# Journal of Scientific & Industrial Research

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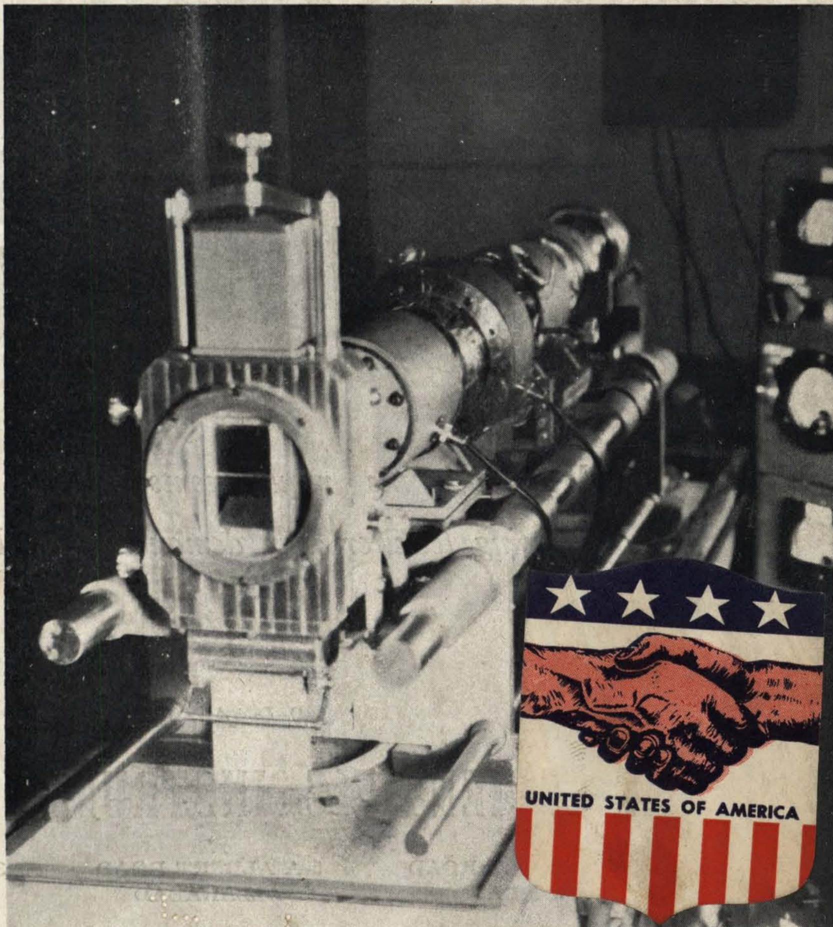
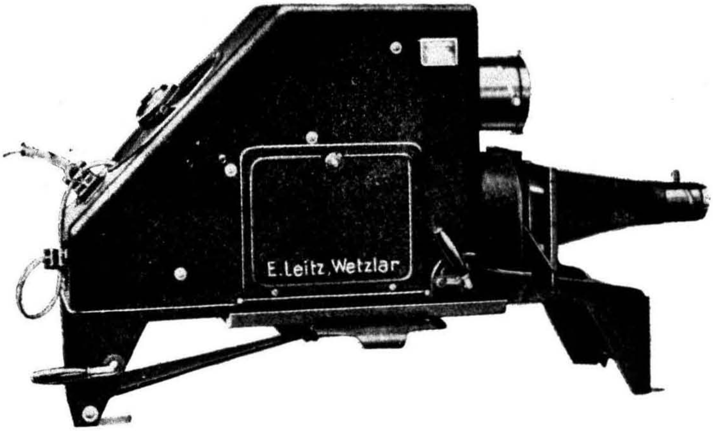


Vol. VIII MAY 1949 No. 5


E. Leitz, Wetzlar

Journal of
# Scientific & Industrial Research

**Vol. VIII** **MAY 1949** **No. 5**

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### COVER PICTURE

Picture on the cover shows a view of the horizontal electron microscope designed at the University College of Science, Calcutta. Designed to give a maximum magnification of 20,000 dia., the instrument has several distinctive features (*see* page 200).

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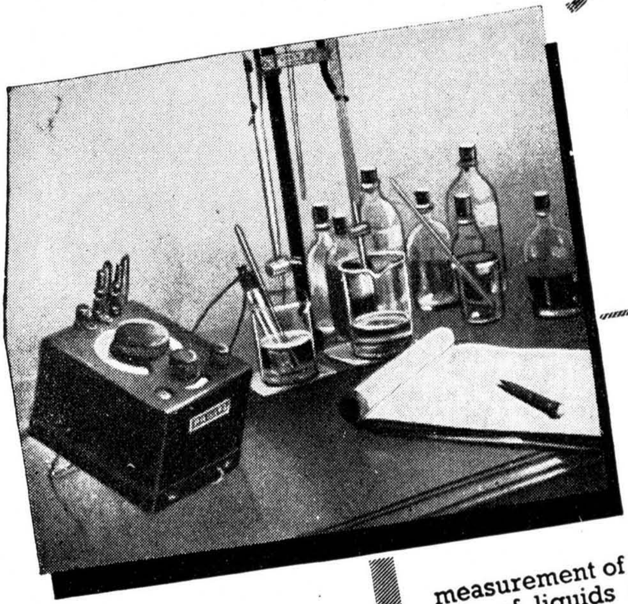

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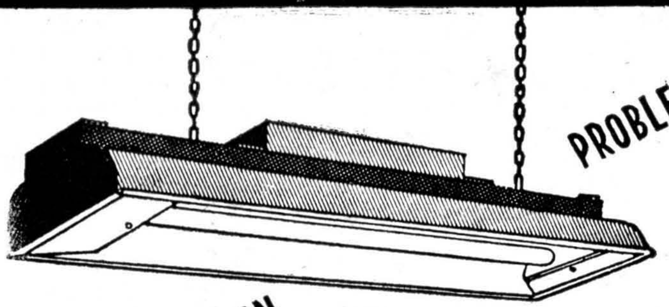

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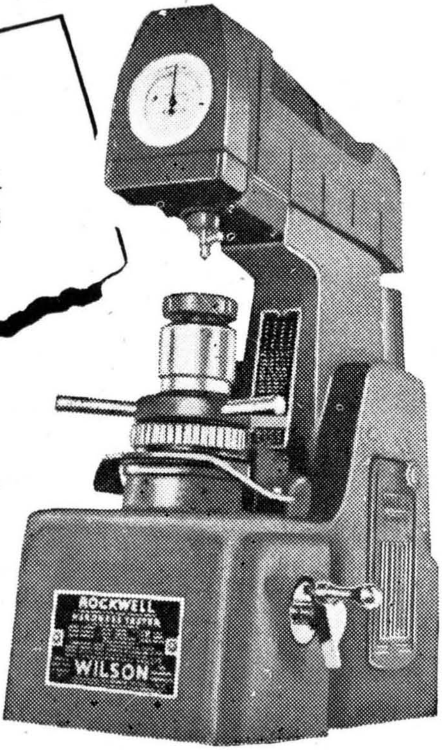
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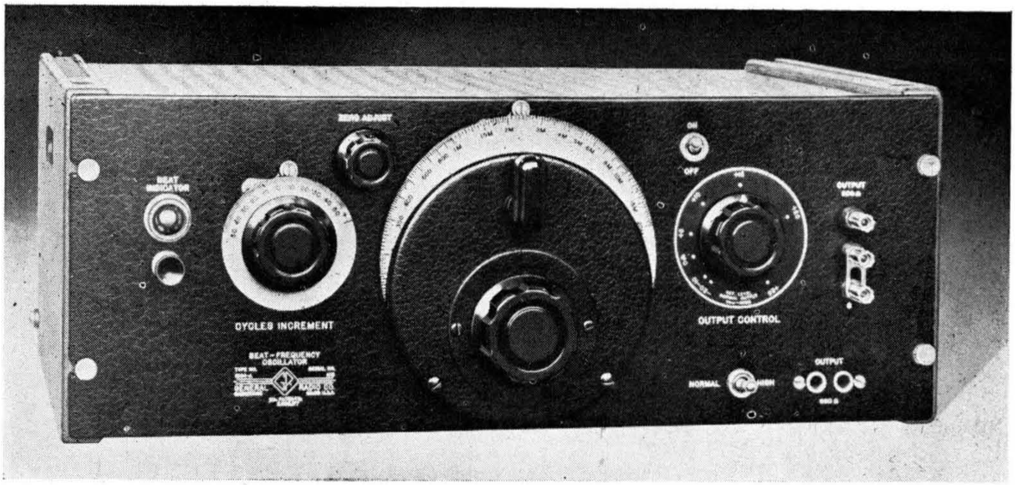
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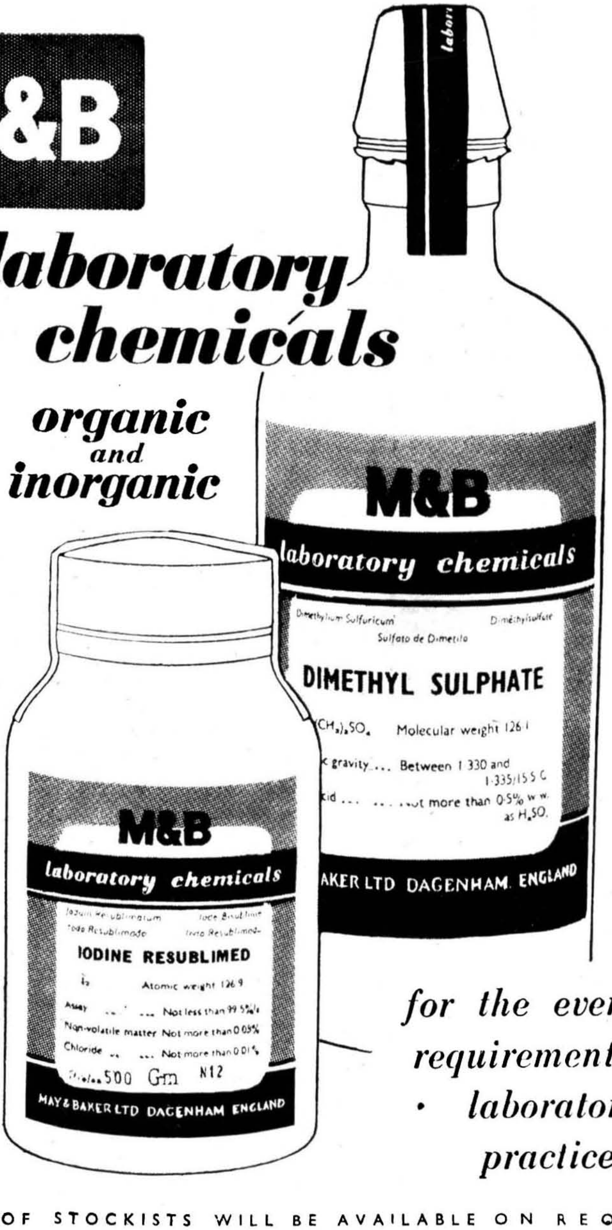
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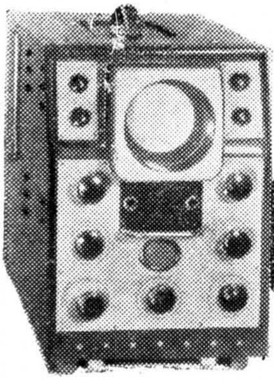

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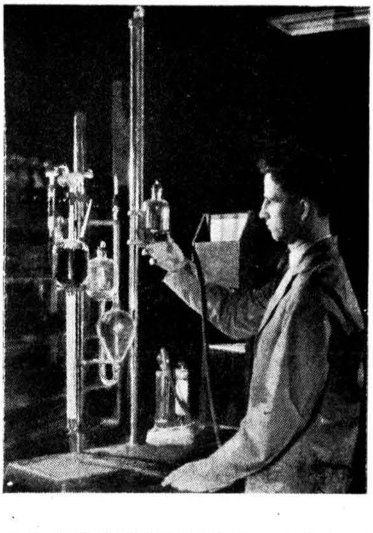

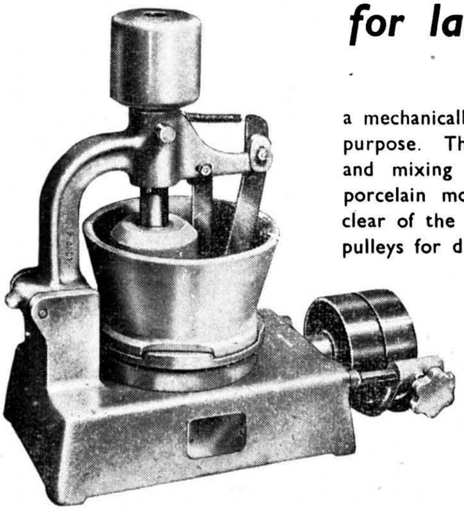

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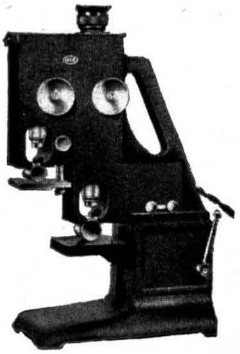

# Stabilization of Plant Names

"INTENDING to write a semi-popular book about the common trees of Malaya, I imagined that their botanical names would have been fixed. But I soon discovered that this was far from being the case. Indeed it was not uncommon to find that what was apparently the same tree was referred to by different names by each of several different authors. Thus, when Hooker, Trimen, Valeton, Merril, and other authors of standard systematic works concerning the flora of Malay Peninsula and adjoining territories showed little uniformity in nomenclature, it was not surprising to find the nomenclature of such standard economic works, such as those of Watt, Heyne and Burkill, to say the least, unreliable... For my own satisfaction, I have adopted the principle that no alteration in botanical names should be accepted unless supported by botanical proof by which means only fact, rather than opinion, becomes the basis of argument."

So wrote Dr. E. J. H. Corner* in 1939. The difficulties which confronted Dr. Corner are familiar to all those concerned with the study of plant life, and arise from the single fact that the *same* plant is often referred to by different authors by different scientific names in different publications. Even a cursory reading of journals devoted to systematic botany would be sufficient to show how changes in plant names are being constantly made. This is perhaps inevitable in a science like botany which is not an exact science in the sense that mathematics, for example, is. Researches constantly bring out fresh evidence for the validity or otherwise of names, and lead to clearer understanding of the relationships among plants. Unfortunately, however, systematists are far too few and the number of plants far too large, so much so that considerable time and labour will be required for locating all the errors and mis-applications perpetuated in literature and rectifying them. In the meanwhile, the difficulty, to which Dr. Corner has given such eloquent expression, continues to confront and confuse those concerned with the use of names for designating plant species.

Too often we find that a name published after an apparently thorough search has been changed a few years — sometimes even a few months — later. There have been numerous cases in which the name of a plant has undergone twenty or more alterations. There are instances where a name considered invalid for years has been revived, shown to be valid, and adopted. *Setaria geniculata* (Linn.) Beauv. has 73 synonyms, *Koeleria cristata* (L) Pers. has 41, and *Echinochloa crusgallii* (L) Beauv. has 46. Arber,* a recognized authority on grasses, who examined the botanical names of the pearl millet, says: "The history of names which have been applied to it (pearl millet) forms an ironical commentary on the botanist's claim that the use of a Latin terminology results in precision of meaning. This plant, early in the nineteenth century, possessed almost as many Latin names as there are floras. It has been called *Pennisetum typhoideum*, *P. alopecuroides*, *P. americanum*, *Penicillaria spicata* and *Panicum spicatum*. Of these *Pennisetum typhoideum* Pers. has been the most popular in recent years."* This name has since been displaced by *Pennisetum typhoides*, and, in more recent years, *Pennisetum glaucum* has been suggested as the correct name for it. Instances like this are indicative of the confusion involved in designating plants and the imperative necessity for arriving at a definite understanding on the selection and use of plant names.

