proportions. They do not

impair the nutritive value of oils or food products, and are economical for use.

Tenox HQ, a highly purified form of hydroquinone, is an effective stabilizer for salad oils, cooking oils, butter, margarine, orange and lemon oil extracts and dried milk. It prevents loss of vitamin A in oils containing it. Tenox BHA, butyl hydroxy anisole, is of special value in stabilizing animal fats, particularly lard. In combination with citric acid, it provides an adequate answer to the stabilization of lard for all uses including frying of potato chips, doughnuts and baking of crackers or pastry (*Chem. Met. Eng.*, 1948, 55, 198).

Sugar Refining

THE COST OF REFINING SUGAR CAN be greatly reduced by a new process which makes possible the recovery of vitamins now largely wasted (*Chem. Eng.*, 1948, 55, 306). The raw sugar of 95 per cent purity can be refined to 99.7 per cent purity by washing with wood alcohol. The valuable constituents are obtained in a residual molasses of pleasant flavour, and can be directly employed to supplement human diet.

U.S. Committee on Pacific Oceanography

THE FIRST MEETING OF THE Committee held during July 1948 was mainly concerned with American participation in the Seventh *Pacific Science Congress* to be held in New Zealand this year. Two symposia, one concerning conservation of marine resources, and the other on marine biogeographical provinces in the Pacific basin, are included in the programme. Another topic discussed at the meeting dealt with the need for standardizing gear and methods, and with methods and arrangements for correlating and integrating programmes of research in the oceanography of the North Pacific Basin.

A special report on the development of Hawaiian oceanographic research, the establishment of the Hawaii Marine Laboratory, and the initiation of a programme of research on tunas, was presented at the meeting. For the training of oceanic biologists, arrangements have been made with Rhode Island State College and its Narragansett Marine Laboratory to provide academic training and field experience for graduate

students prior to their assignment for study under specialists at Woods Hole Oceanographic Institution (*Pacific Sci.*, 1940, 2, 299).

Pacific Fisheries Investigation

A SUM OF \$1,000,000 HAS BEEN made available to the U.S. Fish and Wildlife Service through a U.S. Congress Bill to implement what is known as "Farrington Fisheries Programme". This programme provides for the "exploration, investigation, development and maintenance of the fishing resources; development of the high seas fishing industry of the territories and island possessions of the United States in the tropical and sub-tropical Pacific Ocean and intervening seas". The Bill authorizes U.S. Fish and Wildlife Service to secure laboratories, vessels and personnel to investigate the biological, technological and economic problems connected with tunas and tuna-like fishes. Oscar E. Sette, former Chief, South Pacific Investigations of the U.S. Fish and Wildlife Service, with headquarters at Stanford University, has been appointed Director. A new laboratory and administrative headquarters will be constructed at the University of Hawaii (*Pacific Sci.*, 1948, 2, 299).

Reflective Radiant

Conditioning

A NEW SYSTEM OF MAINTAINING year-round indoor-comfort has been reported from the University of Cincinnati. The new system is called "Reflective Radiant Conditioning". Walls and ceiling surfaces are embossed with aluminium foil and the heating equipment required is a few electrical resistance coils in each room for generating heat rays. Cooling coils, resistance wires for heat and fluorescent colour lighting are all installed in an alcove near the ceiling (*Sci. News Letter*, Dec. 11, 1948).

New Trends in Laboratory Glassware Manufacture

MANY BRITISH FIRMS, IN PARTICULAR, *Messrs H. J. Elliott Ltd.*, Treforest Trading Estate, Glamorgan, have introduced many improvements in the character and packaging of glassware. The firm is marking their volumetric glassware "E-Mil Green Line" complying with the *National*

Physical Laboratory, Grade A specifications and "E-Mil Gold Line" for Grade B standard. The graduations are filled with insoluble green and yellow enamels. The pigments are somewhat fluorescent and are easily visible in poor lighting even with coloured solutions, and the markings are fused into glass.

Stoppers in graduated flasks and cylinders have often proved troublesome owing to a propensity for "seizing". The new "E-Mil" stopper is a valuable accessory. Accurately moulded from an inert plastic, it provides a gas-tight and liquid-tight closure to an orifice of the *British Standards Institution* Specification taper, but does not bind or seize up irremovably.

The firm has introduced an innovation in packaging of articles to reduce losses in handling. Each article is sealed into its own specially fitting carton, which can be retained by the purchaser for safe storage of the article. The extra expenses of carton packing will be offset by dispensing with crates, etc. (*Nature*, 1948, 162, 974).

Plant Disease Forecasting Service

A NATIONAL PLANT DISEASE Forecasting Service has been initiated in America for the benefit of agriculturists. The warning service, which is an undertaking by the United States Department of Agriculture, 37 State agricultural colleges and 6 Canadian provinces, promises to bring farmers more money by keeping down crop losses and by preventing unnecessary expenditure on spraying and dusting for plant disease epidemics that do not develop. Disease forecasting helps to make better use of known prevention and control measures and provides timely warnings against outbreak of plant diseases.

The service was started in 1947 with a warning service on late blight of tomatoes following the disastrous outbreak of this disease in 1946. In 1948 the service was expanded to include late blight of tomatoes and potatoes, blue mould of tobacco and downy mildew of cucumber, cantaloupe and watermelon.

Disease outbreaks are reported by key scientists throughout the United States and Canada. Reports from these scientists go to the Plant Disease Forecasting Headquarters at Beltsville, Mary-

land. Emergency conditions are reported by telegram. A summary of these reports is dispatched to the Agricultural Insecticide and Fungicide Association, which in turn passes it on to manufacturers and distributors so that adequate supplies of dusting and spraying materials can be sent to danger areas and stored (*USIS*).

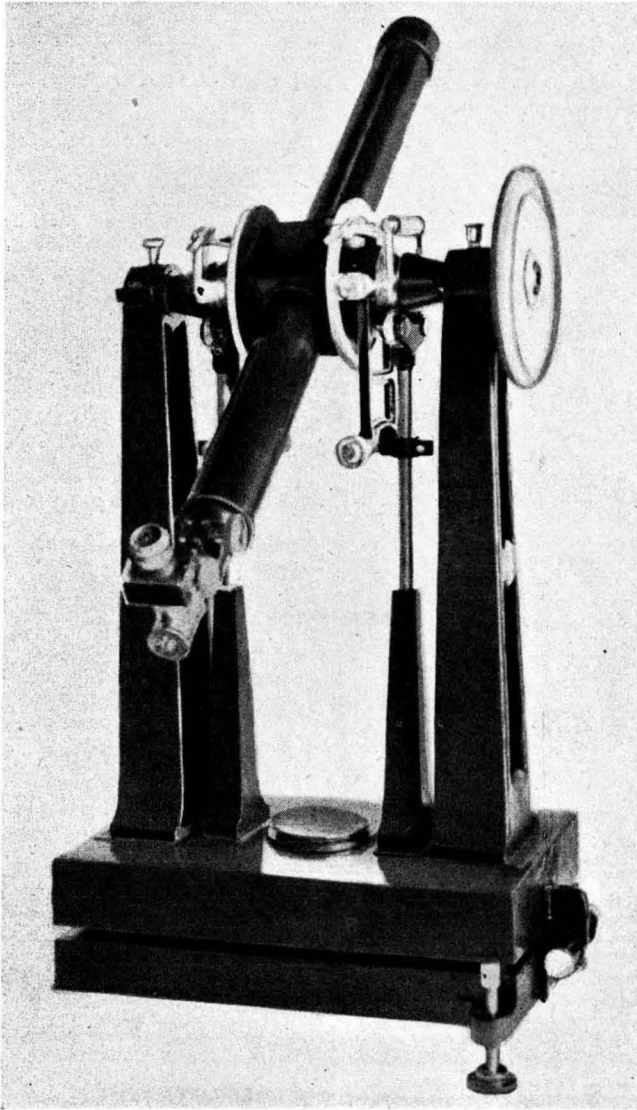
Transit Instrument

THE *Andhra Scientific Co. Ltd.* of Masulipatam has designed and fabricated a transit instrument which was exhibited at the January session of the *Indian Science Congress*. The optical system consists of a 3" or 4" apochromatic objective (triplet), precisely corrected for chromatic and spherical aberrations and coma, and a Ramsden type eyepiece. The aperture ratio is F/15. In the field of view of the eyepiece are placed 2 gratules, one of which is fixed while the other can be moved by means of an accurate micrometer screw. For convenience of observation, the micrometer and eyepiece are fitted into a star diagonal prism adopter.

Two circular scales with 2 verniers each are mounted on the horizontal axis of the instrument which is exactly perpendicular to the optical axis of the telescope. One of the circles is divided into $\frac{1}{2}$ deg. and can be read correct to 1', while the other is divided into $\frac{1}{4}$ deg. and can be read correct to 10". Magnifiers which are focussed by spiral motion are fixed to each vernier for taking readings conveniently. The graduations of the scales are on solid silver worked into the brass body drawn with a fine diamond point on a precision dividing engine. The verniers are of solid silver. Both the scales are given a dull finish and lacquered for protection after filling up the graduations with a black pigment. The scales are tested to a high standard of accuracy.

The micrometer consists of a slide carrying a graticule which moves relative to a fixed graticule with a central vertical line. The movable graticule has 5 vertical lines at 2 mm. intervals (lying in the centre of 2 similar lines 16 mm. apart) with 2 horizontal lines (2 mm. apart) running across them. The screw of the micrometer is of the precision type and made of low expansion steel.

The illumination for the graticule is provided by a right-



TRANSIT INSTRUMENT.

angled prism so fixed in the hollow axis of the instrument as to reflect a small pencil of light entering the axis from an outside source on to the graticule. The whole arrangement is so designed as to give only a diffuse oblique illumination. The prism can be adjusted from the outside by means of a milled knob.

The level tubes mounted on the instrument are $\frac{1}{4}$ " in dia. and are precision ground to give the exacting sensitivity required. These are thoroughly tested to

within very close limits prior to and after mounting.

The axis of the instrument, which is hollow and of good quality brass with a central tube to take the telescope, is provided with hardened steel-bearings moving in gun metal bushes carried on 2 elegant and sturdy cast iron supports. The weight of the axis is mostly taken up by 2 spring loaded anti-friction rollers.

The heavy cast-iron base is provided with coarse and fine

adjustments, and can be clamped in any position.