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*Gardens' Bulletin*, Straits Settlement, 1939, **10**, 2.

**The Graminae*, 1934, p. 24.

Differences in the interpretation of rules for naming plants have added to the confusion, and even in cases where only nomenclatural, not taxonomic, issues are concerned, different names have been considered valid by different botanists for one and the same plant. However, nomenclatural changes are governed, at least to some extent, by international rules, but changes based on taxonomic considerations are beyond the scope of any rules that can be framed. They depend, to a large extent, on the judgement of individual botanists. A valid name of one author may be the invalid name of another, and it is impossible to reconcile them. *Caesalpinia pulcherrima* (L) Swartz and *Poinciana pulcherrima* L. are names of one and the same plant. So are *Chloranthus brachystachys* Bl. and *C. glaber* ( Thunb.) Makino ; *Cleome icosandra* L. and *Polanisia icosandra* L. ; and *Brassica alba* Boiss. *B. hirta* Moench. and *Sinapsis alba* L.

There are other factors which add to the confusion in names. *Cassia obtusifolia* L. has been considered by some to be synonymous with *C. tora* L., while others consider the two to be entirely different plants. This confusion is further heightened by the suggestion that the correct name for *Cassia tora* is *Emolista tora* (L) Britton & Rose. Similarly, *Cerbera odollam* Gaertn. is considered by some to be synonymous with *C. manghas* L., and others hold that the two are entirely different plants. When authorities differ, whose is the opinion to be accepted by a person who " uses names as appellations for plants and not as botanical footballs " ?*

### Is the Confusion Deepening ?

Discrepancies are not uncommon even in the citation of authorities for plant names. Thus *Calotropis gigantea* is variously cited as *Calotropis gigantea* R. Br., *Calotropis gigantea* ( Willd.) R. Br., *Calotropis gigantea* ( Willd ) Dryand, *Calotropis gigantea* ( Dryand ) Ait., *Calotropis gigantea* ( Linn. ) Dryand, and *Calotropis gigantea* Ait. There can be one and only one correct author citation for this plant name, or for that matter, for any other plant name. All other citations must be wrong.

Protests against too many and too frequent changes in plant names have been made from time to time by botanists and non-botanists alike. Such changes are all the more exasperating when they concern economic plants. Stabilization of plant names is the aim of systematic botanists, but they proceed to achieve this end by discovering the earliest valid name for each plant. Unfortunately, however, names considered " earliest " at one time have often been found to have earlier valid names, the discovery of which invalidates the existing names although they may have been in use for years. A systematic and rigid application of international rules of botanical nomenclature will result *ultimately* in the stabilization of plant names, but only in those cases where taxonomy is not involved. But even for this, one has to await patiently the results of extensive researches by large teams of workers well provided with ample laboratory, library and herbarium facilities. When we come to the taxonomic aspect, the position is even more discouraging. Studies on the taxonomy of economic plants are in an amazingly backward state. The literature on many genera is confused. Who is to be accepted as authorities when monographers and specialists differ ? Errors in a monograph can be discovered only by another subsequent and exhaustive monographic research. Usually they have a tendency to persist and perpetuate themselves !!

### Can Usage be Ignored ?

The great inconvenience caused by nomenclatural changes, at least so far as economic plants are concerned, has been well recognized and voiced by many botanists. Thus Bailey wrote : " Botanical names should not be changed lightly for the purpose of regularizing any particular scheme or plan or to make them always conform to an arbitrary set of rules. Botanical names do not belong to botanists to do with them as they will. The public has good rights in these names, and this is particularly true in the names of cultivated plants, for they may then have standardized commercial value. The only stability, of course, is usage, and usage can rarely be forced into hard and fast regulations."* The insistence on usage and the clear recognition of the purpose served by the names given to plants are noteworthy. Chamberlain, the well-known authority on gymnosperms, is reported to have stated : " We believe that when a name has been established in literature, it should be retained. . . Taxonomists

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*GLEASON : *Phytologia*, 1947, **2**, 202.

*Standard Cyclopaedia of Horticulture*, 1947, **1**, xv.

would do well to let familiar, well-established names alone, and not try to revive some forgotten name."* The efforts of botanists should be diverted to the elimination of confusion due to multiplicity and instability of plant names. As Gleason has said: " If botanists search as assiduously for reasons to maintain a name as they do for reasons to change one, a considerable number of well-known names will be saved."†

Pleas for *nomina specifica conservanda* brought before the International Botanical Congress have not been hitherto accepted. At the last session of the Congress (1935), the proposal was defeated by a majority of 147 votes. It is almost certain that specific names will never be officially conserved. What is feasible, however, is the preparation of an agreed list which all botanists should accept as valid for a specified number of years, and the International Botanical Congress should be accepted as the only authority for introducing changes in plant names. This would minimize confusion and prevent the introduction of hasty changes in names by individual workers. There has been no limit to such changes, and many controversies, often bitter, have centred round the validity of suggested or existing plant names. Confusion can be minimized if authors, who find the need for revising plant names, first obtain the approval and support of an international body of botanists. A resolution passed at the last session (1935) of the International Botanical Congress envisaged the appointment of a Committee to draw up a list of names of economic plants according to international rules, which should remain in use for ten years. This is a step in the right direction. Unfortunately, that list has not been published so far. We trust that the matter would be taken up at the forthcoming session of the Congress, and that effective steps would be taken to introduce reasonable stability in so fundamental a requirement as a name for a plant.

V. S. Rao

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* *Chronica Botanica*, 1943, **7**, 439.

† *The preservation of well-known binomials — Phytologia*, 1947, **2**, 203.

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# Characteristics of the Ionosphere over Calcutta (February 1949)

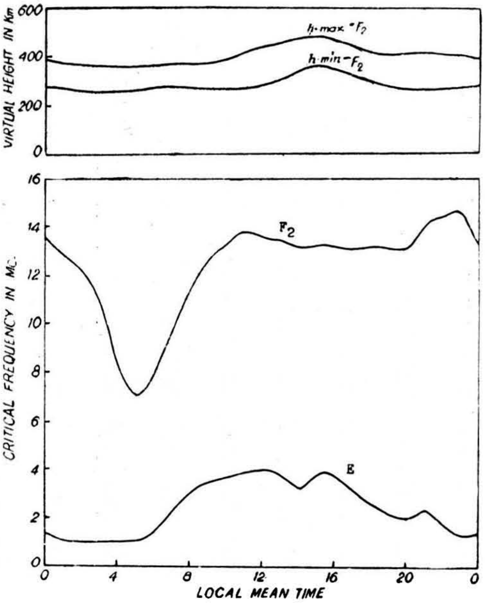
S. S. BARAL, S. N. GHOSH, R. K. MITRA, D. C. CHOUDHURY & (MISS) T. K. PAL
*Wireless Laboratory, University College of Science, Calcutta*

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

Fig. 1 represents the mean hourly values of the virtual heights and critical frequencies of the $F_2$ layer and the critical frequencies of the E layer in graphical form during the month of February 1949. The mean of both normal and abnormal values is presented.

During this month there was high sunspot activity, and day to day observations show that on almost every day the F region attained great heights and thicknesses during daylight hours and high ionization densities during the corresponding heights. The abnormal E ionization was also high during the afternoon and night hours.

Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmissions during May 1949 by reflection at the F region over Calcutta, based on the normal values of the data for February 1949. Table I gives occasions when the E-region ionization was found to be abnormal and the corresponding penetration frequencies and heights.

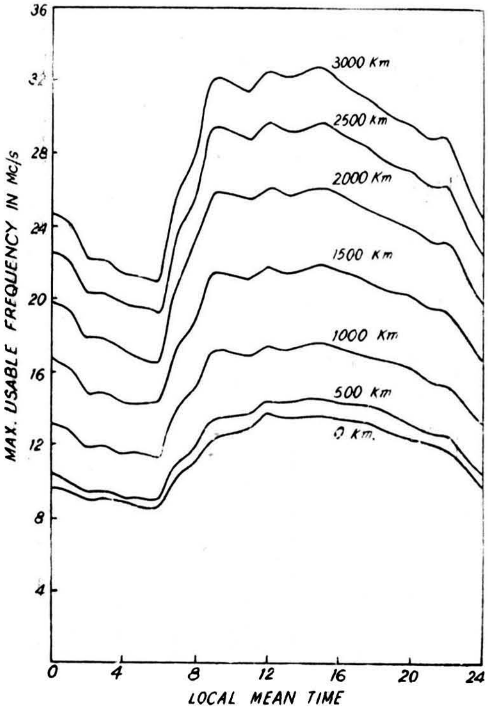


( 5 HOURS 54 MINUTES AHEAD OF G.M.T. )

FIG. 1 — FEBRUARY 1949.

TABLE I

| MONTH & YEAR | DATE | HOUR | $f^{\circ}E_s$ | $^{h}E_s$ |
|---|---|---|---|---|
| | | | Mc. | Km. |
| Feb. 1949 | 11 | 14.00 | 4·75 | 120 |
| | 14 | 18.00 | 3·30 | 120 |
| | 16 | 17.00 | 5·00 | 135 |
| | | 18.00 | 6·60 | 135 |
| | | 21.00 | 4·85 | 120 |
| | | 22.00 | 4·90 | 120 |
| | 17 | 00.00 | 3·20 | 105 |
| | 18 | 01.00 | 3·20 | 105 |
| | | 02.00 | 3·45 | 105 |
| | | 03.00 | 3·55 | 105 |
| | | 04.00 | 3·20 | 105 |
| | 19 | 14.00 | 4·70 | 120 |
| | | 21.00 | 3·00 | 120 |
| | | 22.00 | 3·00 | 120 |
| | | 23.00 | 3·50 | 120 |
| | 20 | 00.00 | 4·50 | 120 |
| | | 01.00 | 4·70 | 120 |
| | | 02.00 | 4·65 | 120 |
| | | 16.00 | 4·70 | 135 |
| | | 17.00 | 4·70 | 135 |
| | | 23.00 | 3·20 | 105 |
| Feb. 1949 | 21 | 13.00 | 4·80 | 120 |
| | | 14.00 | 4·80 | 120 |
| | 22 | 05.00 | 4·25 | 90 |
| | | 06.00 | 3·00 | 90 |
| | 23 | 18.00 | 3·60 | 120 |
| | | 19.00 | 3·50 | 120 |
| | 24 | 22.00 | 3·60 | 105 |
| | | 23.00 | 3·50 | 105 |
| | 25 | 00.00 | 3·25 | 90 |
| | | 08.00 | 3·50 | 105 |



AT POINT OF REFLECTION.

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

# The Prospects of Salt Raising in the Coastal Regions of West Bengal & Orissa

JITENDRA KUMAR NAG
*Special Officer ( Salt ), West Bengal*

THE object of this investigation was to ascertain the prospects of salt raising along the coast-line of West Bengal and Orissa by comparing the climatic factors of the littoral of these two provinces with those of Bombay and Madras, where the salt industry has developed on a wide scale. The possibilities of reviving salt culture in these provinces have been examined by salt experts from time to time since World War I, but no serious attempt has been made so far to implement the recommendations of the experts who found in West Bengal suitable sites for establishing the salt industry.

The first inquiry ( non-official ) was undertaken in 1918 by the late Mr. Kapilram Vakil of Bombay, on behalf of *Messrs Tata & Sons*. Mr. Vakil's investigations were confined to the regions lying to the north of Chilka Lake, which he found to be unfavourable for the economic production of salt. The suitability of the regions near and around the Chilka Lake for salt production had been examined more recently by Mr. F. W. Gooch, who was appointed by *Messrs Kilburn & Co.* to survey the coast-land of Bengal and Orissa. The results of this survey have not been published so far.

There is no record of official investigations undertaken during 1918-24. The Central Board of Revenue, however, put forward the view : " The possibility of manufacturing salt ( in Bengal ) on a commercial scale has been examined by the Government on many occasions only to be rejected. It is rendered impracticable by the low density of the sea water due to enormous discharge of fresh water from the great rivers, the prevalence of damp, cloudy weather, the scarcity of fuel for one method of manufacture ( *pãnga* ) which in any case could hardly yield an output on any magnitude, and the occurrence of storms at critical seasons."

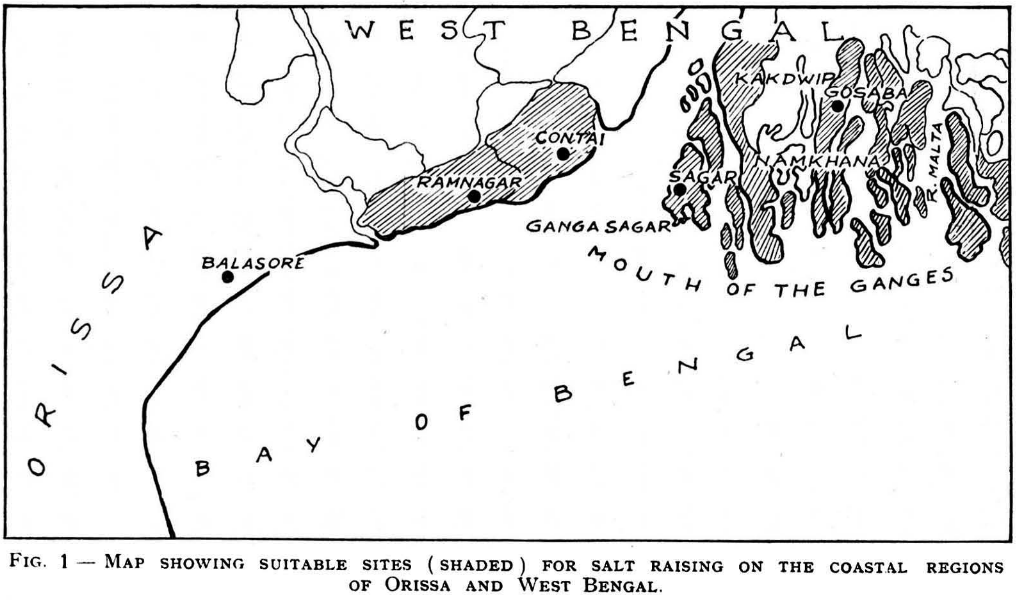


FIG. 1 — MAP SHOWING SUITABLE SITES ( SHADED ) FOR SALT RAISING ON THE COASTAL REGIONS OF ORISSA AND WEST BENGAL.

TABLE I — ANALYSIS OF BRINE

| | DENSITY °Be. | SODIUM CHLORIDE % | MAG. CHLORIDE % | MAG. SULPHATE % | CAL. SULPHATE % | CAL. CARBONATE % |
|---|---|---|---|---|---|---|
| *West Bengal* | | | | | | |
| Contai | 2·7 | 2·2 | 0·24 | 0·19 | 0·1 | 0·01 |
| Sunderbans | 2·8 | 2·2 | 0·27 | 0·19 | 0·1 | 0·01 |
| *Orissa* | | | | | | |
| Balasore | 3·0 | 2·4 | 0·29 | 0·20 | 0·1 | 0·01 |
| *Madras* | 2·9 | 2·6 | 0·28 | 0·18 | 0·13 | ... |
| *Mediterranean Sea* | 3·0 | 2·7 | 0·61 | 0·7 | 0·04 | 0·02 |

TABLE II — AVERAGE R.H. ( 1940-44 )

| PLACE | MONTHS | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | SALT SEASON | | | | | | | | | | | |
| | Jan. | Feb. | Mar. | Apr. | May | June | July | Aug. | Sep. | Oct. | Nov. | Dec. |
| Bombay | 76 | 74 | 77 | 75 | 76 | 82 | 87 | 86 | 86 | 82 | 72 | 70 |
| Sagar Island ( West Bengal ) | 62 | 61 | 73 | 80 | 79 | 79 | 84 | 83 | 80 | 75 | 66 | 63 |
| Madras | 82 | 83 | 82 | 77 | 69 | 64 | 67 | 73 | 76 | 84 | 88 | 89 |
| Vizagapatam | 77 | 80 | 78 | 75 | 74 | 76 | 79 | 77 | 82 | 78 | 70 | 73 |
| Balasore ( Orissa ) | 75 | 72 | 70 | 72 | 73 | 80 | 83 | 85 | 81 | 81 | 74 | 73 |

It may be mentioned here that the salinity of the bay water in different parts of the coast of Bengal had not been properly tested till 1937, in which year 2 officers of the provincial Government recorded the density of the sea water in the estuaries of the Sunderbans. Six years earlier Mr. C. H. Pitt of *Khewra Salt Mine* examined the extensive saliferous coast-land of Bengal on behalf of the Salt Industry Committee, but he visited only Midnapore and Western Sunderbans, leaving out the entire East Bengal seashore region. He was of the opinion that salt raising was not impossible, and suggested the starting of experimental units by private enterprise.*

In 1937 Mr. D. N. Mukerji, now Salt Controller, Government of India, and Mr. V. S. Rao of the Bengal Forest Service undertook an enquiry into the possibilities of developing the salt industry in the Sunderbans area. They stated: " We do not agree with Mr. Pitt that the salinity of sea brine on the Bengal coast never exceeds 1·5°Bé. Mr. Pitt did not visit the Sunderbans to the east of Lothian Island and had therefore no opportunity to study the salinity of the water beyond Lothian Island."

## Factors Affecting Salt Production

The raising of salt by solar evaporation is influenced by five factors, viz. : ( i) salinity of brine ; (ii) relative humidity ; (iii) rainfall and number of rainy days ; (iv) maximum and minimum temperatures ; and (v) wind velocity.

(i) *Salinity* — During the usual season of salt manufacture — December to May — the density of the bay water of Bengal averages to about 2·5°Bé., being almost equal in strength to that employed in the salterns of Madras and Bombay.

It is evident from the analyses that the strength of the sea water of West Bengal is a little less than that in Madras and in the Mediterranean, but not so low as to materially affect salt manufacture. Weak brine evaporates quickly in the initial stages, and does not render salt manufacture difficult or unremunerative.

(ii) *Relative Humidity* — The relative humidity influences the rate of evaporation of the brine. It has been observed, however, that when the temperature is high, and the sun and dry winds prevail, a humidity of 70-75 per cent does not greatly hinder salt raising.

The following is a comparative study of the average humidity for 5 years in places where salt is or can be manufactured on a big scale.

The figures indicate that the relative humidity of the Bengal delta is almost similar to that of the coastal part of eastern or western India. During the dry months of December to March, the West Bengal littoral is more dry than Bombay or

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* Report on the investigations into the possibilities of salt production in Bengal and Orissa, 1932.

Madras coast. The average relative humidity for 5 years for the period December to June is 71 per cent in Sagar Island ( West Bengal coast-land ), and 76 in Bombay. In tropical climates a period of dry weather prevails only for a short while during the winter months, and the atmosphere is moist during the rest of the year. The lands situated beyond 24°N. are arid and afford a suitable climate for sea salt culture and for that reason Karachi and Okha have developed their salt industry extensively. But moist localities like Bombay and Madras, as a whole, produce more salt than the out-turn of Karachi and Okha works taken together.

(iii) *Rainfall & Rainy Days* — In the manufacture of salt, the initial brine from sea or salt lake is concentrated only by solar evaporation till it attains a density approaching its saturation point. The concentrated saline is either led into solar pans ( crystallizing beds ) or into boiling pans for the deposition of salt crystals. Whichever method is adopted, a rain-free period is essential for the smooth working of a salt factory.

Rains not only dilute saline but also arrest evaporation. For this reason the salt season closes before the monsoon breaks out. The *intensity* of rainfall during the monsoon months does not affect salt manufacture as the *duration* of the rainy period. The factor for consideration in regions with heavy annual rainfall is the distribution of rainfall during the salt season irrespective of the intensity during the off season.

The rainy season in West Bengal and Orissa, as in Bombay and Madras, covers about 4 months. In Bengal as well as in Bombay it begins in June and is almost over by September. In Madras the season starts later and continues up to December. Salt raising generally starts towards the end of January and closes before June, but in Madras the salt pans may be worked up to August or even September. Occasional showers do not greatly hinder the sequence of salt culture if they are followed by clear days and sunshine. Table III gives the rainfall of West Bengal and Orissa coast-line during November to August.

The normal rainfall in Contai or Gosaba during the salt season is less than the normal of Gopalpur ( Naupada ) and Cocanada. The normal rainfall of Madras during the non-monsoon months, that is from January to August, is 14″ and the average number of

**TABLE III — AVERAGE RAINFALL IN INCHES & NUMBER OF RAINY DAYS ( 1940-44 )**

*January to May are the months during which salt is actually manufactured in Naupada ( Gopalpur ), Cocanada, Bombay, Contai and Sagar of West Bengal. In Madras salt is raised from February till the season ends in August or September with the advent of the rains.*

| | NOVEMBER | | DECEMBER | | JANUARY | | FEBRUARY | | MARCH | | APRIL | | MAY | | JUNE | | JULY | | AUGUST | | NON-MONSOON MONTHS | | ANNUAL | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Fall | Days | Fall | Days | Fall | Days | Fall | Days | Fall | Days | Fall | Days | Fall | Days | Fall | Days | Fall | Days | Fall | Days | Fall | Days | Fall | Days |
| Gopalpur | 3·9 | 2·4 | 0·5 | 1·0 | 0·3 | 0·6 | 0·9 | 1·0 | 0·6 | 3·0 | 0·7 | 1·8 | 2·1 | 3·2 | 5·5 | 7·4 | 7·2 | ... | 7·7 | ... | 9·1 | 13·0 | 45·0 | ... |
| Cocanada | 4·9 | ... | 0·4 | ... | 0·2 | ... | 0·1 | ... | 0·9 | ... | 1·1 | ... | 1·4 | ... | 4·3 | ... | 6·1 | ... | 3·2 | ... | 8·9 | ... | 41·0 | ... |
| Madras | ... | ... | 5·8 | 5·2 | 1·4 | 1·7 | 0·3 | 0·6 | 0·2 | 0·3 | 0·5 | 0·7 | 1·1 | 1·1 | 1·9 | 3·7 | 3·9 | 7·0 | 4·6 | 7·9 | 14·0 | 23·0 | 50·8 | 57·3 |
| Bombay | 0·1 | ... | 0·1 | ... | 0·8 | ... | 0·1 | ... | 0·1 | ... | ... | ... | 1·2 | ... | 25 *app.* | ... | 30 *app.* | ... | ... | ... | 2·4 | ... | 71·0 | ... |
| Contai | 1·1 | 1·6 | 0·1 | 0·4 | 1·0 | 1·5 | 0·7 | 1·2 | 0·8 | 2·9 | 1·4 | 2·9 | 3·8 | 6·0 | 9·4 | 13·4 | ... | ... | ... | ... | 8·8 | 16·5 | 62·6 | 90·8 |
| Ramnagar | 1·7 | ... | 0·1 | ... | 1·1 | ... | 0·8 | ... | 1·5 | ... | 1·1 | ... | 2·3 | ... | 7·6 | ... | ... | ... | ... | ... | 8·7 | ... | 55·9 | ... |
| Sagar | 1·5 | 1·4 | 0·3 | 0·5 | 0·3 | 0·9 | 1·1 | 1·8 | 1·4 | 2·0 | 1·1 | 2·1 | 4·4 | 5·8 | 11·5 | 12·3 | 14·6 | 15·8 | 14·2 | 16·5 | 10·1 | 14·5 | 69·4 | 79·6 |
| Gosaba | 0·7 | ... | ... | ... | 0·4 | 0·9 | 0·9 | ... | 0·9 | ... | 1·5 | ... | 2·8 | ... | 10·3 | ... | 15·3 | ... | 15·7 | ... | 7·2 | ... | 61·7 | ... |
| Balasore | 1·5 | 1·5 | 0·2 | 0·3 | 0·5 | 1·0 | 1·4 | 2·4 | 1·8 | 2·9 | 1·7 | 3·5 | 4·6 | 5·9 | 9·2 | 11·1 | ... | ... | ... | ... | 11·8 | 17·5 | 62·0 | 76·0 |

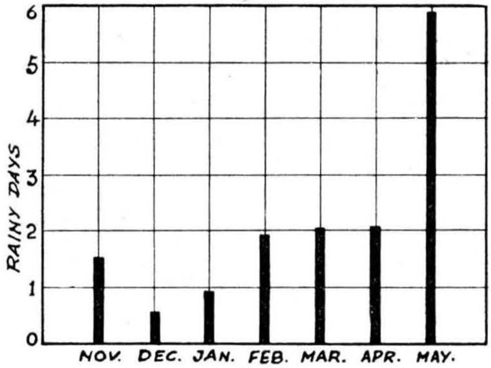

FIG. 2 — SAGAR ISLAND (WEST BENGAL): NUMBER OF RAINY DAYS; AVERAGE FOR 50 YRS. (1871-1920).

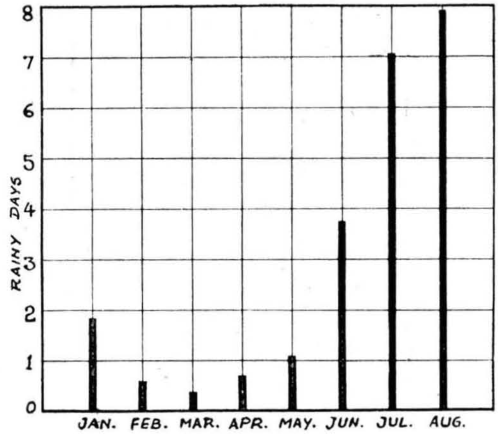

FIG. 3 — MADRAS: NUMBER OF RAINY DAYS; AVERAGE FOR 50 YRS. (1871-1920).

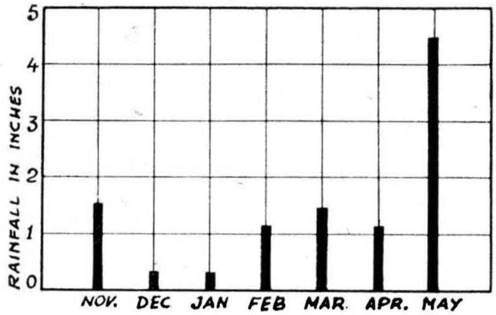

FIG. 4 — SAGAR ISLAND (WEST BENGAL): MEAN RAINFALL IN INCHES; AVERAGE FOR 50 YRS. (1871-1920).

rainy days is 23, and yet salt is manufactured there. Gopalpur, near which Naupada produces about 18 lakh md. of salt per year — being the second productive centre of Madras province — has a rainfall of 9″ and an average of 13 rainy days during the salt season due to the thrust of Norwesters. Contai is similarly subject to rain showers during the months of November to May. This is followed by 15 bright sunny days during the month of June, and if the damage done by the Norwesters be not irreparable, the salt operations are not hampered.

Balasore in northern Orissa has an average rainfall of 11·8″ distributed over 17·5 days during November to May. Balasore shore is only 50 miles down Contai and during the months when salt deposits in solar pans — February to May — the number of rainy days is 14.

It has often been said that the rainfall in Orissa and Bengal is a great impediment for the successful manufacture of salt. This is incorrect. It should be remembered that Madras with frequent showers has successfully developed her salt industry and is now the prime producer of salt in the Union. The salt season in Bengal and Orissa will be shorter than that on the North Coromandal coast, but the prospects for reviving the salt industry in these provinces are indeed bright.

(iv) *Temperature* — The diurnal temperature, greatly influencing the relative humidity and the vapour pressure of the air, is an important factor in considering the amount of evaporation taking place in the solar pans of a salt factory. The temperature of the atmosphere at a given place depends on: (a) latitude; (b) elevation; (c) distance from the sea; (d) character of the wind; and (e) the amount of rainfall. During December to June the temperature of the air in India generally decreases with the increase of latitude and hence, from south to north, the isotherms run across India nearly parallel to the latitude. The climate is moderately dry. During the less arid months, the lands away from the sea are hotter than the coastal tracts, causing a dry land breeze to blow towards the sea during the day which is then longer than the night, thus accelerating the evaporation of brine in the salterns. The average temperatures prevailing in the coastal part of West Bengal and Orissa are shown in Table IV.

**TABLE IV — AVERAGE TEMPERATURES ( °F. ) ( 1940-44 )**

| | SAGAR ISLAND | | | BALASORE ( ORISSA ) | | |
|---|---|---|---|---|---|---|
| | Max. | Min. | Mean | Max. | Min. | Mean |
| Dec. | 85 | 56 | 70 | 80 | 56 | 68 |
| Jan. | 81 | 53 | 67 | 81 | 56 | 69 |
| Feb. | 85 | 56 | 71 | 85 | 62 | 73 |
| Mar. | 91 | 66 | 78 | 93 | 69 | 81 |
| Apr. | 92 | 69 | 81 | 97 | 76 | 87 |
| May | 95 | 71 | 83 | 97 | 79 | 88 |
| June | 96 | 75 | 85 | 93 | 79 | 86 |
| Oct. | 89 | 72 | ... | 88 | 73 | ... |
| Nov. | 87 | 63 | ... | 84 | 63 | ... |

As is evident from the figures, the coastland of Bengal in the western part has a mean temperature of about 81°F. ( normal diurnal 85°F. ) during the months salt is manufactured, and the average mean temperature in Orissa coast is about 79°F. ( normal diurnal about 85°F. ). Such temperatures with a humidity of 70 per cent are favourable for raising salt from brine.

The maximum temperature of Bombay during January to May varies from 84°F. to 91°F. and that of Madras from 83°F. to 97°F. From the table above it is seen that the maximum temperature of Sagar in West Bengal and Balasore in northern coast of Orissa varies from 80°F. to 97°F.

(v) *Wind Velocities* — Both velocity and dryness of the winds during the salt season affect the evaporation of brine.

It may be stated here that when the wind is dry, and is blowing from the land, its velocity is not relatively important. Likewise, provided the breeze from the sea or marsh land is moderate, however moist it be, it does not materially retard the progress of brine evaporation, especially during the day time when the temperature is high and the air has a high saturation capacity. If the humidity is below 75 per cent during the forenoon, the air can still take up moisture. Sea breeze disturbs the surface of brine in the pans and accelerates evaporation.

**TABLE V — AVERAGE WIND VELOCITIES IN MILES PER HOUR ( 1940-45 )**

| | BOMBAY | BALASORE | SAGAR ISLAND ( West Bengal ) | MADRAS |
|---|---|---|---|---|
| Jan. | 5·4 | 1·8 | 6·6 | 9·5 |
| Feb. | 5·7 | 2·6 | 9·0 | 7·0 |
| Mar. | 6·2 | 3·9 | 13·5 | 8·2 |
| Apr. | 6·3 | 5·4 | 15·7 | 8·9 |
| May | 5·6 | 5·7 | 19·7 | 10·2 |
| June | 6·8 | 4·8 | 15·9 | 11·0 |
| Nov. | ... | 1·6 | 6·4 | ... |
| Dec. | 4·5 | 1·6 | 7·0 | ... |

The average wind velocities at Sagar and Balasore differ from those of Bombay and Madras ( TABLE V ). Bombay has a more or less steady wind velocity during the salt season, which is lower than that of Madras. In Sagar Island the wind velocity is higher than that of Madras. The wind velocity in Balasore is, however, poor. Throughout it is very much less than that in Bombay.