The whole instrument is finished in a pleasing dull-black enamel with some brightly nickelled parts as relief, presenting an elegant picture of Indian skill and craftsmanship.

A New Maximum & Minimum Hygrometer

A NEW TYPE OF HYGROMETER, THE "Mahajan Maximum and Minimum Hygrometer", has been developed by Dr. L. D. Mahajan, Director, Meteorological Observatory, Patiala. Just like the maximum and minimum thermometer, this instrument records the maximum and minimum humidities for any given period. The hygrometer is provided with 3 pointers indicating, in addition to maximum and minimum humidities, the percentage humidity of the surrounding atmosphere at the time of reading. It serves the purpose of a hygrograph also and saves much labour and expense. (Contributed)

Standard Atmosphere for Testing

FOR MAKING PROPER AND EQUI-TABLE comparisons of test data on various commodities, materials and equipment, an agreed set of atmospheric conditions under which tests may be carried out are necessary. The I.S.I. Sectional Committee on Textile Standards (TDC 1) prepared a note on the subject of *Standard Atmosphere for India* which has been published in the first number of the *Indian Standards Institution Bulletin*, a quarterly publication issued by the I.S.I. (1949, 1, 5).

No standard atmospheric conditions have been defined for any tropical country, at any rate, for one having atmospheric conditions of the type prevailing in India. This has resulted in:

(1) The non-existence of satisfactory installations of air-conditioned rooms for testing.

(2) Installations of a few testing rooms maintained at varying conditions *arbitrarily* fixed, and testing being carried out without any standard atmosphere.

(3) Confusion in reporting, comparing and evaluating test data.

The proposed standardization of standard atmosphere (SA) in India is based on the following principles:

(1) Temperature and relative humidity conditions prescribed

COUNTRY	CONDITIONS			
	TEMPERATURE		RELATIVE HUMIDITY	
	°F.	Tolerance °F.	%	Tolerance %
U.K.	70	2	65	1
U.S.A.	70	2	65	2
Australia	70	5	65	2
Other European countries	68	+9	65	2
India (proposed)	80	-3-6 3	80	2

must be suited to the majority of tests requiring SA such as tests on textiles, paints, plastics, paper, batteries, organic materials in general including biochemical and biological products, etc.

(2) The equipment required for maintaining SA must be free from complications, and should be economical in initial cost, operation and maintenance.

(3) The standard atmosphere must be comfortable for workers and operators engaged in testing.

(4) Direct and indirect relationships with the prevailing weather conditions during the different seasons of the year in different parts of the country on the one hand, and the three basic requirements mentioned above on the other, should receive due consideration in fixing the SA. For example, the closer the SA conditions are to the general weather conditions, (a) the closer will be the correspondence between laboratory test results and the actual service value of tested materials; (b) the more economical will be the equipment and installations required in respect of initial cost, operation and maintenance; and (c) the more comfortable and healthy will it be for workers who are constantly and alternately exposed to the SA and the outer atmosphere.

A comparison of the SA adopted in various countries with that proposed for India is made in the Table above.

Aerial Survey of Nepal

THE *Air Survey Company of India Ltd.*, a subsidiary of the *British Fairey Aviation Company* which has been mapping the Indian sub-continent for the last 25 years, has undertaken an aerial survey of Nepal. The object of the survey is to complete a topographical map of 29,000 sq. miles of catchment area of the 3 main rivers of the Gurkha country. The equipment of the expedition team includes air cameras, processing machinery and mapping

apparatus weighing more than half a ton.

14 aircraft are to be used at different times from the base at Dum Dum aerodrome, Calcutta. The survey is expected to complete aerial photography in 2 flying seasons (14 months) but the ground staff of cartographers are likely to take two and half years to finish their job. Even then only 20,000 sq. miles of Nepal (out of 54,000 sq. miles) would have been covered.

Aerial survey may cost anything from £10 a sq. mile for elementary work such as recording a surface without contouring to £100 a sq. mile for complex and difficult work requiring the aid of radar and other expensive ground installations.

Water Requirements of Crops

THE GOVERNMENT OF INDIA, on the recommendation of the *Central Board of Irrigation*, have decided to appoint a Standing Advisory Committee for research on water requirements of crops consisting of representatives of the *Central Board of Irrigation*, Ministry of Agriculture and the Meteorological Department.

The Committee will advise on research already being conducted in regard to the most advantageous use of water for agricultural purposes and problems incidental thereto. It will study and co-ordinate research work which is being done by the *Indian Council of Agricultural Research*, Agriculture and Irrigation Departments in Provinces and States and agencies concerned with multi-purpose projects, and advise on the most satisfactory method of achieving optimum results in this field.

Railway Research Council

THE GOVERNMENT OF INDIA HAVE set up an Advisory Research Council for Indian Railways "to improve efficiency, effect economy

and co-ordinate research on subjects connected with the railways". Indian railways are the largest consumers of coal and of ferrous and non-ferrous metals, and spend over Rs. 30 crores annually for the purchase of these requirements. The Council will function as a "screening body" and pass on such technical problems as cannot be tackled by railway administrations to research institutions within the country.

The Council consists of the following members: Mr. K. C. Bakhle, Chief Commissioner of Railways; Dr. S. S. Bhatnagar; Prof. K. S. Krishnan; Dr. D. R. Malhotra; and Mr. P. L. Verma.

Tea Committee for India

THE GOVERNMENT OF INDIA HAVE decided to form a *Tea Committee* which will replace the existing *Indian Tea Market Expansion Board* founded in 1903. The *Committee* will consist of representatives of the tea industry, the Central Government and various tea-growing Provinces and States, and will work under the control of the Central Government. The functions of the *Committee* will be mainly to promote research, collect statistics, improve marketing conditions, and advise tea growers and exporters on all matters relating to tea.

Jubilee Number of "Capital"

THIS BEAUTIFULLY GOT-UP souvenir number, issued by India's well-known journal of economics to mark the completion of its 60 years of publication activity, is a valuable record of permanent value, of developments in the fields of commerce, trade, industry and finance in India. Short biographies of personalities associated with the journal, India's credit structure, joint stock enterprises, insurance and other economic problems, a survey of plantation crops like tea and jute, and of paint, varnishes and salt industries form the subjects discussed in its 150 pages. The last item, "A Chronicle of 60 Years", gives a résumé of important political and economic events in India and elsewhere.

Reward for Groundnut Hand Decorticator Announced

A PRIZE OF RS. 2,000 WILL BE awarded by the *Indian Oilseeds Committee* to any person or body

who designs the best model of groundnut hand decorticator and demonstrates its working to the satisfaction of the *Indian Oilseeds Committee*. Entries accompanied with diagrams showing details of the machine should be submitted to the Secretary, *Indian Oilseeds Committee*, Ministry of Agriculture, New Delhi, not later than October 31, 1949. Competitors should be prepared to demonstrate the working of the machine at such place and time as directed by the *Committee*.

The groundnut hand decorticator should fulfil the following conditions: (i) the hand decorticator should, as far as possible, be made of material easily available in villages. It should preferably be made of wood and should have as few iron parts as possible; (ii) the construction should be simple so that repairs and spare parts can be provided in the villages by the village carpenter or blacksmith; (iii) the grates or sieves used in the machine should be adjustable to enable proper decortication of groundnut pods of different varieties and sizes; (iv) the turn-over and cost of decortication should compare favourably with those of power decorticators; (v) the proportion of split and broken kernels, "nooks" and unshelled pods in the decorticated produce should be significantly small; and (vi) the cost of the hand decorticator should not exceed Rs. 100.

Announcements

UNESCO Grant for Indian Chemical Society — The *Union Internationale d' Histoire des Sciences* has, out of their subscription from UNESCO, contributed a sum of \$500 towards the preparation of "History of Chemistry in Ancient and Medieval India".

Central Food Technological Research Institute, Mysore — A Local Planning Committee consisting of the following members has been set up to expedite the work of organizing the Institute: Hon'ble Mr. K. C. Reddy, Prime Minister, Mysore (*Chairman*); Prof. Mata Prasad (Royal Institute of Science, Bombay); Dr. G. Shankaran (All-India Institute of Hygiene and Public Health); the Director, Indian Institute of Science, Bangalore; the Director, Council of Scientific & Industrial Research or his nominee (*Ex-officio*); a representative each of the Food Ministry and Health Ministry of the Government of India; and

Planning Officer, Central Food Technological Research Institute (*Secretary*).

Central Drug Research Institute, Lucknow — The appointment of a Local Planning Committee consisting of the following members has been announced: Hon'ble Shri C. B. Gupta, Minister, U.P. Government (*Chairman*); Acharya Narendra Deo; Dr. B. Sahni; Mr. A. N. Jha; the Director of Medical and Health Services, U.P.; the Principal, Medical College, Lucknow; Chief Engineer, P.W.D., Lucknow; the Executive Officer, Improvement Trust, Lucknow; a representative of the pharmaceutical industry; a representative of the Central Legislature; and Planning Officer, Central Drug Research Institute (*Secretary*).

Second International Conference on Soil Mechanics & Foundation Engineering — Sets of the printed proceedings (5 vols.) of the Conference held in Holland during June 1948 are available for sale. Those interested may secure copies directly from the Secretary, Organizing Committee, Stichting Waterbouwkundig Laboratorium, Delft (Holland), or may contact the Secretary, Central Board of Irrigation, Simla. The cost per set is Rs. 100.

"British Chemicals & Their Manufacturers" — The 1949 edition of this Directory published by the *Association of British Chemical Manufacturers* includes up-to-date information on the manufacturers of chemicals in Britain and supercedes the 1946 edition.

The Directory can be had free by any inquirer writing on business paper or giving a genuine indication of his being likely to put the publication to good use as a purchaser of chemicals.

REPORTS FROM STATES & PROVINCES

(continued)

On the animal husbandry side, 4 regions have been proposed: (1) *Dry Belt Region* consisting of Eastern Punjab, Delhi, Rajasthan and West U.P.; (2) *Wet Region* including Bengal, Assam, Bihar, Orissa, Madras, Travancore and Cochin; (3) *The Medium Rainfall Region* consisting of Hyderabad, Central India States and C.P. and Berar; and (4) the *Himalayan Region*.

BOMBAY

Ship-building Yard

BOMBAY IS TO HAVE A SHIP-building yard bigger and better equipped than the Vizagapatam ship-building yard, if the proposed 5-year plan submitted by the Provincial Government to the Government of India's Ship-building Committee is adopted. The scheme estimated to cost Rs. 2.5 crores has been drawn up by the Chief Engineer of the Bombay Port Trust.

The proposed yard is to be located at Trombay, about 15 miles north-east of Bombay, and 10 ship-ways are planned so that the construction of 10 vessels up to 15,000 tons capacity could proceed at the same time. Room for further expansion and fitting of 2 berths is provided for in the plan.

The Trombay site is eminently suitable owing to its ample sea frontage for launching vessels and a vast area of land in the rear. All labour required can be had from the adjacent maritime districts.

Manufacture of Penicillin

THE GOVERNMENT OF INDIA HAVE decided to set up a plant to manufacture penicillin in Bombay. The work is to commence in a couple of months. The plant, which is expected to be complete in 2 years, will cost about 2 crores of rupees. It is proposed to manufacture 100,000 million units of penicillin in the initial stages and to gradually increase the output 4 times. Production of large quantities of anti-malarial and sulpha drugs are also included in the programme.

MADRAS

Livestock Improvement

THE GOVERNMENT HAVE OPENED 2 new veterinary institutions at Arkonam and Pithapuram bringing the total of such institutions in the province to 138.

A scheme for the prevention of contagious diseases spreading from adjacent provinces is under consideration. Sanction has been accorded for running a breeding-cum-dairy farm at Vizagapatam. A beginning has been made for breeding the fine wool sheep of the Nilgiris, and necessary lands have been allotted on the Wenglock downs on the Nilgiris. A combined sheep and cattle farm is being established at Kurikuppi in the Bellary district.

Reports from States & Provinces

MYSORE

Gold Prospecting

MYSORE'S BIGGEST IRRIGATION project — the Lakkavalli reservoir on river Bhadra — will submerge extensive tracts of lands with gold-bearing quartz veins. It is, therefore, proposed to recover all the gold before this 10-year irrigation project is put through. Exploratory work at Bellara has disclosed that the extent of the reef is nearly twice the original estimate. A 10-year scheme was approved in 1947 to develop these areas. To meet the increased power requirements for the development programme, the Government have recently sanctioned a scheme at an estimated cost of Rs. 2.46 lakhs to construct a dam across the Suvanamukhi river. Meanwhile steps are being taken to equip the Bellara mines with modern milling and cyanidation plants.

NEW DELHI

Reorganization of Agricultural Research

THE ADVISORY BOARD OF THE *Indian Council of Agricultural Research* at its annual meeting held in New Delhi during January approved a number of research schemes including those on improvement of crops, agronomical conditions in general, soil conservation in Himachal Pradesh, setting up a suitable organization for collection of agricultural statistics, research on tuber crops as a substitute for cereals, and a standard method for milk analysis and dairy development. Particular emphasis was laid on research schemes in East Punjab and West Bengal for agricultural rehabilitation.

In the absence of reliable data on crop yields, the results of Government development plans on the Grow More Food and other schemes cannot be gauged properly, and the Board felt that an organization was required to collect accurate statistics. Accordingly, the Board has recom-

mended that the random sampling method should be adopted in the 5-year co-ordinated scheme for conducting surveys on the principal crops of India. The main objects of the scheme are: to evolve a random sampling technique for crop-cutting experiments under the auspices of the Provincial Agricultural Departments, to demonstrate the feasibility of the method devised, to train the provincial staff in the technique, to estimate the yield per acre for the province as a whole and to revise the present state of district normal yields.

The total expenditure involved in the scheme is estimated to be Rs. 59 lakhs.

A soil conservation scheme of Himachal Pradesh including ecological survey, cultivation of soil binders and collection of erosion data was recommended. A scheme for the improvement of soil conditions in the spent-up orchard land in the Kumaon Hills was sanctioned. A scheme to check soil erosion and the spreading of the desert area in Jaipur was approved.

The Council had at its previous meetings recommended the cultivation of tubers such as sweet potatoes and casava, and had invited Provincial Governments to submit schemes on the subject. The schemes submitted by Bombay, United Provinces, East Punjab, Bihar, Orissa and Mysore were scrutinized and approved.

Schemes for the improvement of rice crops in East Punjab, Coorg and West Bengal, and of maize in West Bengal and Bihar were sanctioned. Several research schemes on the improvement of pulses in Madras, West Bengal, U.P., Bihar, Hyderabad, Orissa, Mysore and Gwalior as also research schemes on the improvement of millets in Mysore and palmyra in Madras were approved.

A scheme for the co-ordination of research on insect pests and on the blast and foot diseases of rice in Madras, and on wilt disease of gram in E. Punjab were sanctioned. The Council recommended that the animal nutri-

tion scheme for Madras which was stopped in 1943 should be revived.

A scheme to explore the possibilities of using milch cows for draught work was considered and approved. It was suggested that the work should be conducted at 4 centres including the Indian Dairy Research Institute, Bangalore. The standardization of methods of analysis of milk and milk products, suggested in a scheme of the Indian Dairy Research Institute, was sanctioned.

Other new schemes considered and approved by the Board were: cattle-breeding in Rajputana, research on insecticides in East Punjab and cultivation of medicinal plants in Himachal Pradesh.

Regionalization of Agricultural Research

AT A MEETING OF THE REGIONALIZATION Committee of the *Indian Council of Agricultural Research* held in January, research on the basis of different soil and climate categories was decided upon with a view to ensure planned agricultural development in all parts of the country. It was tentatively decided to divide the country for research purposes into different regions and to set up Regional Committees.

The following 5 regions were suggested both from the point of view of research on principal crops as well as on the requirements of animal husbandry:

(1) *Dry Northern Region* comprising of East Punjab, Western U.P. and Berar and parts of Rajasthan, where wheat is the chief crop.

(2) *Eastern Region* comprising of Assam, Bengal, Bihar, Orissa, East C.P., East U.P. and East Madras, where rice is the chief crop.

(3) *Southern Region* — Jhansi in south United Provinces, C.P. and Berar, Central India States, Western Hyderabad, Deccan, Western Madras, Eastern Bombay, Baroda and part of Mysore, where millets are the important crops.

(4) *Coastal Region* comprising of parts of Bombay, Madras, west of Western Ghats, Mysore, Coorg, Travancore and Cochin.

(5) *Temperate Himalayan Region* including Assam, Sikkim, Nepal, Bhutan, Kumaon, Gharwal, Simla hills, Kulu, Chamba and Kashmir State.

(continued on page 160)

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 February 1949.

Inorganic Chemicals

31457. BOAKE, ROBERTS & Co. LTD.: Production of aluminium salts of organic acids: *Reacting an alkali, an aluminium salt, an organic acid, and a hydrophylic colloid.*
37640. NATIONAL SMELTING Co. LTD.: Removal of sulphur compounds from gases: *Contacting the gases with alkali metal or alkaline earth metal oxides or hydroxides on a large surface porous support between 100° to 850°C.*
40069. GLAXO LAB. LTD.: Preparation of salts of penicillin: *Penicillin dissolved in a solvent is treated with a salt of primary organic base and an organic acid.*
40070. GLAXO LAB. LTD.: Preparation of the ammonium salt of penicillin: *Ammonium salt of penicillin is treated with an ammonium salt of organic acid.*
39432. NATIONAL RADIO & ENGINEERING Co. LTD.: Electrical moulding composition: *Consisting of shellac, resin, alkaline earth salt of resin and a filler.*
38944. SEAILLES: Method for making a water solution of raw aluminates of lime and its application to the manufacture of alumina: *Dissolving lime aluminate in aqueous solvent containing alkali in dilute condition.*
39249. SEAILLES: Method for making a water solution of raw aluminates of lime and its application to the manufacture of alumina: *Adjusting the proportions of calcium aluminate and alkaline lyes so that a mixture of alkali and calcium aluminate is formed.*
39687. UNITED STATES RUBBER Co.: Recovery of fluosulfonic acid (Addition to No. 39686): *Admixing fluosulfonic acid containing free sulphur trioxide with spent acid, distilling the resulting mixture.*
40259. WILLIAMS: Process for converting sulphur dioxide to hydrogen sulphide: *Passing a mixture of sulphur dioxide, oxygen and steam over incandescent carbon.*

Organic Chemistry

38574. DISTILLATION PRODUCTS INC.: Process of separating delta-tocopherol from vegetable oils: *Treating vegetable oil to obtain mixed tocopherol concentrate and then separating delta-tocopherol by fractional crystallization or chromatography.*
30764. ELLIOTT & AUSTRALIAN DRUG PTY.: Extraction and recovery of alkaloids: *Extracting with acid or neutral aqueous or aqueous alcoholic solution, treating the solution with water immiscible phenol and recovering alkaloid from the phenol phase.*

Analytical Chemistry

38784. AMERICAN CYANAMID Co.: Purification of sugar solutions: *Treating sugar with acid and alkaline contact reagents and regenerating it with hot regenerating agent.*

Medical Research & Practice (INCLUDING CLINICAL APPLICATION OF DRUGS & PHARMACEUTICALS)

38761. ELI LILLY & Co.: Crystallization of penicillin: *Precipitating penicillin salt with acetone containing up to 4 per cent water, dissolving the precipitate in acetone containing up to 10 per cent water and reducing the amount of water.*

Metals & Metal Products

40497. BRITISH NON-FERROUS METAL RES. ASSOC.: Production of metal coatings upon metals: *Dipping metal article into liquid lead-containing nickel, cobalt manganese or chromium.*
39938. TISSOT-DAGNETTE: Aluminium solder and a method for the production thereof: *Comprising of an aluminium-containing metal and a silver-containing metal.*

Paper & Allied Products

40456. COLUMBIA RIBBON & CARBON MANF. Co. LTD.: Carbon papers: *Backing the carbon paper with an oil repellent coating.*

Photographic & Optical Goods

39445. MUDALIAR: Manufacture of watch glasses: *Disc cut from scrap glass pieces or electric bulbs placed in metal dies, heated in furnace and trimming edges on grind stone.*

Rubber & Rubber Products

40722. WINGFOOT CORP.: Frothed latex sponge rubber: *Containing frothed rubber latex sponge particles of at least 32 mesh.*

Textile & Textile Products

38010. I.C.I. LTD.: Manufacture and application of new textile treatment agents: *Mixing polycarbonate with formaldehyde.*
36795. SYREENI: Method of producing colour mixtures: *Mixing dyestuffs or colouring materials according to a classification.*
37868. INDIAN JUTE MILLS ASSOC. RES. INST.: Preparation of a fast-to-light substantially white jute: *Subjecting bleached jute to an acetylation treatment.*
38260. I.C.I. LTD.: Improvements in colouring processes: *Dyeing in the presence of benzyl alcohol.*