## Conclusion

From an examination of the climatic factor and the salinity of brine obtainable along the sea coast of West Bengal and Orissa it may be concluded that the littoral is not unsuitable to salt manufacture. This conclusion is supported by the fact that a flourishing salt industry had been established in these two provinces before the days of East India Company. The question arises whether climate and salinity factors are favourable for salt culture by solar evaporation. The span of the salt season is almost equal to those of Bombay or northern Madras. The only factor for consideration is the rain and storm due to Norwesters. Naupada is equally subject to rain and storm during April and May. It would appear that salt can be raised in West Bengal and Orissa as in Naupada ; if it cannot be done entirely by solar evaporation, it should be possible to manufacture salt by combining solar evaporation ( 70 per cent ) with boiling by heating ( 30 per cent ).

The old salt industry in these regions was dependent both on solar and artificial heat, and the process in vogue was different from that now used in southern Orissa and Madras. The people of the coastal regions used salt earth instead of salt water to get concentrated brine, which they boiled to recover salt. This method of leaching the salt earth and boiling the leachate is current on a cottage scale, having been revived after the lifting of the restriction on salt making in 1930. The people in the southern coastal area of Orissa have adopted the Madras practice of raising salt from brine by solar evaporation.

For the commercial manufacture of salt along the coast-land of West Bengal and Orissa, the method as followed in other places of India is recommended. Successful results have been achieved in a few of the West Bengal salt factories which have introduced this method. It is, however, advisable to provide for boiling if the level of production has to be raised.

The sea-board of Contai in the district of Midnapore has been surveyed by the author on behalf of the Provincial Government. It affords vast tracts of waste land, flooded by tidal flows of the sea, highly suitable for salt manufacture. From the land records of the Government, these tracts cover 9,000 acres of land and creeks of which only 150 acres have been developed to salterns by private limited companies. The sea water can be impounded during high tides and utilized, but irrigation pumps will be necessary towards the end of the manufacturing season.

If this area is fully developed, more than 40 lakh md. of salt, the present annual requirement of West Bengal, can be produced. This level of production can be maintained, if provision is made as stand by for boiling during adverse weather conditions.

In the combined process of solar evaporation and boiling, as practised in Burma, the cost of production is a little higher than that in the solar process, but the quality of salt is better and the rate of production can be maintained. The cost of salt production in Contai factories is estimated to be little higher, but as the markets are near, there will be considerable saving on transport.

As to the availability of fuel ( coal ) for boiling, Calcutta will be the source of supply, and the vessel which would tranship salt from Contai to the port of Calcutta will carry coal on their return voyage. The quantity of steam coal required is about 25 sr. per md. of salt, and the transport of coal to the Contai salt sources will not be uneconomical.

Two firms have been established at Contai for manufacturing salt ( combined annual output of 30,000 md.) by solar evaporation. The bigger of these two firms has furnaces to boil saturated saline when necessary. The following data, collected from the factories, will prove useful for developing the areas surveyed by the author.

Average densities of brine on the Contai sea-board are as follows :

TABLE VI — AVERAGE BRINE DENSITY ON CONTAI SEA-BOARD

| | °Be | | °Be |
|---|---|---|---|
| Nov. | 1·0 | Mar. | 2·6 |
| Dec. | 1·5 | Apr. | 2·7 |
| Jan. | 1·8 | May | 2·8 |
| Feb. | 2·5 | June ( up to 15th ) | 2·7 |

TABLE VII — APPROXIMATE RATE OF EVAPORATION PER DIEM

| | °Be | INCHES |
|---|---|---|
| Pond I | 2·5-4 | 0·20 |
| ,, II | 4-8 | 0·18 |
| ,, III | 8-12 | 0·17 |
| ,, IV | 12-18 | 0·16 |
| ,, V | 18-23 | 0·15 |

Besides the lands already surveyed on the Contai sea-board, there are suitable lands in the 24 Parganas ( Sunderbans ). The estuaries to the west of the rivers Hugli, Saptamukhi, Thakuran and Matla are salty during the dry season, and on both sides of each of these estuaries there are lands suitable for raising of salt. In the Sunderbans area about 5,000 acres of waste land will be available, from which 15 to 20 lakh md. of salt can be anticipated. The transport by water will be easy and the prospects for raising salt in this region are very bright.

## BIOS FINAL REPORT No. 1363 *

# German Dyestuff Industry

K. VENKATARAMAN

(*Continued from April issue*)

### APPENDIX 10

### BLAUTHIOSAURE

*Principal Use* — For DB-Naphthen.

*Equipment* — Homogeneous leaded stirring vessel, 4·5 cu. m. with a cap and descending leaden cooler (reducing vessel), and iron stirring vessel, 6 cu. m. (distilling receiver). Iron stirring vessel, content 7 cu. m., with a coil (condensation vessel). Homogeneous leaded and brick-lined stirring vessel with a coil, content 7 cu. m.; 1 iron press, size 600×900 mm. and 2 wooden presses, size 800×800 mm.

*Process: Reduction to Mercaptan* — Into the reducing vessel are introduced 600 l. of water, 230 kg. of zinc dust and then 225 kg. of " Blausulphochlorid ". The temperature is maintained at 20°C. so that no reaction sets in yet. While still cooling, 1,000 kg. of sulphuric acid (73 per cent) are run in. Only when the last third of sulphuric acid runs in, the cooling is stopped, so that the reduction begins. Now, the temperature is raised to 95°C. by heating the vessel. For 3 hr. this temperature is kept. Then cooled to 50°C. and 130 kg. of iron powder introduced; the mercaptan is distilled off by direct steam. Time required: about 15 hr.

*Condensation* — The mercaptan and the water from distillation are pressed out of the receiver into the condensation vessel. Here it is dissolved with 380 kg. of caustic soda solution (33 per cent) and while adding about 300 kg. of ice, it is condensed at 0°-5°C. with 125 kg. of chloracetic acid. Then it is heated to 95°C. and after adding 10 kg. of clearing carbon it is filtered into the brick-lined stirring vessel by passing through the iron press. The residue contained in the press is then washed out with about 1 cu. m. of water at 70°C. from the condensation vessel. Cooled down to 15°C. and 440 kg. of hydrochloric acid added whereby the " Blauthiosaure " is precipitated. After stirring for 10 hr. the acid is filtered on the 2 wooden presses, washed with about 1 cu. m. of water and dried at 80°C.

*Process Control Tests* — (1) After finishing the distillation, a sample from the reducing vessel is again distilled with steam and the distillate mixed with lead acetate. No yellow colouration is allowed (yellow lead salt of mercaptan).

(2) After finishing the condensation, a spot test, on lead acetate paper, shows the presence of non-condensed mercaptan by yellow colouration.

(3) Determination of dry content (99·8 per cent), determination of the melting point and ash.

*Raw Materials, Intermediates & Finished Product Specifications* — The " Blausulphochlorid " has to be further worked up immediately after manufacturing. The finished product has to be clearly soluble in caustic soda solution; m.p., 120°-130°C. White crystals.

### APPENDIX 19

### *p*'-METHOXY-PHENYL-PARAMINIC ACID

*Principal Use* — For the manufacture of Blue-base F.

*Equipment* — (1) 1 *montejus* for pasting up " *p*-nitrochlorsaure ", content 7·5 cu. m.; (2) 1 brick-lined condensation vessel with direct lead of high pressure steam, about 6 cu. m. useful content and connected with a small separator with Raschig rings and water spraying; (3) 1 brick-lined condensation vessel, about 9 cu. m. useful content with direct lead of high pressure steam; (4) 1 brick-lined reducer, 10-11 cu. m. useful content, with direct steam connection; (5) 1 brick-lined precipitating vessel for *p*'-methoxy-phenyl-paraminic acid, 12 cu. m., and a pump for conveying to the suction filter; (6) 1 brick-lined receiving *montejus* for the condensation mixture, about 8 cu. m.; (7) 2 rubber-lined suction filter receivers for the precipitation of the final liquors, 9 cu. m. each; (8) 1 brick-lined suction filter for the

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*This reference was inadvertently omitted from page 130 of the April issue.

finished product ; (9) 1 rinsable " Monstre " press ( for iron sludge ), 32 frames, 1,200 × 1,200 mm. ; (10) 1 " Monstre " press for the precipitate from the mother liquor, 35 frames, 1,200 × 1,200 mm.

*Process* — 772 kg. of " *p*-nitrochlorsaure " ( 100 per cent, mol. 327·5 ), as moist neutral product of 40-50 per cent, are pasted up in a *montejus* with water to a paste of 3·3 cu. m. and 85 kg. of magnesium oxide are added while stirring ( 84 per cent = about 71 kg. of MgO, 100 per cent ) ; this paste is pressed into the brick-lined condensation vessel. To this are added from a pressure vessel 400 kg. of *p*-anisidine in molten condition. The mixture is heated to 160°C. by high pressure steam at 10 atm. pressure and kept at a temperature of 160°-165°C. for 3 hr. The pressure in the condensation vessel is 6 atm. Then the pressure is let off to a separator, filled with Raschig rings and sprayed with water, where parts of non-condensed *p*-anisidine are collected. When the temperature has fallen to 100°-105°C., the contents are pressed off to a brick-lined stirring *montejus* ; the main quantity of the magnesium salt of 4′-methoxy-4-nitrodiphenylamine-2-sulphonic acid is already deposited in the hot liquor. The surplus magnesia is neutralized by adding about 100 kg. of sulphuric acid ( 30°Bé.). The hot condensation mixture is now, in portions, pressed into the brick-lined reducer containing 450-480 kg. of sifted iron filings, with water and a small quantity of sulphuric acid, 30°Bé. The reduction mixture is constantly kept boiling. The reduction of the total nitro-acid is finished when the drop test on filter paper shows no more a yellow, but a purple border. Further, the end of the reduction is tested with Mohr's salt. The reduction finished, the mixture is made slightly alkaline by addition of 85 kg. of soda and filtered boiling hot from the iron sludge through a rinsable " Monstre " press. The iron sludge is washed out with boiling water. The reduction liquid, combined with the washing water, 8·5-9 cu. m., containing about 11 volumes per cent of material, is conducted to a brick-lined precipitating vessel, 2 kg. of zinc dust and 2 kg. of hydrosulphite are stirred in. After the addition of 1,300-1,500 kg. of salt, the liquid is cooled to 20°C. The sodium salt of *p*′-methoxy-phenyl-paraminic acid deposits in well-formed crystals. A sample of the mother liquor shows a sp. gr. of 22°-22·5°Bé. The product is filtered on a suction filter and sucked dry. From the mother liquor, the free *p*′-methoxy-phenyl-paraminic acid is precipitated in the rubber-lined suction filter receivers by addition of 80-90 kg. of sulphuric acid at 60°Bé. and pressed off in the " Monstre " press. This acid is added to the next reduction. The sodium salt of *p*′-methoxy-phenyl-paraminic acid is, as a rule, 64-68 per cent ; mol. wt., 294, and contains 4-5 per cent of sodium chloride. It contains 1 mol. of water of crystallization.

*Yield* — 870-880 kg. of *p*′-methoxy-phenyl-paraminic acid ( 100 per cent, mol. wt., 294 ; 91-92 per cent of the theoretical ).

*Process Control Tests* — Test for the end of the reduction by the colour of the drop border and with Mohr's salt. Determination of the sp. gr. of the mother liquor after the precipitation of 4′-methoxy-phenyl-paraminic acid. Determination of the percentage of *p*′-methoxy-phenyl-paraminic acid by titration with normal nitrite solution.

*Raw Materials, Intermediates & Finished Product Specifications* — *p*-Anisidine : melting point, 57°C. ; magnesia should be 83-85 per cent and as far as possible free from carbonate ; " *p*-nitrochlorsaure " must be neutral. Iron filings should be free from coarse elements. *p*′-Methoxy-phenyl-paraminic acid is a grey-violet, finely crystalline product, containing 1 mol. of water of crystallization. Average composition : 65 per cent of *p*′-methoxy-phenyl-paraminic acid, mol. wt. 294 or 69·9 per cent of the sodium salt, mol. wt. 396, 24-25 per cent of water ( including water of crystallization ) and 4-5 per cent of sodium chloride.

## APPENDIX 22

### " AMIDOL " ACID

*Principal Use* — For producing dyestuffs.

*Equipment* — (1) 1 dissolving vessel for β-naphthol, 4·5 cu. m. ; (2) 2 vats for brine, nitrous acid treatment with cooling, 12 cu. m. ; (3) 2 leaded transport vessels, 7·5 cu. m. ; (4) 3 acid-proof brick-lined rearrangement vessels, 15 cu. m. useful capacity, with twirling stirrers and well working hood ; (5) 1 acid-proof brick-lined suction filter with a corresponding rubber-lined receiver ; (6) stock and measuring vessel for nitrite solution ( 40 per cent ), stock and measuring vessels for sulphuric acid ( 30 per cent and 60 per cent ), stock and measuring vessel for bisulphite solution ( 38-40 per cent ), stock vessel and vat for weighing caustic soda solution ( 33·5 per cent ) ; and (7) various receivers for sulphureous acid absorption.

*Process* — In a *montejus* 576 kg. of β-naphthol scales are dissolved at about 33°C. in 3,000 l. of water and 478 kg. of caustic soda solution. The solution is made up to 4,500 l. and pressed into the vat for treatment with nitrous acid. Here the β-naphthol solution is cooled with brine to 3°C. The β-naphthol is precipitated in a finely divided form by adding about 560 kg. of iron-free sulphuric acid (30 per cent strength) through a distributing nozzle. The liquid must have a slightly alkaline reaction to phenolphthalein. In order to obtain, as far as possible, a fine and homogeneous distribution of the β-naphthol, the mixture is stirred for some time, and then 690 kg. of nitrite solution of 40 per cent strength are added. Now, at 5°-8°C., further 680 kg. of iron-free sulphuric acid of 30 per cent strength are slowly run in through the distributing nozzle. The liquid must now have a distinctly acid reaction to Congo paper and must indicate nitrite. The nitroso-β-naphthol precipitates as a slightly yellow, finely divided product. After additional stirring at not more than 8°C. for several hours, the excess acid is neutralized by adding 30-40 kg. of caustic soda solution. The liquid must have a neutral reaction. 2,400 kg. of sodium bisulphite solution are run into this paste of nitroso-β-naphthol. The temperature rises to 18°-20°C. and a solution is obtained showing a few dark, flocky, separated particles. After adding 500 kg. of rock salt, the solution is pressed into the rearrangement vessel containing 5 cu. m. of the mother liquor from the preceding batch with a content of 5·5-6 per cent by volume of sulphuric acid. The mixture (about 13 cu. m.) is acidified with 700 kg. of sulphuric acid of 60 per cent strength and heated to 38°C. by direct steam. The temperature slowly rises to 50°-52°C. The "Amidol" acid begins to separate as a slightly grey crystalline precipitate. The precipitation is completed by allowing to stand for about 36-40 hr. and by occasional stirring to avoid complete solidification of the mass. Then the mass is thoroughly stirred and the "Amidol" acid is discharged on an acid-proof suction filter and sucked off. The product is washed free from acid with about 4-5 cu. m. of water and the neutral product of about 25 per cent strength, mol. wt., 239, is worked to "Diazoamidol" acid. For dispatch the product is filtered by suction as dry as possible (32-36 per cent).

*Yield*—765-770 kg. of "Amidol" acid of 100 per cent strength, 80-81 per cent of the theory.