Miscellaneous

33995. CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE: Production of nuclear energy: *Comprises a mass of uranium or thorium having helium or deuterium distributed therein to change the velocity of neutrons below the range of resonance velocities.*
34000. CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE: Apparatus for the production of energy by nuclear fission: *During bombardment of uranium with neutrons the unlimited reaction chain is allowed to develop and checked periodically.*

Journal of Scientific & Industrial Research

Vol. VIII B

APRIL 1949

No. 4

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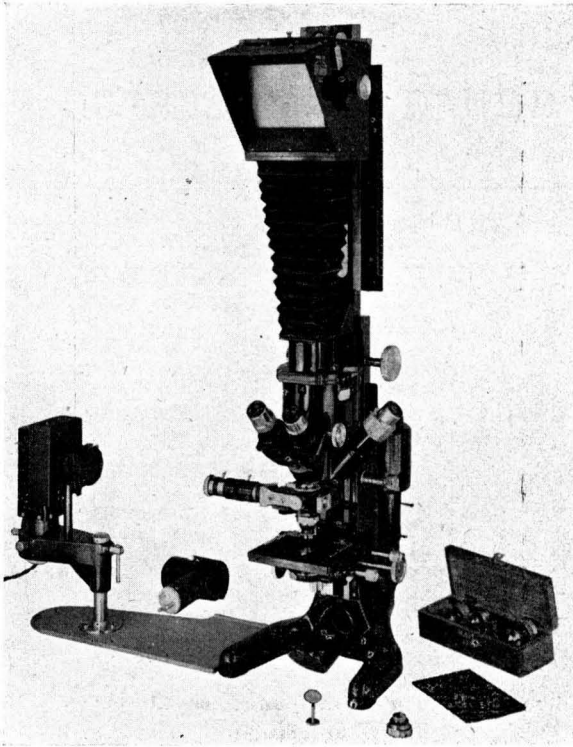
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NEW DELHI

A Study of Coarse Filtering Media for Portable Producer-Gas Plant Filters*

S. K. DAS GUPTA & MOHAN LAL KHANNA

Physical Laboratories, Council of Scientific & Industrial Research, Delhi

TOWARDS the close of 1941 a search for the alternative motor fuels was made in this country due to petrol shortage. One of the means to overcome such a difficulty was the development of producer-gas plants suitable for use on transport vehicles. At the time of conversion of the existing types of transport vehicles to producer gas, the Indian manufacturers had nothing to guide them, because a scientific study on the subject had not been undertaken earlier. The manufacturers of the producer-gas plants showed a great ingenuity in the design of the filters by using the various available fibrous materials for gas filtration. During the past seven years, however, an increasing amount of attention has been paid to the scientific development of transport using producer gas as a source of power, together with the design, manufacture and testing of mobile producer-gas plants.

Abrasive dust, tarry matter and corrosive gases are present in the gas. The life of an engine running on producer gas mainly depends on the extent to which these impurities have been got rid of from the gas as finally supplied to the engine. The satisfactory performance, on the other hand, demands free and ample flow of clean gas with the least pressure drop in the delivery system. This pressure drop mainly takes place in the filtration system. To meet the situation, two different types of filters, namely wet and dry, have been developed and employed in various countries.

In India the general practice followed for gas filtration is to pass it through a

series of dry filters, generally three in number. Fibrous materials and textile fabrics are made use of in these filters. After a careful study of the available textile fabrics in this country, the material most suited for use in the final stage of gas filtration¹ has been previously recommended and a suitable filter design has been suggested. It was, therefore, felt desirable to investigate the various filtering media available for use in the first two stages of gas filtration. A knowledge of the flow characteristics of gas through these media with respect to the pressure drop, depth of packing, packing density and the influence of these factors on the size of particles passing through the filter bed are absolutely necessary for the development and design of these filters. Investigations relating to these factors are described in the present paper.

In the filters of a portable producer-gas plant the materials most frequently used as coarse filtering media are cotton waste, jute, sisal, coir and *munj*. While selecting these materials, the various factors to be considered are :

- (i) cheapness ;
- (ii) easy availability ;
- (iii) number of times it is fit for re-use in the filter after cleaning or dusting ; and
- (iv) compressibility.

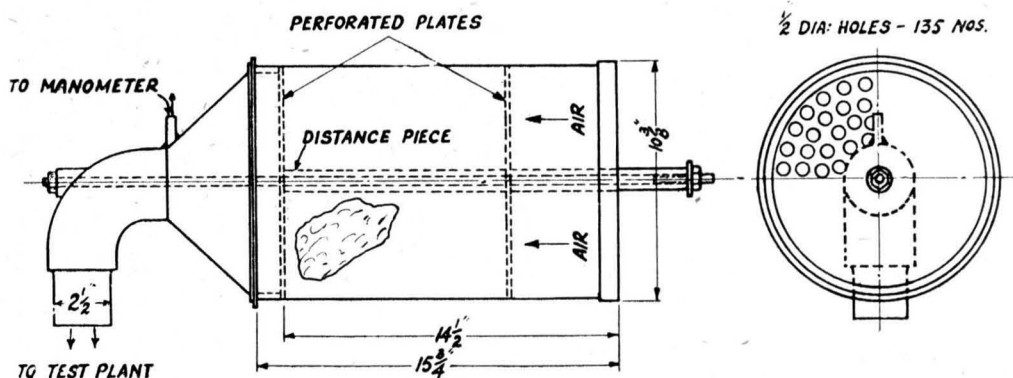
The selected materials were procured from the market. Before undertaking any test, these materials were carefully cleaned by hand-picking of any foreign materials present and dried in the sun. General description, price, place of origin and the

*Paper read before the Engineering and Metallurgical Section of the 36th Indian Science Congress Session, Allahabad.

TABLE I

No.	MATERIAL	ORIGIN	COLOUR	PRICE PER LB., ANNAS	AVERAGE FIBRE DIA., IN MICRONS	CHARACTERISTICS OF THE MATERIAL
1.	Cotton waste	Mill waste	White	3	428*	Soft, easy to pack, sufficient control on quantity and sizes of particles passing, very difficult to clean
2.	Jute	Bengal	Light yellow	4	73	Soft, easy to pack, considerable control on quantity and sizes of particles passing, easy to clean
3.	Sisal	Bihar and Orissa	White	...	158	Hard, easy to pack, less control on quantity and sizes of particles passing, easy to clean
4.	Coir	South India	Brown	5	360	Hard, brittle and difficult to pack, much less control on quantity and sizes of particles passing, very easy to clean
5.	Mumj	Punjab	Light yellow	4	228	Very hard, brittle, very difficult to pack, little control on quantity and sizes of particles passing, very easy to clean

* The diameter of yarn is given in this case.



LINEAR FILTER

FIG. 1

behaviour of these materials have been summarized in Table I.

As indicated in Table I, all these materials are available in this country and the cost per lb. of the material is not more than five annas in each case. The common practice followed in cleaning them is by dusting or beating the material with a stick. Some of these materials are easy to clean while others are not. This negative property of the material leads to frequent renewals and, therefore, increases the cost of maintenance. Some of them can be compressed more easily than others by keeping the packing space constant. They are generally soft, can be easily handled, give uniform packing, require less filtering area and have sufficient control on the quantity and sizes of dust particles passing through the pores created by the packing material. The main requirements

of a good filtering material generally are that it should allow the least quantity of dust particles to pass through the filter bed and, at the same time, allow a sufficient quantity of clean gas to pass through a given filtering area with the least pressure drop.

The apparatus employed during this investigation was the producer-gas bench-testing unit² installed in these laboratories. A 29.4 h.p. Chevrolet engine was made to act as a pump of the positive displacement type and was coupled through a Chevrolet standard gear box to a 25 h.p. 3-phase induction motor. The material under investigation was packed in a filter, which was connected at the end of the intake pipe of the testing unit. Air was drawn through the filter at various rates of flow and was discharged to the atmosphere through a

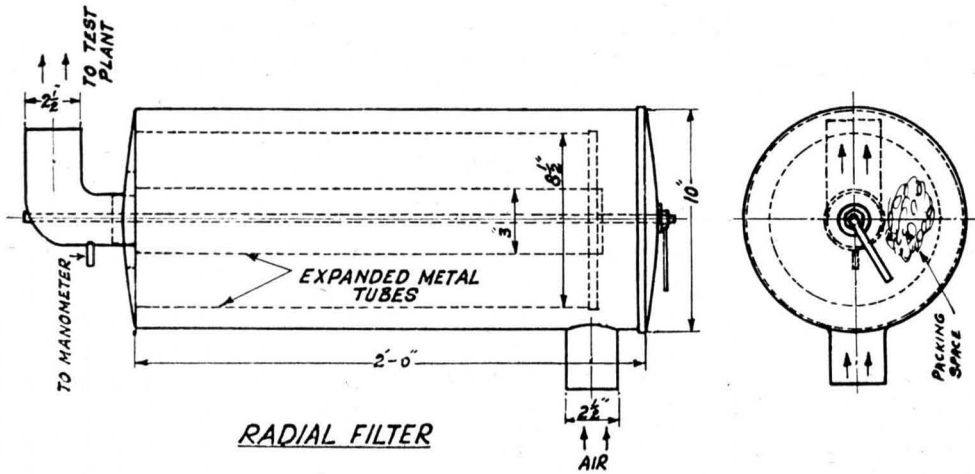


FIG. 2

calibrated orifice meter via an anti-pulsating tank. The atmospheric pressure and temperature prevailing at the time of test were recorded. The flow was corrected to 15°C. and 762 mm. of Hg in every case.

Two different types of filters, namely linear and radial, were used for these tests. They are named according to the flow of gas inside these filters. A linear filter is diagrammatically illustrated in Fig. 1. It consists of a cylindrical shell of 10 3/8" diameter by 14 1/2" long with two M.S. perforated circular plates. The plate at the gas outlet end was fixed in position, whereas the position of the plate at the gas inlet end could be adjusted according to the depth of packing required. The pressure difference across the filter bed was measured with the help

of a water manometer. An arrangement for connecting it across the filter was provided.

Each of these materials was packed and tested for pressure drop at maximum packing density, which could be attained by hand packing. The depths of packing chosen for experimental purposes were 2", 5", 8" and 11". The pressure drop across the filter bed for the various rates of air flow at different depths of packing at maximum packing density was determined. These values were also checked for decreasing rates of flow of air. These results in the case of cotton

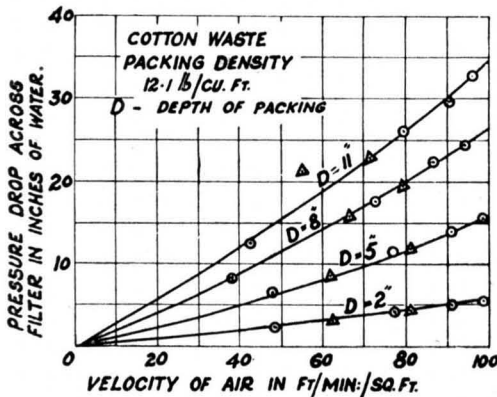


FIG. 3 — PRESSURE DROP vs. VELOCITY OF AIR FOR COTTON WASTE.

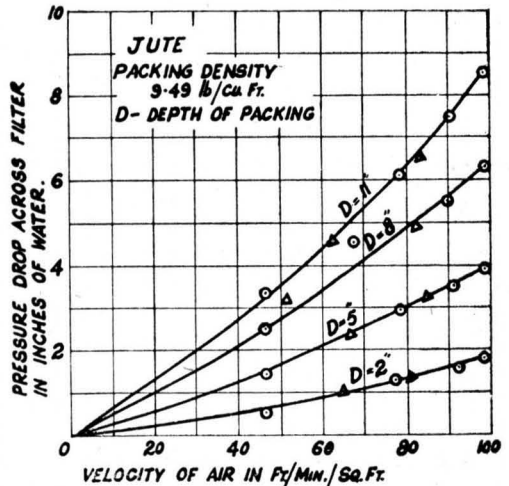


FIG. 4 — PRESSURE DROP vs. VELOCITY OF AIR FOR JUTE.

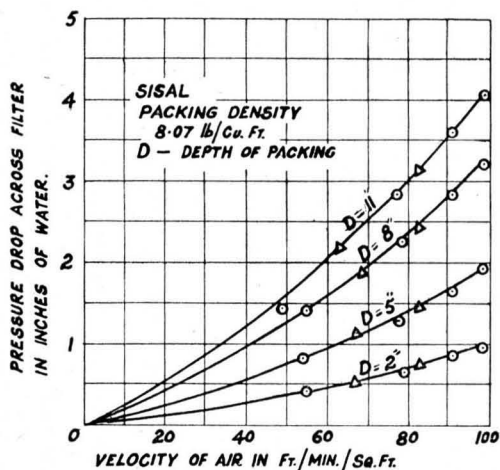


FIG. 5 — PRESSURE DROP VS. VELOCITY OF AIR FOR SISAL

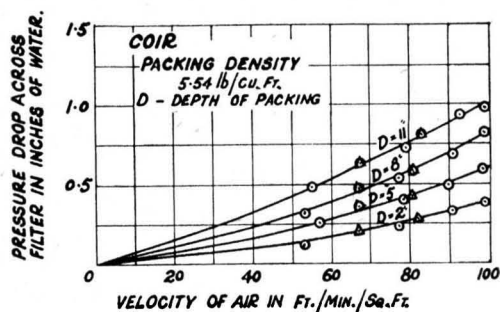


FIG. 6 — PRESSURE DROP VS. VELOCITY OF AIR FOR COIR.

waste, jute, sisal, coir and *munj* have been graphically represented in Figs. 3, 4, 5, 6 and 7 respectively. The pressure drop for the empty filter for the various rates of air velocity was also determined. In Fig. 8 are given the curves showing the relationship between pressure drop and depth of packing at only maximum packing density for the five materials studied presently.

At the end of every set of pressure drop test, the filter unit was connected at the end of the conical filter unit³. It consisted of two conical funnels of about 21" diameter with provision for clamping a 20" filter paper between them. In the filter unit a standard filter paper³ was clamped so as to arrest the dust particles passing through the filter paper. Air at the rate of 50 cubic feet per minute⁴, which represents the full rate of gas flow for a heavy-duty mobile producer-gas plant, was sucked through. Charcoal

powder having particle size in the range of 1-1,000 microns was dusted at the inlet end of the filter unit for a period of ten minutes. The dust, which passed through the filter bed, was collected on the filter paper and examined under a microscope for pore size determination. In every case the predominant size of charcoal dust particles was determined. The total number of observations by various workers were 24 in each case. The mean of these values together with the probable error are summarized in Table II. These results have been obtained while using the linear filter.

A radial filter is shown diagrammatically in Fig. 2. It consists of a cylindrical shell of 10" diameter by 24" long. Inside it, there are two co-axial expanded metal cages 21½" long and 8½" and 3" outside and inside diameters respectively. The space between the two tubes was filled with the material under test. The packed unit was put inside the cylindrical shell. While packing the

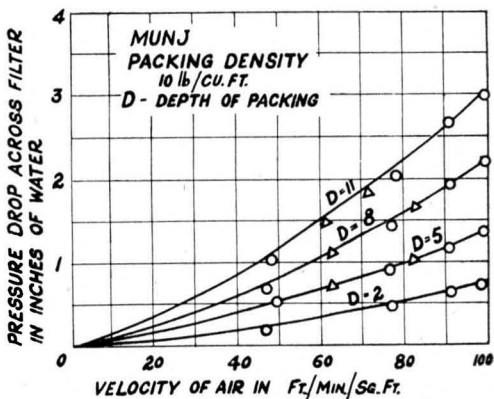


FIG. 7 — PRESSURE DROP VS. VELOCITY OF AIR FOR *munj*.

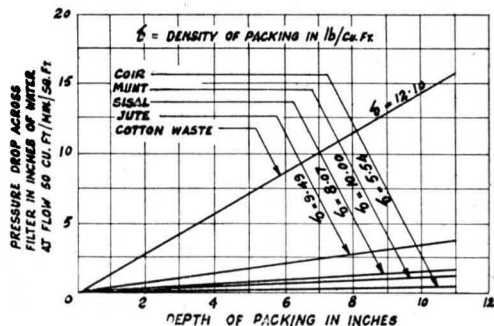


FIG. 8 — PRESSURE DROP VS. DEPTH OF PACKING FOR THE FIVE MATERIALS IN A LINEAR FILTER.

TABLE II

MATERIAL.	FIBRE DIA., IN MICRONS	DENSITY OF PACKING, IN LB. PER CU. FT.	DEPTH OF PACKING, IN INCHES	SIZE OF PARTICLES COLLECTED ON FILTER PAPER, IN MICRONS
Cotton waste	428†	12.10	2	37.2 ± 2.2
			5	21.0 ± 1.9
			8	16.6 ± 1.9
			11	9.7 ± 1.9
Jute	73	9.49	2	*120 ± 7.2 (?)
			5	19 ± 1.9
			8	13 ± 1.9
			11	6.3 ± 1.9
Sisal	158	8.07	2	216 ± 7.2
			5	215 ± 7.7
			8	210 ± 8.2
			11	*67.4 ± 7.2
Coir	360	5.54	2	281 ± 13.9 (?)
			5	249 ± 7.2
			8	242 ± 11.1
			11	212 ± 7.2
Munj	228	10.00	2	465 ± 19.8
			5	445 ± 31.6
			8	459 ± 7.2
			11	339 ± 7.2

† In this case, dia. of yarn is given.
 * Irregular values.

material in the cages and fitting them inside the filter, every possible care was taken to avoid any short circuit paths for gas. In order to measure the pressure drop, a water manometer was connected across the filter bed. The results of pressure drop at various rates of air velocity at maximum packing density for the five materials have been graphically represented in Fig. 9. The pressure drop for the empty filter was also recorded.

Discussion

A study of the pressure drop curves for the various materials show that cotton waste has exhibited the highest and *munj* the least resistance to the flow of gas. A glance at Table II reveals that the size of dust particles passing through the filter bed decreases with an increase in the depth of packing. The compressibility and the fibre diameter of the material, in general, play an important part in the dust-collecting efficiency.

From the characteristics of the materials and the size of dust particles passing through the filter bed (see TABLES I and II), the various materials investigated may be divided into three groups:

Group I — Cotton waste and jute.

Group II — Sisal and coir.

Group III — *Munj*.

Group I — Of all the materials investigated, cotton waste and jute have been found to

be the best. Cotton waste offers a high resistance to the gas flow. It cannot be easily cleaned and, therefore, the chances of its being used repeatedly for gas filtration are limited. But, in spite of these shortcomings, the dust-retention capacity of cotton waste is very prominent. During the various gas purity tests conducted in these laboratories, it has been most frequently noticed that the colour of the standard filter paper used to arrest the particles of dust present in the gas as finally supplied to the engine, does not appreciably change throughout the dust test when using cotton waste in one of the filters of the gas producer. From the summary of the results given in Table II one is led to think that jute is as good a filtering medium as cotton waste. As already stated, the criterion of a good filtering medium is that it should allow the least quantity of the smallest dust particles through the filter bed. Considering the gas filtration qualities of the two materials, cotton waste is found to be superior to jute and is most suitable for use in the second stage of producer-gas filtration. Though the size of dust particles passing through the jute filter bed is almost the same, yet it allows to pass slightly more quantity of dust and increases the work of the final filter. This could, however, be compensated by increasing the depth of packing of the filter bed. Jute is recommended in place of cotton waste for use in the second stage of gas

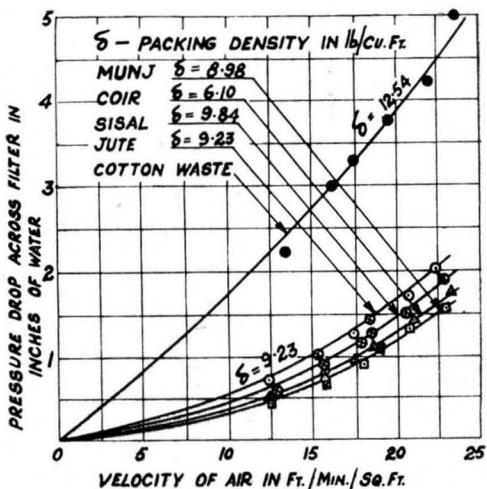


FIG. 9 — PRESSURE DROP VS. VELOCITY OF AIR FOR THE FIVE MATERIALS IN A RADIAL FILTER.

filtration, provided the depth of filter bed is increased.