*Process Control Tests* — Test as to complete dissolution of β-naphthol in caustic soda solution. Test of the nitrous acid treatment as to an acid reaction of the liquid and as to an excess of nitrite solution. Test of the "Amidol" acid as to its neutral reaction. Determination of the content of the moist "Amidol" acid by diazotizing in neutral solution with copper sulphate as catalyst.

*Raw Materials, Intermediates & Finished Product Specifications* — β-naphthol (scales) must be soluble, as clear as possible, in caustic soda solution; m.p. 120°C., tech. product of 100 per cent strength.

Nitrite solution of 40 per cent strength.

Bisulphite solution of 38-40 per cent strength.

Sulphuric acid of 30 per cent strength; must be as free from iron as possible.

Caustic soda solution of 33·5 per cent strength, commercial product.

Sulphuric acid of 60 per cent strength, commercial product.

Rock salt, commercial product.

The moist "Amidol" acid is a slightly grey, crystalline product in paste form of about 25 per cent strength.

The product, well sucked off for dispatch, is 32-36 per cent.

## APPENDIX 23

### DIAZOAMIDOL-ACID

*Principal Use* — For dyestuffs.

*Equipment* — (1) 2 pasting vessels for the amidol-acid, 7·5 cu. m., one of them rubber-lined; (2) 1 diazotizing vat with high-speed stirrer and brine cooling; (3) 2 leaded receivers for the diazo solution, 7·5 cu. m.; (4) 2 small washable clarifying presses, each containing 18 chambers, 80 × 80 cm.; (5) 2 acid-proof, brick-lined precipitating vessels with twirl-stirrer, 10·5 cu. m.; (6) 2 homogeneously leaded pendulum centrifuges; (7) 1 acid-proof suction filter with corresponding receiver.

*Process* — 700 kg. of amidol acid (100 per cent; mol. wt., 239) as a moist neutral paste of about 25 per cent are stirred with water so as to obtain about 3,000 l. of a 21-21·5 per cent paste. The diazotizing vessel, fitted with high-speed stirrer, contains 1,000-1,200 l. of water of the washing liquor (which is obtained by washing out the residue of the preceding operation), 2·5 kg. of crystallized copper sulphate dissolved in some litres of water and 540 kg. of nitrite solution

( 40 per cent ) respectively. To this the 3,000 l. of amidol acid paste are pressed in the course of about 3 hr. Within this time the slowly rising temperature must not exceed 25°C. The amidol acid paste totally dissolves — while forming the diazo salt — to a dark, golden-yellow solution. Care is taken that there always exists an excess of nitrite. Then the solution is rendered slightly acid to litmus by adding sulphuric acid ( 30 per cent ) rendering the dark flocculent residue easily filterable. The solution is filtered through a clarifying press and the residue washed out with warm water at 40°-45°C. The first portion of the washing water ( about 500 l. ) is combined with the clear filtrate. The bulk ( about 1,000 l. ) is used again in the following diazotization: the filtrate combined with the first washing water is acidified by slowly adding 1,350 kg. of sulphuric acid ( 30 per cent ) within about 8 hr. at 25°-28°C. The diazotized amidol acid separates as well-crystallized, greyish-orange product. Separation is finished as soon as the mother liquor indicates 5·5-6 per cent by volume of sulphuric acid. The separated diazoamidol acid is centrifuged from the mother liquor on the homogeneously leaded pendulum centrifuges and obtained as a sandy, greyish-orange product with a content of 86-88 per cent ( mol. wt., 250 ). For avoiding losses the mother liquor passes from the centrifuge to a suction filter. For working up to chlorodiazoamidol acid the product must be dried below 50°C. when a brownish dusting product ( 92-93 per cent ; mol. wt., 250 ) is obtained.

*Yield* — 688-695 kg. of moist diazoamidol acid calculated as a product of 100 per cent ( 94-95 per cent of the theory ).

*Process Control Tests* —

Test of the amidol acid paste as to neutrality.

Test of the diazotizing process as to presence of nitrite solution.

Test as to the total separation of the diazoamidol acid by determination of the acidity of the mother liquor.

Testing the content of the moist and of the dry finished product by determination of the diazo nitrogen evolved on decomposition with cuprous chloride solution.

*Raw Materials, Intermediates & Finished Product Specifications* —

Nitrite solution of 40 per cent strength.

Sulphuric acid of 30 per cent strength.

Crystallized copper sulphate : commercial quality.

Acidity of the mother liquor after separation : 5·5-5·6 per cent by vol.

The product is clearly soluble in caustic soda solution.

The diazoamidol acid contains 1 mol. of water of crystallization.

## APPENDIX 25

## NITRO-ETHYLIMIDE BASE

*Principal Use* — After reduction to aminoethylimide base for Sirius Light Blue FFRL.

*Equipment* — (1) boiler, 1 cu. m., cast iron vessel with jacket of silica stones, stirrer and lid coated with $V_2A$ steel ; (2) 1 pressure suction filter, 1·5 sq. m., brick-lined, with receiver ; and (3) 1 stone suction filter with nozzle, 2·5 sq. m.

*Process* — Introduce 200 kg. of ethylimide base ( N-ethyl carbazole ) at ordinary temperature into 180 kg. of chlorobenzene and dissolve by stirring for 1 hr. Temperature 20°-25°C. 305 kg. of nitric acid ( 35·5 per cent ) are run in within about 5 hr. Temperature 27°-28°C. ( cooled by water or brine ). After-stir overnight at a temperature between 25°-30°C. ( a sample should have a melting point of 127°-130°C.) ( Test a ). Cool to 10°C. and stir at 100°C. ; after 8-10 hr. filter on the pressure suction filter and wash thrice, each time with 10 kg. of chlorobenzene. Then bring the batch on the stone suction filter and wash there 2-3 times, each time with 250 kg. of Leonil SB-solution of 1 per cent strength ( Test b ). Finally, wash until neutral with 50 kg. of water. Dry in the circulating air chamber at a temperature of 50°-60°C.

*Process Control Tests : Test a*— Filter the sample by suction, wash it for a short while with chlorobenzene and then wash thrice until neutral with Leonil water of 2 per cent strength and finally with water. Dry on clay tile, 129°-130°C.

*Test b* — Distil 100 gm. of the material on the filter with steam in order to test whether the chlorobenzene has been completely washed out with Leonil and water.

*Raw Materials, Intermediates & Finished Product Specifications* — Ethylimide base ( N-ethyl carbazole ) : point of solidification, 64·5°-65°C., clearly soluble in chlorobenzene. The nitro-ethylimide base is beautiful yellow crystals., m.p., 129°-129°C. The product must be completely neutral to Congo paper.

## APPENDIX 26

### CARBAZANIL

*Principal Use* — Starting material for carboxazine, for Sirius Supra Blue F3RL.

*Equipment* — (1) Brick-lined stirring vessel, 800 l., with steam jacket and leaded reflux condenser ; and (2) filter press, 800 × 800 mm.

*Process* — 8·5 kg. of chloranil are introduced into a suspension of 125 kg. of spirit of 50 per cent by volume, 12 kg. of aminocarbazole and 6 kg. of anhydrous sodium acetate within ½ hr. at room temperature. The mixture is heated to boiling and kept at boiling temperature for 6 hr. while stirring and refluxing. After cooling down to 25°-30°C., the product is filtered off and washed with 25 kg. of spirit of 25 per cent by volume. Then it is freed from ash by washing at first for 2 hr. with hot water and finally for ½ hr. with cold water. The pressed cake is blown with nitrogen until dry and dried in the vacuum-drying closet at 65°-70°C. The spirit solutions are regenerated.

## APPENDIX 27

### " TETRA ACID "

*Principal Use* — Starting product for producing Indanthren Printing Brown B and 5R and Indanthren Scarlet GG.

*Equipment*— Brick-lined ( 2 layers ) iron vessel, 16 cu. m. with wooden stirrer and leaded cover ; open, brick-lined suction filter with stone filter, 4 sq. m. ; cemented iron vessel, 5 cu. m., with stirrer ; clarifying filter ; iron vessel, 6 cu. m. ( clarifying receiver ) ; 2 iron filter presses, 800 × 800 mm. each ; rubber-lined iron vessel, 16 cu. m., with stirrer.

*Process* — In the brick-lined vessel containing a moist paste of " diketimide " ( 250 kg. of dry product ), is added a mixture of 2 cu. m. of water and 2,217 kg. of concentrated hydrochloric acid. The mixture is heated to 40°C. by blowing in steam, and 145 kg. of sodium chlorate are carried in within 2 hr. The temperature rises to 60°C. After stirring for 8 hr., the mixture is made up to 10 cu. m. by addition of cold water ; the product is sucked off on the suction filter and washed to neutral reaction.

In the cemented iron vessel with stirrer, 2,130 l. of water, 385 kg. of caustic soda solution ( 22 per cent ) and 650 kg. of sodium hypochlorite liquor are heated to 40°C. and one-third of the suction filter material is carried in. The temperature rises to 50°C. and is maintained for 2 hr. The mixture is neutralized by addition of 50 kg. of bisulphite solution, clarified through a clarifying filter into the iron vessel (6 cu. m.) and washed with 4 cu. m. of hot water. This water is again used for the following batch.

The clarified liquor is carried back into the oxidation vessel, 60 kg. of caustic soda solution ( 22 per cent ) and 575 kg. of sodium hypochlorite are added and the mixture is oxidized at 30°-40°C. by introducing about 120 kg. of potassium permanganate, added shovel by shovel. The temperature rises to 70°C. After 20 min., the excess of potassium permanganate is destroyed by addition of 50 kg. of bisulphite and the manganese mud is filtered in 2 filter presses. The filtrate runs into the rubber-lined vessel. The manganese mud is washed hot, pasted again, filtered and washed until tetra acid can no longer be detected in the filtrate. In the filtrate the tetra acid is precipitated by addition of 1,000 kg. of crude hydrochloric acid, sucked off on a suction filter and washed with 800 l. of water and 20 l. of hydrochloric acid ( 1 per cent ). The tetra acid is dried at 80°-90°C.

*Process Control Tests*—(a) After the reaction with sodium chlorate, a sample is sucked off, washed first with cold water to neutral reaction and then with hot water. In this filtrate no " diketimide " must be precipitated by addition of salt. The fluorescence effected by exposing to the rays of a quartz-lamp must only be weak-bluish, compared with the test substance. When " diketimide " is present, 10-20 kg. of sodium chlorate and 300-500 kg. of hydrochloric acid are added.

(b) After the oxidation by potassium permanganate, a sample spread on filter paper must have a green bleeding which should not disappear after stirring for 20 min. In an acidified sample of the filtrate the tetra acid has to precipitate in well-formed crystals.

(c) After the precipitation of the tetra acid, the filtrate must still turn Congo paper blue.

(d) When further acid is added to a sample of the filtrate, no tetra acid must be precipitated.

(e) When a sample of the manganese mud is extracted with hot dilute caustic soda solution, the acidified extract must not show any precipitation.

*Raw Materials, Intermediates & Finished Product Specifications* — The finished product is condensed with orthamine and glacial acetic acid to Indanthren Scarlet GG and the latter is tested as to its dyeing properties. The yield should amount to 136 per cent of the used tetra acid. Tetra acid should be tested for organically bound chlorine, iron and ash content.

## APPENDIX 29

### PYRENIT S

*Principal Uses* — Starting material for Pyram, intermediate for Siriuslichtblau F3GL.

*Equipment* — Clay pot with $V_2A$ steel high-speed stirrer, 700 l. ; brick-lined suction filter ; brick-lined receiver with connection to the vacuum piping ; and swinging mill.

*Process* — In the clay pot, 100 kg. of pyrene ( ground on the Colloplex mill and afterwards on the swinging mill ) is stirred 405 kg. of nitric acid of 22 per cent strength for 8 hr. at 40°-45°C. ( test ), then sucked off, washed until neutral and dried. The mother liquor is stored and used again.

*Process Control Test* — The m.p. must be 148°-150°C.

*Raw Materials, Intermediates & Finished Product Specifications* —

Pyrene, free from iron.

Finished product : yellow-brown powder, m.p., 148°-150°C.

## APPENDIX 30

### PYREN-CHLORID

*Principal Use* — Starting material for pyrenequinone.

*Equipment* — Enamelled cast-iron stirring vessel, 2 cu. m., in a diphenyl bath, with coil for steam of 15 atm. ( reaction vessel ) ; brick-lined iron suction filter, 2·5 sq. m., with brick-lined receiver ; brick-lined iron stirring vessel, 4·5 cu. m., with iron cooler and iron separator, 6 cu. m. ; brick-lined iron suction filter, 3 sq. m.

*Process* — In the reaction vessel, 1,350 kg. of trichlorbenzene are heated to 180°C. While stirring, one-third the tetrachlorpyrene tetrachloride ( corresponding to 180 kg. ) and 36 gm. of ferric chloride are introduced, at first in small portions, until splitting off of hydrochloric acid occurs. The reaction finished, the product is sucked off on the suction filter at 30°C. and washed with 400 kg. of trichlorbenzene. The mother liquor is used for the 2 following batches. The material from the suction filter is freed from the solvent in the distiller, sucked off on the second suction filter, washed until neutral and dried at 60°-70°C. The remaining two-thirds of the tetrachloride is treated in the same way. The yield of 3 batches amounts to 400 kg. The trichlorbenzene is recovered in a special equipment. The loss of solvent amounts to 20 per cent.

*Process Control Tests* — Final point of the splitting off ; termination of the evolution of hydrogen chloride.

*Raw Materials, Intermediates & Finished Product Specifications* — Hexachlor-pyrene is a yellow, crystalline product, m.p., 360°-375°C. The pure product melts at 382°C.

## APPENDIX 31

### PERCHLORPYRENE

*Principal Use* — Starting material for hexachlor-pyrene.

*Equipment* — Brick-lined, homogeneously leaded stirring vessel, 3 cu. m., with leaded heating candle ( reaction vessel ) ; brick-lined iron suction filter, 2·5 sq. m., with brick-lined receiver ; enamelled, cast-iron stirring vessel, 2 cu. m., in a diphenyl bath with coil for steam of 15 atm. ( pasting up vessel ).

*Process* — Into the reaction vessel containing 1,800 kg. of trichlorbenzene, 120 kg. of pyrene and 120 gm. of iodine, 500 kg. of chlorine are passed in at 108°-110°C. within 24 hr. After cooling to 90°C., a further quantity of 120 kg. of pyrene and 120 gm. of iodine are introduced and the chlorination is continued at 108°-110°C. ( 12-15 kg. of chlorine per hour ). In all, 480 kg. of pyrene are chlorinated, the chlorine stream finally amounting to 8 kg. per hour. The hydrogen chloride gases together with the excess of chlorine go through separators to the chimney. Duration of the chlorination : 6 days. The product is sucked off at 30°C. on the suction filter and washed with 400 kg. of trichlorbenzene from the reaction vessel. The material from the suction filter is stirred in the pasting-up vessel with 63 kg. of trichlorbenzene, sucked off on the same suction filter and washed with 200 kg. of trichlorbenzene and processed to hexachlor pyrene.

The filtrates and washing liquors of trichlorbenzene are worked up in a special equipment. Yield : 540 kg.

*Process Control Tests* — Final point of the chlorination : 50 c.c. of the suspension are filtered and the white residue on the filter is washed with carbon tetrachloride ; the product shall be completely soluble in 10 times its quantity of chlorobenzene. Tetrachlorpyrene is insoluble. In 10 times its quantity of nitric acid ( sp. gr. 1·4 ), no change of colouration should occur. Traces of tetrachlorpyrene give a brown colouration with nitric acid.