Group II — Taking into consideration the size of particles passing through the filter bed, depth of packing, the quality of gas filtration, sisal has been found to be better than coir. Coir fibres are generally brittle and, therefore, the facilities of its repeated use for gas filtration are limited. There is, however, not much to choose between the two materials so far as the pressure drop through them is concerned. Of the two, sisal is to be preferred for efficient gas filtration, provided it is available in large quantities at a reasonable price. Coir and sisal are only suitable for use in the first stage of gas filtration.

Group III — Though *munj* has almost the same packing density as jute, yet the size of particles passing through its filter bed is the highest among the various filtering media studied in this investigation. Particles as big as 364 microns have been found to pass through the filter bed, even though the depth of packing and the packing density are maximum. This clearly points to its poor quality. The material is very difficult to pack and to spin. Such materials, therefore, are unsuitable for any stage of gas filtration.

By packing these five materials at maximum packing density in a radial filter, an idea of the resistance offered to gas flow has been obtained by the study of pressure drop

at various rates of air velocity. The results obtained in the two cases are quite consistent with each other.

In conclusion it may be stated that the data collected so far helps one in choosing the materials most suited for the first and second stages of gas filtration and in designing efficient radial filters so commonly used in India.

A mathematical study of the gas flow through these materials is in course of investigation.

The authors take this opportunity of thanking Dr. L. C. Verman, Director, Indian Standards Institution, New Delhi and Mr. K. A. Nair for the kind help and encouragement received from them during the course of the investigation.

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Effect of the Water-soluble Matter in Jute on the Microbiological Deterioration of the Fibre

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LITTLE is known of the precise nature of the water-soluble matter in natural vegetable fibres. In cotton, the presence of 0.5 per cent malic acid and 0.07 per cent citric acid has been reported¹. Some of the nitrogenous compounds present are probably water soluble. According to Marsh and Wood² raw cotton contains 0.2 per cent nitrogen. Jute has a nitrogen content of 0.2-0.3 per cent, nearly half of which is extractable by boiling water³. While these components and also soluble carbon sources may help fungal growth, they are present in the fibre in amounts probably too low to have any appreciable effect. On the other hand, substances of vitamin nature are required by living organisms only in comparatively minute quantities and may cause a striking growth response. The presence of such water-soluble growth factors or nutrilites in natural vegetable fibres is only to be expected and have actually been demonstrated, directly or indirectly, in some instances. Thus Robbins and Ma⁴ showed that *Ceratostomella* species having deficiencies of thiamin, biotin and pyridoxine would grow normally when an extract of cotton batting was added to the medium. Robbins and Schmitt⁵ found evidence for the presence, in cotton, of an unknown factor that would stimulate the germination of *Phycomyces* spores. Sherwood and Singer⁶ demonstrated the presence of folic acid in cotton bolls and even in absorbent and non-absorbent cotton linters. Constantin⁷ reported in carded cotton a water-soluble substance promoting the growth of yeast which did not appear to be any of the known members of the vitamin B complex. Buston and Basu⁸ have established the presence of nicotinic acid, thiamin, riboflavin, pyridoxine, pantothenic acid and biotin in the water extract of jute; quantitative assay showed that the raw fibre contained 0.116-0.471 μg . of these vitamins per gm. except for biotin; this substance, active in much smaller amounts, was present in a lower concentration than the others.

Many known vitamins — and probably some yet unidentified — play a part in the metabolism of fungi as in many other living organisms, but while some fungi can synthesize their own requirements of all of these compounds, others cannot. These latter would not grow well on synthetic media but would do so on natural materials due to the presence there of these growth factors. That vegetable fibres, and fabrics made therefrom, should support good growth of such deficient organisms is, therefore, not surprising. This phenomenon is not of much direct economic importance in the textile industry in the case of many so-called superficial fungi but its industrial significance cannot be overlooked when active cellulose decomposing species are found to suffer from vitamin deficiencies.

Cases of such fungi responding to the stimulation of micro-nutrients have been reported in recent years. Zuck and Diehl⁹ have found that certain slow-growing species, not commonly associated with cellulose decomposition, nevertheless cause loss in strength of cotton duck; these were able to grow and sporulate well on cotton extract agar but not on synthetic sugar-based media. Zuck¹⁰ also found cotton extract agar a particularly good medium for growing the closely related genera *Memnoniella* and *Stachybotrys*; these organisms, very well known as cellulose decomposers and believed to cause extensive damage to cotton stores, have later been found to require pre-formed biotin for growth^{8,11}. It was shown by Buston and Basu⁸ that jute extract had the same effect on *Memnoniella* as the corresponding amount of pure biotin. The growth and perithecial fruiting of another well-known cotton-destroying species *Chaetomium globosum* was also shown by the same authors to be markedly stimulated, but this effect was believed to be due to an yet-unidentified factor in the jute extract.

In view of these previous findings it was considered worth while to investigate, in terms of actual loss in strength, whether the

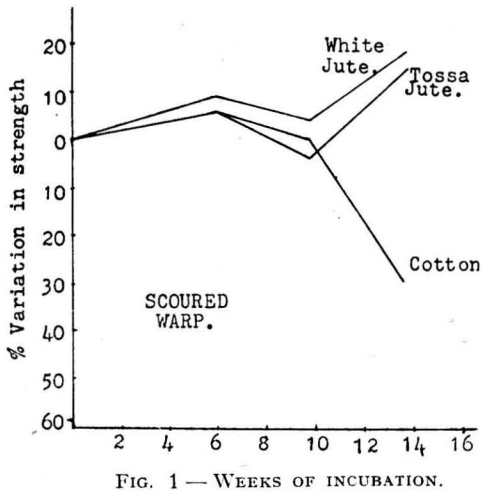


FIG. 1 — WEEKS OF INCUBATION.

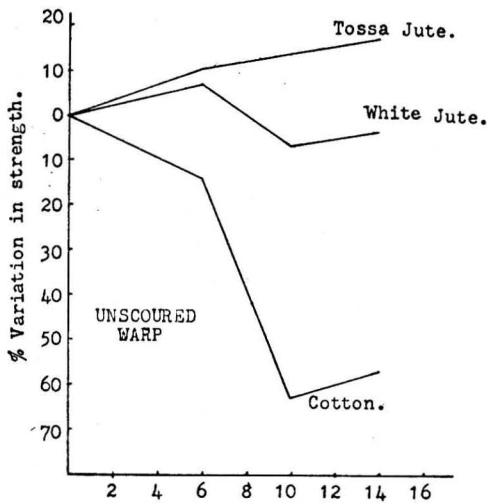


FIG. 2 — WEEKS OF INCUBATION.

water-soluble fraction of jute fibre did actually influence its deterioration and if so to what extent. The results presented below substantiate the expectation that the effect of the water-soluble constituents on the biological decay of the fibre is by no means negligible.

Experimental

In the first instance, the rates of deterioration of scoured and unscoured canvases made from white jute (*Corchorus capsularis*) and tossa jute (*C. olitorius*) and also from cotton were compared. Scouring was done by steeping in a solution of 1 per cent soap and 0.5 per cent soda ash for 1 hr. at 80°C.

The samples were rinsed in water and, after inoculation with soil extract, incubated in a humidity box highly charged with spores of numerous mildew fungi and containing water at the bottom. Portions were withdrawn at intervals and loss in tensile strength determined in the usual way. The results shown graphically in Figs. 1-4 demonstrate that scouring improved mildew resistance in all cases (see also FIGS. 5-8). Incidentally, it may also be noted that cotton appears to be more susceptible to rot than jute, and that there was no definite difference between tossa and white jutes so far as this property was concerned.

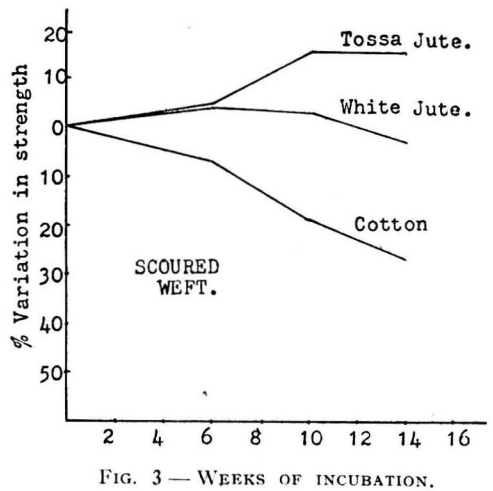


FIG. 3 — WEEKS OF INCUBATION.

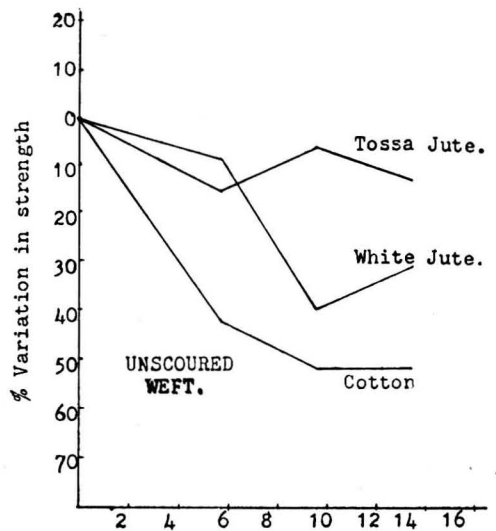


FIG. 4 — WEEKS OF INCUBATION;

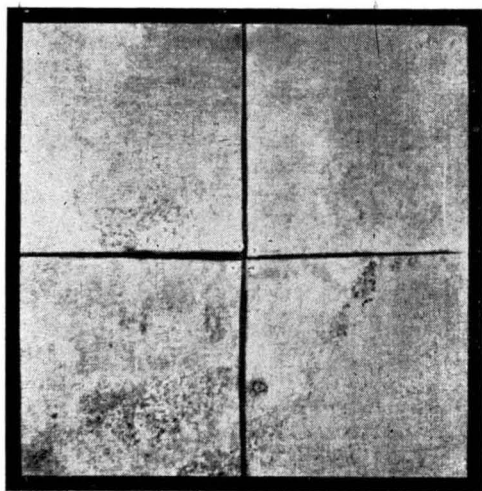


FIG. 5 — UNSCOURED JUTE (TOSSA).