### APPENDIX 32

### " PYRANIL ", CRUDE

*Principal Uses* — Starting material for " Leucopyranil " ; intermediate product for Sirius Light Blue F3GL.

*Equipment* — A 3 cu. m. enamelled boiler with steam jacket, stirrer ; spirit mother liquor vessel ; spirit regenerating apparatus ; and a " Monstre " press, 1,200 × 1,200 mm.

*Process* — In the 3 cu. m. reaction vessel, 145 kg. of " Pyram ", 102 kg. of chlorophenol, 104 kg. of chloranil and 116 kg. of anhydrous sodium acetate are condensed in 1,200 kg. of spirit ( 95 per cent ) at first at 0°C. ( cooling with 200 kg. of solid carbonic acid ), then at 20°-25°C. for 12 hr. Then filtered in the "Monstre" press. Finally, the filtered product is washed with water until neutral and free from spirit. The moist product is dried at 90°-100°C. in a vacuum drying closet. The spirit mother liquor and the washing waters are collected and distilled.

*Raw Materials, Intermediates & Finished Product Specifications* —

(1) Aminopyrene ( " Pyram " ) : m.p., 114°-116°C.

(2) Chloranil : not below 99 per cent.

(3) *o*-Chlorophenol : about 90-97 per cent of *o*-chlorophenol.

(4) Sodium acetate, anhydrous.

(5) Spirit not below 90 per cent by weight.

### APPENDIX 33

### " PYRANIL " PURE

*Principal Uses* — Starting material for " Pyroxazine " ; intermediate product for Sirius Light Blue F3GL.

*Equipment* — A 2 cu. m. enamelled vessel with oil circulation heating ; 7·5 cu. m. expeller, cooler ; recovering equipment for nitrobenzene ; and a suction filter.

*Process* — In the enamelled vessel, containing 1,390 kg. of nitrobenzene, 110 kg. of " Leucopyranil " are quickly introduced at 190°C. and after-stirred for 45 min. Then the reaction mixture is pressed into the expeller with 3,000 l. of water and the nitrobenzene is driven off by steam. This product is then sucked off on a suction filter and washed free from ashes by means of hot water.

*Process Control Tests* — After the reaction the microscopic structure shall show fine, hair-shaped, brown crystals and no longer colourless crystals of " Leucopyranil ".

*Final Test* — Dry weight and ash determination. Reduction to the " Leucopyranil " according to prescription. The yield in per cent of the amount used gives the degree of purity for " Pyranil " pure.

*Raw Materials, Intermediates & Finished Product Specifications* — " Leucopyranil " must be absolutely dry.

Finished product : fine, hair-shaped, crystallized, brown product, m.p., 331°-332°C.

### APPENDIX 34

### PYRAM

*Principal Use* — Intermediate product for pyranil ( intermediate for Sirius Light Blue F3GL ).

*Equipment* — Iron stirring vessel with jacket, 4 cu. m., with reflux condenser, iron pressure filter with removable lid and cloth filter, 3 sq. m.

*Process* — In the 4·5 cu. m. iron stirring vessel 320 kg. of Pyrenit S are dissolved in 820 kg. of spirit ( at least 94 per cent ) by refluxing for 2 hr. 260 kg. of hydrosulphide ( with 96 kg. of $H_2S$ ) are dissolved in 130 l. of water in casks ; the solution is siphoned off from the contaminations and runs into the Pyrenit solution within 3 hr. By the violent reaction the batch is kept boiling. After running in is finished, refluxing is continued for further 3 hr., then 600 l. of water are filled in and the whole is cooled to 20°C. Pyram crystallizes within 15 hr., is filtered off on the pressure filter, washed with 240 kg. of spirit ( 50 per cent ) and afterwards with about 1,000 l. of water until neutral, and dried at 60°-70°C.

*Raw Materials, Intermediates & Finished Product Specifications* — Pyrenit : m.p., 144°-145°C. ( pure product 147°C. ).

Finished product : m.p., 116°-117°C.

### APPENDIX 35

### " LEUCOPYRANIL "

*Principal Uses* — Starting material for " Pyranil pure " ; intermediate product for Sirius Light Blue F3GL.

*Equipment* — A 2 cu. m. enamelled vessel, stirrer, circulation cooler ; pressure filter, leaded and brick-lined ; receiver, 4·5 cu. m., leaded ; and apparatus for the recovering of chlorobenzene.

*Process* — In the 2 cu. m. enamelled vessel 1,320 kg. of cholorobenzene and 135 kg. of crude " Pyranil " are heated to boiling. Then 30 kg. of phenylhydrazine run in in the course of 1 hr. ( test ). The reaction mixture is cooled to 70°C., pressed on the suction filter and after-washed twice with 180 kg. of 80°C. hot chlorobenzene. The product is steamed on the suction filter until free from chlorobenzene ; after cooling, dried in the drying closet at 70°C. The chlorobenzene mother liquors are worked up in the chlorobenzene recovery apparatus.

*Process Control Tests* — After the reaction a sample taken out of the vessel is filtered on a suction filter at 70°C. in the laboratory. The product shall look light-grey to light-yellow. Microscopic structure : colourless leaflets. In case brown needles of the crude " Pyranil " are still present, further phenylhydrazine must be added to the batch.

*Raw Materials, Intermediates & Finished Product Specifications* —

(1) " Pyranil ", crude : test in the laboratory.

(2) Phenylhydrazine, supplied freshly distilled, b.p., 233°C.

(3) Finished product : weakly, yellowish to grey crystalline product.

## APPENDIX 41

## INDANTHREN BRILLIANT ORANGE GR & INDANTHREN BORDO RR

*Principal Uses* — For printing and dyeing cotton and artificial fibre, Indanthren Bordo besides as mixed dyestuff.

*Equipment* — Iron stirring vessel with hose, 4·5 cu. m., brick-lined heatable pressure filter with stirrer that may be lifted and lowered, stone filter bottom, 3 sq. m., iron stirring vessel with hose, 2 cu. m., rubber-lined stirring vessel, 4·5 cu. m., horizontal iron vessel, 3 cu. m., iron stirring vessel with jacket, 4 cu. m., with iron condenser and iron receiver, 6 cu. m., brick-lined stirring vessel, 7 cu. m., 2 wooden presses 1,200 × 1,200 mm.

*Process* — In the iron stirring vessel 400 kg. of caustic potash powder are dissolved at 40°C. and 2,000 kg. of spirit and 200 kg. of Indanthren Scarlet GG base P are filled in at 75°C. and are held for 1 hr., then cooled to 20°C. The separated potassium addition product of the Indanthren Brilliant Orange is filtered on the suction pressure filter. In the 2 cu. m. iron stirring vessel the suction material is added to 420 kg. of spirit ( 83 per cent ) and 200 kg. of caustic potash lye, heated to 75°C., cooled to 20°C. and again filtered on the pressure filter. Washed in 3 portions with a washing spirit consisting of 480 kg. of spirit ( 95 per cent ), 75 kg. of water and 225 kg. of caustic potash lye, produced in the same iron stirring vessel of 2 cu. m. The suction material, Indanthren Brilliant Orange, is hydrolysed in 1-1½ hr. in the rubber-lined 4·5 cu. m. stirring vessel in 3 cu. m. of warm water at 60°C. and the obtained dyestuff is filtered hot into a wooden press and is washed neutral and filled into wooden barrels. The spirit filtrates are collected in the horizontal 3 cu. m. iron vessel and pressed off to the 4 cu. m. iron stirring vessel, in which the spirit is distilled off and collected in the 6 cu. m. iron spirit receiver. The residue, containing the Indanthren Bordó, is slowly pressed off ( foaming ) into the 7 cu. m. brick-lined stirring vessel in which 1,000 l. of hydrochloric acid crude and 1,000 l. of water of 80°C. are added. The reaction should finally be slightly acid. After stirring for another 1 hr., the dyestuff is filtered to the second wooden press, 1,200×1,200 mm., washed neutral and filled into wooden barrels.

*Process Control Tests* — (a) In the second suction material small red points indicate unconverted scarlet. (b) After acidifying the Bordo residue the reaction finally has to be acid to Congo.

*Raw Materials, Intermediates & Finished Product Specifications* —

(a) If difficulties occur, the " tetra acid ", already used for Indanthren scarlet, must be examined in a laboratory batch.

(b) The spirit is spindled.

(c) The Indanthren Brilliant Orange GR is examined as double paste in printing, the Indanthren Bordo RR as suprafix paste also in printing compared with standards. The yields are fixed by the weight of the filter cakes and by dry determination. The yield of Brilliant Orange should be 59 per cent. Indanthren Brilliant Orange GR double paste contains 10 per cent pure colour. Indanthren Bordo RR suprafix double paste contains 20 per cent pure colour.

## APPENDIX 42a

## INDANTHREN BROWN GR

*Principal Use* — Vat dyestuff for cotton.

*Equipment* — An iron melting vessel of 8 cu. m. provided with stirrer and high pressure water heating; an iron diluting vessel of 9 cu. m.; and a " Monstre " press.

*Process* — 1,800 kg. of caustic potash are introduced into the melting vessel heated to 140°-150°C. Then the trianthrimide is introduced into the vessel in the course of 4 hr. and heated to 190°-195°C. within 4-5 hr. While maintaining this temperature the whole is stirred for 8 hr. (Sample 1 to be taken). After cooling down to 150°-160°C., water is slowly added until the vessel is filled completely. The contents are allowed to run into the vessel of 9 cu. m. which is then filled up with water. The product is oxidized at 30°-40°C. by introducing air while stirring (Sample 2 to be taken). Then the whole is filtered into the " Monstre " press and washed with water until neutral.

*Process Control Tests : Sample* 1 — After the melt is finished, a test portion should be capable of being vatted completely without leaving any residue.

*Sample* 2 — Before filtering the melt, a test portion dropped on filter paper must show a bleeding coloured only weakly yellow.

*Final Sample* — An examination is made by producing a dyeing and comparing with the type.

*Raw Materials, Intermediates & Finished Product Specifications* — Final product: brown paste, its vat being reddish brown.

## APPENDIX 43

### INDANTHREN TURQUOISE BLUE GK

*Principal Use* — Dyestuff for dyeing cotton.

*Equipment* — 1 enamelled batch vessel, 2 cu. m., with jacket; 1 brick-lined decomposing vessel, 10 cu. m.; 1 filter press, 1,000×1,000 mm.

*Process* — In the batch vessel, 750 kg. of chlorosulphonic acid are heated to 35°C. Within 3-4 hr. 127 kg. of " acridone acids " are introduced through a sieve at a temperature of 35°-40°C. The mixture is then stirred for 2½ hr., and when everything is dissolved, the solution is then pressed into the decomposing vessel with 4 cu. m. of water and 2,000 kg. of ice. The temperature must not exceed 25°C. The mixture is stirred for 2 hr., filtered in the filter press and washed until neutral with water at 60°C. Yield: 114 kg. The colour of the solution in concentrated sulphuric acid is brownish yellow. The vat colour is brown; by transmitted light, dull violet. The powder is of 76·8 per cent strength, the paste of 10 per cent strength.

*Process Control Tests* — After introducing and stirring, a sample is tested as to complete solution by rubbing in the hands.

## APPENDIX 50

### INDANTHREN PRINTING BROWN B BASE

*Principal Use* — Printing dyestuff for cotton.

*Equipment* — 1 brick-lined autoclave, 3 cu. m., heatable, with enamelled stirrer; 1 brick-lined, box-shaped suction filter with stoneware filter, 2·5 sq. m. filtering area.

*Process* — In the autoclave 124 kg. of naphthalenetetracarboxylic acid (tetra acid), 170 kg. of ethoxy-1·2-phenylenediamine (ethoxyorthamine) and 80 kg. of 30 per cent acetic acid are heated at 165°C. under 6 atm. excess pressure for 16 hr. while stirring (Test 1). Then the mixture is cooled to 30°C., sucked off, washed with water until neutral and dried in the drying closet at 100°C. For standardizing against commercial article the product dried and ground is first dissolved in sulphuric acid and reprecipitated.

*Process Control Tests : Test* 1 — A sucked-off sample from the vessel is extracted with boiling dilute caustic soda solution and filtered. When the filtrate is acidified, flakes of tetra acid must no longer precipitate.

*Raw Materials, Intermediates & Finished Product Specifications* —

$\left.\begin{array}{l}\text{Tetra acid}\\ \text{Ethoxyorthamine}\end{array}\right\}$ test for dyestuff.

Finished product: brown powder, dissolves in conc. sulphuric acid to a red solution; vat, wine-red.

## APPENDIX 51

### INDANTHREN PRINTING BROWN 3R

*Principal Use* — Dyestuff for printing cotton.

*Equipment* — An iron vessel of 7 cu. m. provided with heating and cooling coil; an enamel vessel of 2 cu. m.; and a "Monstre" press.

*Process* — 100 kg. of Indanthren Printing Brown 3R base well made into a wet paste are introduced into the vessel of 7 cu. m. containing 4,000 kg. of alcohol (20 per cent); 125 kg. of caustic soda solution (35 per cent) are added and the whole is heated to 70°C. Then 60 kg. of hydrosulphite powder are introduced. The reduction is performed at 80°C. ( Sample 1 ). After cooling down to 25°-30°C., $CO_2$ is introduced until the leuco compound of the dyestuff is completely precipitated ( Sample 2 to be taken ). This takes about 4 hr. The whole is brought into the " Monstre " press, washed with cold water until neutral and pressed. The filtrate and the first washing water are collected in order to recover the alcohol.

The leuco compound of the dyestuff is introduced into the vessel of 2 cu. m., mixed with 5 per cent of caustic soda ( calculated on dry dyestuff ) used in the form of a lye. The whole is heated to 50°C. and air is blown in. The oxidation is finished after about 3 hr. The dyestuff may be adjusted to conform with the Suprafix paste.

*Process Control Tests : Sample* 1 — The vat must be clear apart from some flakes.

*Sample* 2 — A drop test on filter paper must not produce a halo that turns brown in air. The filtrate when mixed with bisulphite must not show any precipitate.

*Final Test* — A sample of the oxidized dyestuff is sifted through a screen of 250 mesh/sq. cm., then adjusted to a content of 16 per cent of dyestuff and tested by printing. If the print is uneven, oxidation was not sufficient. If the fixation of the dye is insufficient, iron chloride may be added.

## APPENDIX 61a

## Anthrasol Brown IRRD

*Principal Use* — For printing and dyeing textiles.

*Equipment* — Iron stirring vessel with jacket, 2 cu. m. ( esterification vessel ) ; iron stirring vessel with jacket, 7·5 cu. m. ( distiller ), with iron condenser and iron receiver, wooden filter press 800 × 800 mm. ; iron stirring vessel with jacket, 7·5 cu. m. ( salting out vessel ) ; open iron suction filter, 3 sq. m., with cloth filter and iron receiver ; and an iron stirring vessel with high-speed stirrer and lower outlet, 0·5 cu. m.

*Process* — Into 750 kg. of pyridine II contained in the esterification vessel 175 kg. of chlorosulphonic acid is run in at 15°-20°C. within 3-4 hr. Then 100 kg. Indanthren Brown RRD ( 100 per cent ), dried and ground, are introduced and after stirring for a short time 60 kg. of electrolytic iron are added. The mixture is heated to an internal temperature of 40°C. by 60°C. warm water. After heating has been stopped the temperature still mounts to 50°C. and is kept there. After about 3 hr. the esterification is complete.

The batch is pressed by nitrogen into the distiller, which contains a solution of 270 kg. of soda in 3,500 l. of water. After addition of 0·5 kg. of rape seed oil ( or other anti-frothing agent ) the pyridine is distilled off into an iron receiver at 30°-35°C. under vacuum ( duration 10-14 hr. ).