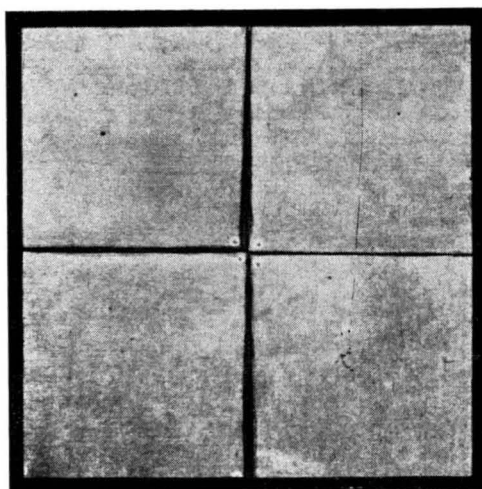


FIG. 6 — SCOURED JUTE (TOSSA).

While scouring much improved mildew resistance, this treatment probably removed somewhat more matter than the water-soluble fraction. In the next experiment samples of hessian cloth and jute fibre were leached in boiling distilled water for 1 hr. After air drying, these were inoculated by pressing against a heavily mildewed cloth. The samples along with the untreated controls were incubated over water in a desiccator. The day temperature varied from 23°-38°C. Microscopic examinations were made at monthly intervals with the following results (TABLE I).

At the end of 3 months, the fibre samples were subjected to the microscopic test devised by Macmillan and Basu for the detection of damage on jute¹². Briefly, the test consists in swelling a number of fibres according to a particular procedure when damaged and undamaged fibres appear different. The percentage of damaged fibres is expressed as "damage count". This factor is a measure of the tensile strength inasmuch as it has been found to increase with decreasing strength of the sample. Weighed portions of the test samples and of a normal

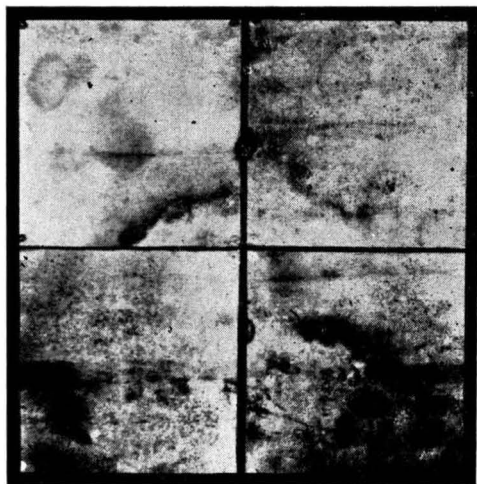


FIG. 7 — UNSCOURED COTTON.

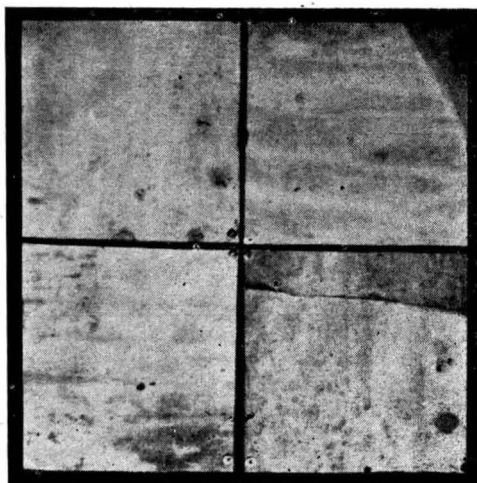


FIG. 8 — SCOURED COTTON. (THE STRAIGHT-EDGED PATCHES ARE NOT DUE TO MILDEW.)

TABLE I—FUNGAL GROWTH ON LEACHED & UNLEACHED MATERIALS

INCUBATION PERIOD	UNLEACHED		LEACHED	
	Fabric	Fibre	Fabric	Fibre
1 month	Moderate growth	No detectable growth	Mild growth	No detectable growth
2 months	Moderate growth; visible peritheca of <i>Ch. indicum</i>	Visible peritheca of <i>Ch. indicum</i>	Mild growth	No detectable growth
3 months	Heavy growth; numerous peritheca of <i>Ch. indicum</i>	Moderate growth; many peritheca of <i>Ch. indicum</i>	Mild growth; no <i>Ch. indicum</i>	No detectable growth; no <i>Ch. indicum</i>

fibre were also plated out on a suitable medium to determine the nature and extent of growth. The results are set out in Table II.

Here also it is seen that attack on the unleached sample proceeds much more rapidly than on the leached fibre. It is interesting to note that *Chaetomium indicum* which actively decomposes jute and cellulose³ and which seems to be a native infection on jute frequently developing on the rotting fibre¹³ showed profuse growth on the unleached fibre but no growth on the leached sample. The *Phycomyces*, which are known to be devoid of any cellulose decomposing power, were absent on the leached fibre which suggests that these species thrive on the superficial nutrients.

The stimulating action of the water extract of jute was then more directly demonstrated by comparing the growth rates of species on a synthetic medium with and without added jute extract. Liquid Czapek-Dox medium (neutral) with 1 per cent glucose was the basal medium and growth on this was compared with that on the same medium plus an extract of 2 gm. of jute for every 100 c.c. In Figs. 9-10, the positive acceleration of growth by jute extract can be seen. Of the species tested, *Chaetomium globosum* and *Memnoniella echinata* are well-known cellulose decomposers, although rarely found on jute materials. That substances of the nature of growth factors stimulate the growth and sporulation of these species is already known^{8,11}. On the other hand, *Chaetomium indicum* and *Aspergillus terreus* grow and sporulate readily on ordinary synthetic media. It is interesting to note that these species

TABLE II—DAMAGE CAUSED & NATURE OF ATTACK ON LEACHED & UNLEACHED SAMPLES

TEST	UNINCUBATED FIBRE Normal	INCUBATED FIBRE	
		Leached	Unleached
Damage count	12-14	22	32
Fungal count (per gm.)	30,000	60,000	200,000
Fungal sp. isolated	<i>Pen. citrinum</i> , <i>Asp. terreus</i>	<i>Pen. citrinum</i> , <i>Asp. terreus</i> , <i>Asp. glaucus</i> , <i>Paccilomyces varioti</i>	<i>Pen. citrinum</i> , <i>Asp. terreus</i> , <i>Ch. indicum</i> , <i>Phycomyces</i>

which are frequently isolated from rotting jute materials are also markedly stimulated by jute extract (see FIG. 10).

Discussion

It is unlikely that the effect observed was due to the presence in the jute extract of nutrients such as particularly readily available sources of carbon or nitrogen. The extract was found not to contain any sugar, and the nitrogenous matter in it, although qualitatively an excellent nutrient for many species³, must have been quantitatively inadequate. In the last experiment reported above, calculation shows that jute

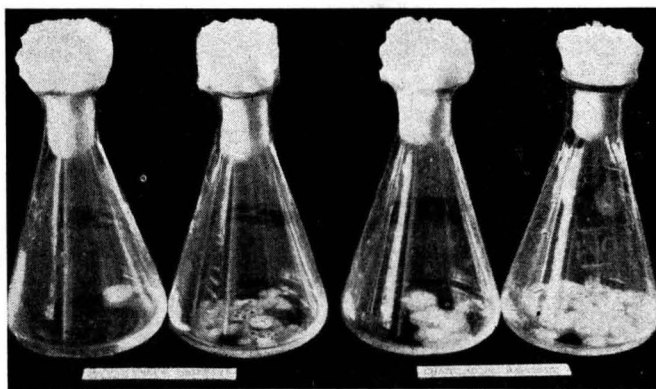


FIG. 9—GROWTH OF *Memnoniella echinata* (AT 22°C.) AND *Chaetomium globosum* (AT 30°C.) AFTER 6 DAYS, WITHOUT AND WITH JUTE EXTRACT.

extract supplied only some 6 per cent of the total nitrogen present in the medium. Moreover, it had been found previously in the case of *Chaetomium globosum* that organic nitrogen sources did not produce much better growth than nitrate⁸.

Fungi are also known to be markedly stimulated by traces of mineral elements¹⁴. Beeson¹⁵ has shown the presence of K, Ca, Mg, P and S in raw cotton. According to Marsh and Wood², raw cotton gives 1 per cent ash which has in it Ca, K, Mg, Al and Fe. Parsons¹⁶ reports 0.8-1.5 per cent ash in jute and he quotes Cross and Bevan as showing it to consist chiefly of Si, K, P with traces of Mn and Cu. Ca and Fe are also probably present. It is possible, therefore, that the stimulatory effect of jute extract is due to the oligodynamic action of mineral micro-nutrients. However, these elements are required in such minute traces that enough of them are probably present in synthetic media prepared from chemicals not specially purified, so that it would be difficult to explain the results shown in Figs. 9 and 10. That stimulation was caused by growth substances of vitamin nature remains, therefore, the most likely possibility, particularly in view of previous evidence as summarized in the introduction.

From the results obtained it would seem likely that the simple measure of hot water leaching, if applied on the finished goods or the raw jute fibre, should appreciably reduce mildew trouble in actual use of jute materials.

Summary

The water-soluble fraction of jute fibre has stimulatory action on many fungi including those known to attack strongly jute and cellulosic materials. This is probably due to the presence in it of growth factors of vitamin nature. Fibre and fabrics boiled in water actually showed increased mildew resistance.

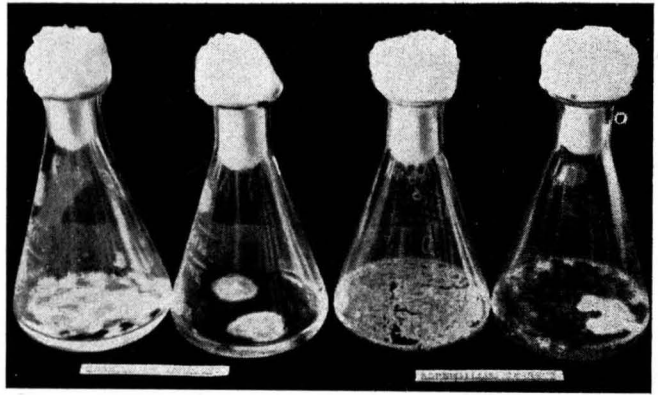


FIG. 10 — GROWTH OF *Chaetomium indicum* AND *Aspergillus terreus* AFTER 5 DAYS (AT 30° C.), WITH AND WITHOUT JUTE EXTRACT.

Acknowledgements

The author wishes to thank Mr. S. N. Ghose, M.Sc., for his help with some of the tests, and the Committee of the *Indian Jute Mills Association Research Institute* for permission to publish the results described.