After distillation is over, the product is filtered off in the 800 mm. wooden filter press at about 20°C. The filtrate runs into the sewer ; it must contain only traces of anthrasol. The crude ester in the same 7·5 cu. m. distiller is pasted up with 5,500 l. of water, some soda and 10 kg. of kieselguhr, heated to 90°C. and filtered into the 7·5 cu. m. iron salting-out vessel through a wooden filter press, pre-heated by steam. The filter product is washed with hot water, until anthrasol can no longer be detected in the filtrate. The wash water at first runs to the mother liquor and then into the distiller, where it is used for the next batch. The filtrate is cooled to about 30°C. while stirring weakly, salted out with 6 per cent of salt and cooled to 20°C. The ester salted out is sucked off on the open iron suction filter ( duration 15-20 hr. ). The filtrate runs into an iron receiver. The suction filter product is made into a paste with 6 kg. of 33 per cent caustic soda solution in the 0·5 cu. m. iron stirring vessel and dried at 55°-60°C., at first under normal pressure and then in the vacuum. The manufactured article is obtained with a net content of about 55 per cent. The dry material is ground in a cross-beater mill and standardized against type ( about 50 per cent of pure dyestuff ) with sodium-dimethyl-aniline-sulphonate and sodium sulphate.

In order to dry the pyridine, its aqueous solutions are azeotropically distilled in a special equipment after addition of benzene and then redistilled. The loss amounts to 8-10 per cent.

*Process Control Tests* — (a) After esterification is finished, a sample from the esterification vessel is added to a soda solution, sucked off at 50°-60°C. and washed with warm water. The filter residue is tested for absence of vat dyestuff by vatting.

(b) Before the distillation is started, a test as to alkaline reaction to phenolphthalein must be made.

(c) The distillate is tested for anthrasol with acid and sodium nitrite.

(d) Distillation is finished as soon as the pyridine content of the distillate is $<1$ per cent.

(e) Before clarifying, a test as to complete dissolution of anthrasol is necessary. If required, water has to be added.

(f) The residue on the paste is tested for anthrasol by extracting with hot water and to vat dyestuff by vatting.

(g) A sample of the anthrasol salted out is sucked off and the filtrate is tested for anthrasol with nitrite and acid.

*Raw Materials, Intermediates & Finished Product Specifications* — Vat dyestuff, pyridine and iron have to be tested in the laboratory by esterifying a sample.

In the finished product the dyestuff content is determined by gravimetric analyses with iron chloride and acid. The yield is indicated as pure dyestuff of 100 per cent.

A sample is standardized against Anthrasol Brown IRRD Powder with 10 per cent of sodium dimethyl-aniline-sulphonate and sodium sulphate and compared with the type by textile printing. Anthrasol Brown IRRD Powder contains 50 per cent of pure dyestuff. For testing the fastness to storing, a sample is maintained at 55°C. for 3 weeks; after this time no decomposition must appear.

## APPENDIX 61b

### ANTHRASOL BRILLIANT PINK I3B

*Principal Use* — For printing and dyeing textiles.

*Equipment* — Iron stirring vessel with jacket, 2 cu. m. (esterification vessel); iron stirring vessel with jacket, 7·5 cu. m. (distilling vessel), with iron cooler and iron receiver; wooden filter press 800 × 800 mm.; iron stirring vessel with jacket, 7·5 cu. m. (salting-out vessel); open iron suction filter, 3 sq. m., with cloth filter and iron receiver; and an iron stirring vessel with high-speed stirrer and lower outlet, 0·5 cu. m. (conche).

*Process* — In the esterification vessel, 180 kg. of chlorosulphonic acid are run into 1,000 kg. of pyridine II within about 3 hr. at 15°-20°C. Then 100 kg. of Indanthren Brilliant Pink 3B of 100 per cent strength, dried and ground, are introduced and, after stirring for a short time, 80 kg. of electrolytic iron are added. The mixture is heated with water of 60°C. to an inner temperature of 40°C. After the heating is finished, the temperature rises to 48°-50°C. and is kept at that level. After 2-3 hr. the esterification is finished. The batch is pressed with nitrogen into the distilling vessel containing a solution of 280 kg. of soda in 4,000 l. of water. After addition of 0·5 kg. of rape oil (or any other anti-frothing agent), the pyridine is vacuum distilled into an iron receiver at 30°-35°C. in 12-14 hr.

As soon as the distillation is finished, the contents of the vessel are filled up to 4,000 l., and after addition of 10 kg. of clarifying carbon, filtered at 80°C. through a wooden filter press pre-heated by steam into the iron salting-out vessel of 7·5 cu. m. The pressed product is rinsed with water of 60°C. and washed with warm water until anthrasol is no more detected in the filtrate. At first, the washing water runs to the mother liquor, then into the distilling vessel, where it is used for the next batch. Gradually, the filtrate is salted out with 10 per cent salt, cooled down while being stirred slowly and sucked off on the open iron suction filter. The filtrate runs into an iron receiver.

The product with 12 kg. of caustic soda solution (33 per cent) are converted into a paste in the iron stirring vessel of 0·5 cu. m.; then they are dried at 50°-60°C., at first under normal pressure, then in the vacuum. The product is 55 per cent pure. The dried product is ground on a gross beater mill and standardized against type (30 per cent of pure dyestuff) with Glauber's salt and 6-8 per cent of sodium dimethyl-anilinesulphonate.

In a special equipment, for drying, the pyridine, after addition of benzene, is azeotropically distilled from its aqueous solutions and redistilled. The loss amounts to 8-10 per cent.

*Process Control Tests* — (a) After finishing esterification, a sample from the esterification vessel is mixed with soda water, filtered at 60°C. and washed with warm water. The residue on the filter is tested by vatting as to absence of vat dyestuff.

(b) Before beginning of the distillation, a test is to be made as to phenolphthalein-alkaline reaction.

(c) The distillate is tested with acid and sodium nitrite as to a probable anthrasol content.

(d) The distillation is finished as soon as the pyridine content of the distillate is less than 1 per cent.

(e) Before clearing the ester solution, it must be ascertained whether the anthrasol is completely dissolved. If necessary, water has to be added.

(f) The pressed product is tested by extraction with hot water as to anthrasol, and by vatting as to vat dyestuff.

(g) A sample of the salted-out anthrasol is sucked off and the filtrate is tested with nitrite and acid for anthrasol.

*Raw Materials, Intermediates & Finished Product Specifications* — Vat dyestuff, pyridine and iron are to be tested by a test esterification in the laboratory.

In the finished product, the content of dyestuff is gravimetrically determined by analysis with iron chloride and acid. The yield is indicated as pure dyestuff of 100 per cent strength. A sample is standardized with Glauber's salt and 6-8 per cent of sodium dimethylaniline sulphonate to Anthrasol Brilliant Pink I3B powder and compared with the type by textile printing. Anthrasol Brilliant Pink I3B powder contains 30 per cent of the pure dyestuff. For testing the resistance to storing, a sample is kept for 3 weeks at 55°C. After that time it must not show any decomposition.

## APPENDIX 61c

### ANTHRASOL RED IFBB

*Principal Use* — For printing and dyeing cotton and artificial fibres.

*Equipment* — Iron vessel, 2 cu. m., with stirring device and jacket ( esterification vessel ) ; leaded iron vessel, 7 cu. m., with stirring device ( acid separator ) ; wooden suction filter with filter cloth, 3 sq. m. ; iron vessel, 7·5 cu. m., with stirring device and jacket ( distiller ), with iron cooler and iron receiver ; 2 wooden filter presses, 800 × 800 mm. iron vessel, 7·5 cu. m., with stirring device and jacket ( vessel for salting out ) ; and an iron vessel, 0·5 cu. m., with high-speed stirrer and lower outlet.

*Process* — In the esterification vessel 168 kg. of chlorosulphonic acid are run into 1,000 kg. of pyridine at 15°-20°C. within 3-4 hr. Then, 95 kg. of Indanthren Red FBB 100 per cent, dried and ground, and 100 kg. of bronze copper grindings are added at 23°-25°C. The temperature rises to 40°-43°C. ; the esterification is mostly finished after half an hour. The batch is pressed by nitrogen into the acid separator which contains 4,000 l. of water of 10°C. After stirring for half an hour, the pyridine salt of the ester becomes solid and granular ; it is sucked off in the suction filter and washed with some water from the vessel.

In the distilling vessel the pyridine salt of the ester is introduced into 4,000 l. of water and 120 kg. of caustic soda solution ( 33 per cent ) and after addition of 0·5 kg. of rape seed oil ( or other anti-frothing agent ), the pyridine is distilled under reduced pressure at 30°-35°C. into an iron receiver. When the distillation is finished, the vessel is filled up to 3,600-4,000 l. and filtered through a steam pre-heated wooden filter press into the vessel for salting out. The mass remaining on the filter is rinsed with water of 50°C. and washed until anthrasol is no longer detectable in the filtrate. The filtrate is salted out at 45°C. by addition of 10-12 per cent of salt ( about 450 kg. ) and cooled to 20°C. while stirring from time to time. The ester is filtered off in a wooden filter press and washed with about 1,000 l. of common salt solution.

The pressed product is pasted with 4 kg. of caustic soda solution ( 33 per cent ), 32 kg. of molasses, 8 kg. of urea and 8 kg. of dimethylsulphanil salt in the vessel with high-speed stirrer and dried at 50°-60°C., at first under normal pressure and then under reduced pressure. The manufactured product has about 35 per cent purity and is adjusted to type with Glauber's salt after grinding in a cross beater.

The pyridine is azeotropically distilled. The loss amounts to 8-10 per cent.

*Process Control Tests* — (a) When the esterification is finished, a test portion taken from the esterification vessel is added to water and washed. The pyridine salt is dissolved in dilute caustic soda solution. On filtering only copper sludge, which is not coloured red by vat dyestuff, should remain.

(b) Before the beginning of the distillation a test as to reaction alkaline to phenolphthalein has to be carried out.

(c) The distillate is tested as to the absence of anthrasol by nitrite and acid.

(d) The distillation is finished when the pyridine content of the distillate is less than 1 per cent.

(e) Before clarifying, a sample is tested as to whether the anthrasol has been completely dissolved. If necessary, water has to be added.

(f) The residue remaining on the filter press is tested for anthrasol by extracting with hot water and as to vat dyestuff by reducing.

*Raw Materials, Intermediates & Finished Product Specifications* — Vat dyestuff, pyridine and copper grinding has to be tested by test esterification in the laboratory.

The dyestuff content of the finished product is gravimetrically determined by means of iron chloride and acid. A sample is adjusted to Anthrasol Red IFBB powder by addition of Glauber's salt and compared with the type by textile printing. Anthrasol Red IFBB powder contains 30 per cent of pure dyestuff.

## APPENDIX 62

### TETRANITRO CARBAZOLE

*Principal Use* — Standardizing of Nirosan (plant protective agent).

*Equipment* —

(a) *Sulphonation* — Supply vessel for sulphuric acid, 12 cu. m. Sulphonation vessel, 5 cu. m., with stirrer and heating.

(b) *Nitration* — 2 nitration vessels, 9 cu. m., with stirrer, 2 reservoirs, 20 cu. m.; 2 measuring vessels, 3 cu. m.

(c) *Effusion & Insulation* — 5 effusion vessels, 12-20 cu. m., lined with Igelit and bricks, with stirrer and cooling coil; 2 admission vessels, 7·5 cu. m.; 2 filter presses 1,200 × 1,200 mm., 32 frames; 3 pasting vessels, 12 cu. m., with stirrer; 1 intermediate vessel, 7·5 cu. m., with stirrer; 1 vessel for dissolving sodium carbonate, 5 cu. m., with stirrer; 1 container for hot water, 5 cu. m.; 2 centrifugal pumps; 1 ventilator made of Igelit; 1 clay tower; 2 stoneware suction filters; and 2 electrical vacuum pumps, 15 kW.

(d) *Drying* — 3 cylinder drying machines, heating surface, 23 sq. m.

(e) *Standardizing* — 4 Perplex mills with mixing drum; 1 Excelsior mill with mixing drum; and 1 ventilator.

*Process* — Carbazole is dissolved in sulphuric acid, 95·5 per cent (formation of disulphonic acid and trisulphonic acid). Then it is mixed with nitrating acid containing 15 per cent of nitric acid and 85 per cent of oleum (20 per cent) and heated. The product is poured into water, filtered and washed. After conversion into a paste with water, the product is dried on a cylinder drying apparatus. The dried material is ground and mixed with bleaching clay.

*Process Control Tests* — Control of the strength of the sulphuric acid and of the nitrating acid.

Determination of the dry content of the tetranitrocarbazole and its nitrogen and sulphur contents. Determination of the sedimentation of a Nirosan suspension after 10 and 30 min.

*Raw Materials, Intermediates & Finished Product Specifications* — Carbazole is controlled by determination of its degree of purity. The efficiency of the finished products, tetranitrocarbazole and Nirosan, against pests is controlled in the plant protectives laboratory.

## APPENDIX 63

### CARBAZOLE ESTIMATION (" IMIDO BASE ")

*Principle of the Method* — The carbazole is separated as N-nitrosocarbazole. It is then decomposed by boiling with ferrous sulphate solution and the nitric oxide produced is measured. Phenanthrene, anthracene and acridine do not interfere with the estimation.

For the quantitative estimation of carbazole, the determination of the m.p., solubility and chlorobenzene-insoluble substances are carried out. Besides, the anthracene content is also determined.

*Details of Analysis* — About 0·6 gm. (*a*) of the substance is dissolved by heating in a 300 c.c. Erlenmeyer flask with 40 c.c. glacial acetic acid using a reflux condenser. The solution is allowed to cool to 80°-90°C. and 30 c.c. of 0·5N sodium nitrite solution is added, the mixture is shaken and immediately placed in ice water. After frequent shaking and then letting it stand for 15 min., 150 c.c. of ice-cold water are added, and the mixture is allowed to remain in ice-cold water for a further 30 min. The separated nitroso-compound is carefully filtered on a filter funnel with paper filter. Any residue in the flask is brought on the filter paper with the help of the filtrates and washed 5 times with about 20 c.c. ice-cold water each time (test for nitric oxide).

The precipitate is again taken up in the flask with the help of 75 c.c. of pure glacial acetic acid and the flask is closed with a 3-holed rubber stopper carrying: (1) a tube provided with a glass stopper for passing carbon dioxide, the tube reaching the bottom of the flask; (2) a conducting bout tubing connected to an azotometer of 100 c.c.; and (3) a 50 c.c. dropping funnel. A strong current of carbon dioxide is passed through the flask till all the air is displaced which is established by allowing the gas to collect over pure 30 per cent caustic potash solution in an azotometer and observing whether it is wholly absorbed.

The glass stopper of the tube is now closed and 25 c.c. of iron sulphate solution is added through the dropping funnel ( 200 gm. of $FeSO_4.7H_2O$ plus 400 c.c. water plus 100 c.c. sulphuric acid ), well shaken, and heated over a flame. In the end it is heated to boiling till the gas stream is completely absorbed. Now, for a short time, a moderate stream of carbon dioxide is passed through the flask which is again heated to boiling till all the gas stream is absorbed. The gas is allowed to remain over 30 per cent potassium hydroxide solution for 30 min., and its volume is measured. Simultaneously the temperature and pressure are determined. If $b$ c.c. of nitric oxide, corrected to N.T.P., are obtained, the sample contains $0{\cdot}746\ b/a$ plus 1·4 per cent carbazole, 1·4 being the solubility correction.