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Water Content of Lime-puzzolana Mortars

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ADDITION of correct amount of water in lime-surkhi mortar has a direct bearing on its strength development. Studies carried out at the *Building Research Station, D.S.I.R., England*¹, show that 2 : 1 : 9 puzzolana : hydrated lime : standard sand mortar gives higher strength at 12 per cent moisture as compared with 14 and 16 per cent. Still further increase is noticed at 8 to 10 per cent moisture.

It has been shown by Khan and Verman² that dry mixes are superior to wet mixes. In this investigation two discrete values of consistency were used to determine the effect of consistency on the strength development of lime-surkhi mixes. The results obtained in these experiments indicated the superiority of only one value over the other. It was further pointed out by them that there seems to be one particular consistency at which the strength of lime-surkhi mortar is maximum. In the present investigation the scope of work was further extended and the strength of lime-surkhi mortar was determined at different water contents. The ratio of lime to surkhi was also one of the variables.

Experimental

Four samples of bricks were collected for the purpose of the present investigation : two from a brick-field, Roorkee, and two from a brick-field, Ballawali, a place twenty miles east of Roorkee. By visual examination the bricks were grouped into well-burnt and under-burnt bricks. It may be pointed out that bricks collected from these fields do not represent burnt clay alone but a mixture of sand and clay in unknown proportions. No attempt was made to ascertain the exact proportion of sand and clay mixture used in the manufacture of bricks, as the composition of soil changes from one brick-field to the other and in the same brick-field. Both types of bricks were powdered by means of a wooden hammer until the surkhi so obtained could be passed through a 30-mesh sieve.

The composition of the slaked lime used in the experiments is given in Table I. Before using it for the purpose of making test briquettes, it was also passed through a 30-mesh sieve.

TABLE I—CHEMICAL COMPOSITION OF LIME

Silica-SiO ₂	1.84
Al ₂ O ₃ +Fe ₂ O ₃	1.64
MgO	6.01
CaO	65.76
Ignition loss	24.37
Sulphuric anhydride	- 0.10
Alkaline salts as NaCl and KCl	0.20

The test briquettes were matured for 28 days. The initial maturing for 7 days was done in a damp box at a relative humidity of 90 to 95 per cent. For the rest of the period maturing was done under tap water containing 18 parts of soluble salts in 100,000 parts of water. The briquettes were matured at room temperature. The maximum and minimum temperatures were recorded during the day. The mean of average daily temperatures during the period of maturing are shown in Table II.

Batches of briquettes after maturing period were tested for their tensile strength. Each batch consisted of 6 briquettes. The average of test results was taken to be the tensile strength of the batch. Further the briquettes were gauged by the same operator under uniform conditions.

Further, 200 gm. of lime-surkhi mix was subjected to compaction in a compacting cylinder having a diameter of 2.5". It was compacted in one layer receiving 15 blows from a 6.6 lb. hammer falling through a height of 10".

Discussion

The variation of strength due to changes in the moisture content of lime-surkhi mortar under the test conditions is clearly demonstrated. It is seen that maximum strength at 1:7 and 1:4 lime-surkhi ratios is obtained between 25 to 28 per cent moisture

content, while it requires a higher water content to get the maximum strength at 1:1 lime-surkhi ratio.

It has been observed that when lime-surkhi mix is compacted, cracks are developed at a particular moisture content. The result of compaction experiments on surkhi sample B are given in Table III. It is to be noted from the results obtained that at 1:7 lime-surkhi ratio cracks are produced at 27.08 per cent water content, while at 1:4 and 1:1 lime-surkhi ratios the development of cracks occurs at 27.50 per cent and 33.90 per cent respectively. It may thus be concluded from the results shown in Table II and Table III that maximum tensile strength of lime-surkhi mix is obtained just below the moisture content at which cracks develop. The strength of the mix decreases as the

percentage of moisture in the mortar is raised above this limit.

It may be further pointed out that the minimum tensile strength of lime-surkhi mortar is obtained in the proportion of 1:1 as compared to the ratios 1:7 and 1:4 except in surkhi C where the strength is low on the whole. However, it is difficult to compare the strength of the mortars at 1:7 and 1:4 ratios as the difference in strengths shows such variations that it is easily masked by the effect of change in temperature.

Work carried out in *Building Research Station* in United Kingdom³ has shown that the temperature coefficient of the rate of strength development of lime-puzzolana-sand mixes is very high. It is also clear from the results of the present investigation that during maturing temperature has a

TABLE II—TENSILE STRENGTH OF LIME-SURKHI MORTAR AT DIFFERENT WATER CONTENTS

SAMPLE No.	RATIO OF LIME TO SURKHI								
	1:7			1:4			1:1		
	MOISTURE %	TENSILE STRENGTH IN LB. PER SQ. IN.	TEMP. °F.	MOISTURE %	TENSILE STRENGTH IN LB. PER SQ. IN.	TEMP. °F.	MOISTURE %	TENSILE STRENGTH IN LB. PER SQ. IN.	TEMP. °F.
A Well-burnt surkhi from Roorkee	22.9	46	74.3	22.8	45	72.5	27.6	10	69.8
	26.1	90		26.0	83		30.3	30	
	28.9	70		29.1	76		33.0	47	
	32.0	40		32.1	51		37.9	30	
	34.8	37		35.0	31		42.8	27	
B Under-burnt surkhi from Roorkee	23.6	103	83.4	23.0	54	88.5	29.0	100	90.2
	27.3	170		27.3	177		32.4	105	
	30.1	140		30.0	167		35.3	79	
	33.0	121		33.1	153		37.3	74	
	35.5	106		35.5*	135		39.1	64	
C Well-burnt surkhi from Ballawali	23.2	10	60.3	24.3	8	57.0	27.5	9	56.2
	25.0	11		27.5	12		30.6	14	
	27.2	11		30.4	12		33.4	12	
	30.4	10		33.5	6		36.5	11	
	33.4	7		35.1	5		39.5	10	
D Under-burnt surkhi from Ballawali	25.0*	102	100			69.9	29.8	72	73.3
	23.6	91		23.5	102		32.5	81	
	26.4	98		26.4	108		35.4	58	
	29.5	83		29.5	88		38.6	50	
	32.6	72		32.4	72		41.3	48	
	52	35.5	57	44.5	41				
	26.4*	153	100						

* represents tensile strength after 14 days maturing.

TABLE III—FORMATION OF CRACKS IN SURKHI SAMPLE B

No.	RATIO OF LIME TO SURKHI					
	1:7		1:4		1:1	
	MOISTURE %	FORMATION OF CRACKS	MOISTURE %	FORMATION OF CRACKS	MOISTURE %	FORMATION OF CRACKS
1	23.45	No cracks	23.70	No cracks	31.90	No cracks
2	24.96	do	25.44	do	33.50	do
3	25.50	do	26.37	do	33.90	Fine cracks
4	26.68	do	27.21	do	34.01	Cracks
5	26.96	do	27.50	Fine cracks	34.73	do
6	27.08	Fine cracks	27.67	Cracks	35.50	do
7	27.18	Cracks	28.55	do	36.30	do
8	27.52	do	29.95	do

marked effect on the strength development of lime-surkhi mixes; the higher the temperature, the greater is the strength developed in a given time. Comparing the results of well-burnt surkhi C and under-burnt surkhi B we find vast differences in strength. This much of difference cannot be explained due to a difference in the chemical composition of surkhi. The effect of temperature is also to be considered. In order to confirm this point further samples of surkhi C and D from Ballawali were matured for a period of 14 days at 100°F. It is clear from the results obtained that after 14 days, tensile strengths of the two samples matured at 100°F. are much higher than the same samples matured at room temperatures for 28 days (TABLE II). So it leads us to the fact that in all studies on lime-surkhi mortars the effect of temperature on its strength development is of fundamental importance. It may be mentioned here that Singh and Bahri⁴ did some work on the incorporation of puzzolana in kanker lime. Better results could have been obtained if the workers had taken into consideration the effect of temperature also. Unless a systematic study of the subject is made, it may be too early to suggest that when early strength development in a mortar is desired, the construction with lime-surkhi mortar should preferably be undertaken in the summer months of the year.

Investigations carried out at *Building Research Station* in U.K.⁵ have established that for every clay there exists a temperature of firing (between 600°-800°C.) which gives it the highest activity. Heating at a temperature higher than 1,000°C. appears to destroy the puzzolanic activity of clay. Studies of Khan and Verman² conclusively proved that higher temperature of burning gave higher strength of surkhi when used in lime mortar. However, in the above investigations there are samples of under-burnt surkhi which show, at initial stages of maturing, superiority over well-burnt surkhi. In the present investigation also under-burnt surkhi seems to give better early strength as compared with well-burnt surkhi. Probably the chemical composition of clays plays an important part in determining the optimum temperature at which a puzzolana of highest activity is obtained. It may be mentioned here that we have 2 major soil groups in this country: brown to black soils of the Deccan Trap and the Indo-Gangetic alluvium. Besides, there are red soils of Madras and lateritic soils of Mysore.

These soils differ widely in their physical properties and chemical composition. It may be pointed out that results obtained by Khan and Verman are based only on surkhi made from Bengal soils. Though the clays used in the experiments were of varied nature, still it might be interesting to extend the scope of the work to other types of soils available in different parts of the country.

In practice one finds that wet consistency is often employed. This practice has proved to be harmful from tensile strength point of view. Moreover, as shown above, when lime-surkhi mortars are subjected to compression at wet consistency, cracks are developed. This provides a greater scope for the penetration of rain in the structure.

It may be pointed out here that mortars which exhibit high tensile strength are not necessarily those which adhere most strongly to all types of bricks. A high tensile strength is valueless unless there is a good adhesion to the brick on each side. Thus, actual recommendations as to the quantity of the water to be used in lime-puzzolana mortar in actual construction cannot be made unless a study of the effect of moisture on the adhesive strength of the mortars is also made. Proper allowance will have to be given for the absorption of water by the brick from the mortar.

CONCLUSIONS

There is a critical moisture content value at which the early strength of lime-surkhi mortar is maximum. Further, when lime-surkhi mix is subjected to compaction at higher moisture content, cracks are produced.

As the percentage of lime increases in the mortars, the quantity of water required to get the maximum strength also increases.

The early strength of lime-surkhi mix at 1:1 ratio is the minimum as compared to other ratios, except in surkhi C.

The effect of temperature on the strength development of mortar is very pronounced. Higher rate of strength development is obtained at higher temperature.

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