NOTE — If, instead of a commercial carbazole, a crude anthracene sample whose carbazole content is less than 30 per cent is taken for analysis, double the amount is weighed.

The solubility correction amounts to less than 1·4 per cent in the case of 15 per cent content and to 0·8 per cent in the case of 5-15 per cent content, and is very much lower in the case of 5 per cent content.

*Qualitative Tests* — (1) Determination of m.p. ; (2) solubility determination.

7·5 gm. of 100 per cent according to analysis are dissolved by short heating in a 150 c.c. Erlenmeyer flask containing 100 c.c. of chlorobenzene. It is allowed to cool and remain overnight in a thermostat at 20°C. The precipitate is filtered in a weighed Gooch crucible, and the precipitate sticking to the walls of the flask is brought on the filter with the help of the filtrate, the precipitate is pressed well, washed with 25 c.c. benzene, dried and weighed. If the weight is $c$ gm.

$$L = ( 7{\cdot}5 - c ) \times \frac{100}{7{\cdot}5} = \text{solubility}$$

$$L \text{ for pure carbazole} = 8{\cdot}8.$$

(3) Estimation of chlorobenzene insolubles : 2 gm. of the dried sample is dissolved by short heating in a 300 c.c. Erlenmeyer flask containing 100 c.c. of chlorobenzene, and provided with an air condenser. The hot solution is filtered in a weighed Gooch crucible, washed with pure 50 c.c. of chlorobenzene at 70°-80°C. dried and weighed. If the weight is $d$ gm.

$$d \times 50 = \text{per cent chlorobenzene insoluble matter.}$$

*Estimation of Anthracene in Carbazole* — (a) *Gravimetric Estimation* — The gravimetric estimation of anthracene in carbazole is carried out with difficulty according to the method of Luck with the modification that the quantity of chromic acid solution is doubled.

(b) *Anthracene Estimation in Ultra-violet Light : Principle of the Method* — Anthracene in carbazole can be estimated with the help of an ultra-violet lamp based upon the strength of fluorescence by comparison with carbazole solution of known anthracene content. The fluorescence can be considerably intensified if the fluorescence due to carbazole is removed, i.e. if the carbazole is converted into a non-fluorescent derivative, while at the same time the fluorescence of anthracene is retained. This is easily accomplished by the conversion of carbazole into its nitro-derivative. This does not fluoresce and any disturbance due to the yellow colour of the nitroso-carbazole can be easily overcome by dilution.

*Details of the Determination* — 0·10 gm. of the carbazole under investigation is dissolved in 100 c.c. of glacial acetic acid in a 300 c.c. Erlenmeyer flask provided with a glass stopper, and slowly heated. The solution is allowed to cool, 1 c.c. of normal nitrite solution is added, the flask is closed, and allowed to stay for 10 min. and occasionally shaken. From this solution 5 c.c. are taken and diluted with 95 c.c. of glacial acetic acid, and its fluorescence is compared under an ultra-violet lamp with that of a solution of known concentration and known anthracene content.

## APPENDIX 64

### DIFLUORDICHLORMETHANE

*Principal Use* — Refrigerating agent.

*Equipment* — (1) 4 autoclaves, cast-steel lined with sheet iron, working pressure 50 atm. over normal ; 2 of them with contents of 2,700 l. ( 1 of them in use, 1 in reserve ) ; 1 of them with content of 250 l., and the other of 100 l. ; (2) 2 Igelit washing towers filled up with Raschig belts of graphite ; (3) 2 iron washing towers ; (4) 1 iron gas container, contents of 6 cu. m. ; (5) 1 compressor ( capacity per hr. 40 cu. m., maximal pressure 20 atm. excess pressure ) ;

(6) 1 iron supply vessel for crude Frigen, content of 2 cu. m., working pressure of 13 atm. over normal; (7) 1 iron supply vessel for concentrated hydrofluoric acid, contents of 20 cu. m., working pressure of 3·5 atm. over normal; (8) 1 iron weighing vessel for concentrated hydrofluoric acid, contents of 0·5 cu. m., working pressure of 3·5 atm. over normal; (9) 1 iron supply vessel for carbon tetrachloride, contents of 20 cu. m.; (10) 1 iron supply vessel for sulphuric acid, contents of 10 cu. m.; (11) 1 iron supply vessel for caustic soda solution, contents of 10 cu. m.; (12) 1 iron distilling vessel for Frigen, contents of 2 cu. m., working pressure of 2 atm. over normal while working with column; (13) 1 iron receiver for first running; (14) 3 iron receivers for pure Frigen, contents of 5·5 cu. m. each, working pressure of 13 atm. over normal; (15) iron vessel for crude $F_{11}$, contents of 0·9 cu. m., working pressure of 3·2 atm. excess pressure; (16) iron vessel for pure $F_{11}$, contents of 0·9 cu. m., working pressure of 3·2 atm. over normal; (17) iron distilling vessel for $F_{11}$, contents of 0·8 cu. m., working pressure of 3·2 atm. over normal while working with column.

*Process* — The autoclave (1) is charged with 400 kg. of antimony trichloride. Then, out of a measuring vessel, 1,540 kg. of carbon tetrachloride, in which about 20 kg. of chlorine are dissolved, are run in (1). Finally 500 kg. of hydrofluoric acid (98-99 per cent) are weighed in (8) and pressed into (1) by compressed air. The gas is allowed to blow off the autoclave which is heated with low pressure steam at about 30 atm. Control through a baffle plate with a differential manometer. Finally the autoclave is entirely released and cooled down. In (2) the released gas is set free from the main bulk and, in a washing tower (3), sprayed with caustic soda solution from the last traces of hydrochloric and hydrofluoric acid. The gas collects in (4), is dried in a washing tower (3), which is sprayed with concentrated sulphuric acid, and liquefied to crude Frigen through (5) with a brine, condenser fixed behind. During this process Frigen and inert gases, containing eventually $F_{11}$, are allowed to blow off through a release valve.

The cooled-down autoclave is filled up once more. The catalyser, i.e. antimony chloride, remains in the autoclave until exhaustion.

The crude Frigen yield of one operation (about 1,200 l.) and the first runnings of a previous distillation (about 200 l.) are drawn off into the distilling vessel (12); here they are fractionated in the column with little steam (steam jacket), while beginning with depriving of air up to the constancy in pressure and, after having taken off first runnings (about 200 l.), the pure Frigen is collected in one of the receivers (14). The residue in (12), 70-90 l., is, after the release, still crude $F_{11}$ contaminated with Frigen and higher-boiling constituents, and is pressed to (15). This crude $F_{11}$ either goes back to the manufacturing process instead of carbon tetrachloride or is fractionated in (17), where pure $F_{11}$ is collected in (16). The development of an additional apparatus for the technical manufacture of $F_{13}$ is still in its beginnings. According to the above-mentioned method the by-product $F_{13}$ results only in a small quantity, so that its manufacture is not paying.

Yield of pure Frigen = 1,090 kg.

*Process Control Tests: Examination of Frigen* —

(a) *Determination of Water* — 300 l. Frigen gas, measured with a gas meter, are conducted through 3 weight tubes filled with phosphorus pentoxide. The increase in weight gives the content of water. The percentage of water in the Frigen

$$= \frac{\text{increase of weight}}{750} = 100$$

(b) *Constituents Insoluble in "Bayoloil"* — 100 c.c. Frigen gas are taken off with a Hempel mercury burette. This burette is connected with a Hempel absorption pipette filled with "Bayoloil", in which the gas is shaken up to the constancy of volume of insoluble gas determinated in the burette.

(c) *Boiling Interval & Higher-boiling Contaminations* — A little more than 100 c.c. liquid Frigen are put into a Dewar vessel with graduations, the Frigen is evaporated in the presence of several carborundum crystals and the boiling temperature is determinated from 5 to 5 c.c.

(d) By adding 5 c.c. of Frigen to a solution of silver nitrate in 5 c.c. of absolute alcohol, the solution should not be rendered turbid.

*Raw Material, Intermediates & Finished Product Specifications* — (a) The first boiling point of Frigen near 760 Torr must not be higher than −29°C. The boiling interval between the first boiling point and the point when only 15 per cent of the sample are left, must not exceed 0·5°C. Frigen must not contain more than 0·0025 per cent water. Constituents insoluble in " Bayoloil " must not exceed 5 per cent by volume. Frigen must be entirely neutral ( free from hydrochloric acid ).

(b) The hydrofluoric acid must be of a strength of more than 96 per cent, if possible, 98-99 per cent.

### APPENDIX 65

### OCTADECYL-ISOCYANATE ( Intermediate for Persistol VS )

*Principal Use* — For producing water-repelling effects on fibres.

*Equipment* — 3 enamelled vessels, 1 cu. m. each ; 2 homogeneously leaded receivers, 500 l. each ; 1 iron distilling vessel with receiver, 500 l. ; and 1 equipment for destroying phosgene.

*Process* — Into the 1 cu. m. enamelled vessel 300 kg. of chlorobenzene are filled, heated to 60°C. and 300 kg. of octadecylamine, previously melted, are added. The temperature is raised to 90°C. and the amine transformed into its hydrochloride by introducing about 40 kg. of hydrochloric acid gas. The batch is next pressed into the second enamelled vessel and, at a temperature of 105°-110°C., 10-15 kg. of phosgene per hour are next introduced. The total quantity for the conversion into the isocyanate is about 150 kg. of phosgene.

After stirring for 2 hr., nitrogen is passed through the batch for 3-4 hr. in order to expel hydrochloric acid and phosgene and then the chlorobenzene is distilled off *in vacuo* ( about 100 mm. ). Temperature can be raised up to 160°C. The residual raw isocyanate is distilled *in vacuo* ( 3-5 mm.) at a temperature of about 210°C. to the pure product. The unreacted phosgene, escaping together with hydrochloric acid, is destroyed in an absorption plant.

Yield from one batch : about 280 kg.

*Process Control Tests* — The final point of the phosgenation is determined by distillation of a sample. There must not be more than 5-6 per cent of undistillable residue.

The progress of the conversion is recognizable by the fact that in a cooled sample the quantity of octadecylamine-hydrochloride, insoluble in chlorobenzene, decreases more and more, a clear, brown solution finally resulting.

*Raw Materials, Intermediates & Finished Product Specifications* — Octadecylamine : the raw product of about 90 per cent is previously distilled *in vacuo* to the pure product. The finished product is a clear, transparent oil.

( *To be continued* )

# Quality of Sugar Manufactured in India

THE DIRECTOR, INDIAN INSTITUTE OF SUGAR Technology, Kanpur ( India ), has issued his review on the quality of sugar manufactured in India by the central sugar factories and refineries for the season 1947-48.

The data supplied by 124 factories show that there is no significant improvement in the quality of sugar manufactured in 1947-48 over the sugar of the previous season. In fact, the production of ISS. No. 28, which had occupied a position next to the standard ISS. No. 27, has registered a drop from 23·28 per cent in 1946-47 to 17·76 per cent in 1947-48. As before, the most predominant colour grade in 1947-48 was ISS. No. 27, which accounted for 56·33 per cent of the total production. The production of ISS. No. 26 was slightly higher. 98 per cent of the total production was in the colour grades ISS. Nos. 28, 27, 26 and 25.

Regarding grain size there was a reduction in size A and size C, while a corresponding increase in size E was recorded. Over 97 per cent of the total production was covered by the sizes C, D, E and F, the size D alone accounting for 47·68 per cent.

What is disappointing is that some factories produced a large number of grades of sugar to the extent that one factory recorded 23 different grades. It is highly desirable that methods of standardization should be strictly adhered to so that the number of grades produced by any factory is restricted.

G. G. RAO

# REVIEWS

**Waste-heat Recovery from Industrial Furnaces,** being a treatise based upon a series of papers presented to the *Institute of Fuel* ( Chapman & Hall Ltd., London ), 1948, pp. 384+ix. Price 35*s*. net.

THE VOLUME UNDER REVIEW CONSISTS OF A number of papers by different authors. The papers were presented in the first place to the *Institute of Fuel,* London, and owe their origin to the urgent necessity during war-time to conserve the fuel of Great Britain. They are all concerned with the study of heat transference and heat insulation and the recovery of waste heat from industrial furnaces with the object of increased overall efficiency. The Committee of the *Institute* responsible for the symposium is to be congratulated on presenting an important and useful treatise which, in spite of its mixed origin, gives the impression, not of piecemeal or patch-work composition, but of a coherent volume. This is attributable no doubt to able and careful editing.

The papers or chapters carry the following headings: (i) Total, Recoverable, and Returnable Heat in Combustion Gases by P. O. Rosin; (ii) Some Simplified Heat Transfer Data by Margaret Fishenden and O. A. Saunders; (iii) An Experimental Determination of the Factors Governing the Design of Regenerators with special reference to Coke-ovens by T.C. Finlayson and A. Taylor; (iv) Tubular Metallic Recuperators by G. N. Critchley and H. R. Fehling; (v) Waste-heat Boilers, by W. Gregson; (vi) The Utilization of Waste Heat in the Carbonizing Industries by J. G. King and F. J. Dent; (vii) Waste-heat Recovery in the Metallurgical Industry by J. A. Kilby, W. G. Cameron, E. C. Evans, A. H. Leckie, J. L. Harvey and A. E. Balfour; (viii) Waste-heat Recovery in the Glass-container Industry by W. A. Moorshead.

There is a brief but pertinent introduction by G. N. Critchley, showing in striking figures the money value of the fuel saved by installing fuel-saving ( i.e. waste-heat recovery ) equipment. The cost of the equipment is stated. There is an excellent index.

In the body of the book the main industrial furnaces considered are those dealing with coal carbonization, the metallurgical industries and the glass industries. The first two chapters are devoted to basic fundamental considerations; and they are the more important as Rosin's treatment of " Total, Recoverable, and Returnable Heat in Combustion Gases ", and the simplified " Heat Transfer Data " of Fishenden and Saunders are often referred to in the subsequent portions of the book. It is consequently advisable for the serious readers to spend time on these early chapters. They comprise only 66 pages.

Rosin's paper is an attempt at simplification of the usually tedious calculations involved in working out combustion and heat losses: it should be read with care. Perhaps its main service will be to help the technologist to visualize roughly but rapidly the prevailing combustion and heat conditions. Rosin's work is seldom easy reading — and this chapter is ( to the reviewer ) no exception. At the outset we come across an irritating statement. " Combustion ", he says, " is an industrial process for the manufacture ( *sic* ) of hot gases which are to be used in another industrial process for the production of goods of various kinds, such as metals, ceramics, coke, or chemicals." While a definition must delimit, it must also embrace all that it should — and to define combustion in the above terms is simply not true. If we are dealing with a particular *aspect* of combustion, we should say so. But perhaps the main objection to the statement is that by the end of the sentence we have lost sight of the combustion process itself and are dabbling in " metals, ceramics, coke, or chemicals ".

Rosin in this chapter spends time on developing a general or " statistical " relation between the net calorific value, the air requirement and the combustion gas volume for different fuels, and he deduces useful approximations. Perhaps the *most* useful is that the heat content of unit volume of the combustion gases obtained with theoretical combustion is roughly constant and independent of the calorific value of the fuel.

A later portion of the chapter is devoted to the heat content of combustion and waste gases and to the I/t diagram. The substance of this section requires care from the reader by way of interpretation, and the same remarks apply to the last section on " Recoverable and Returnable Heat ". Chapter I is of basic importance and contains much valuable matter ; but it is perhaps not written with the clarity which it deserves.

Chapter II by Fishenden and Saunders is a competent statement on the elements of heat transfer. It contains a brief treatment of the " numbers " of Reynolds, Prandtl ( Stanton ), and Nusselt. The complex nature of heat transmission is indicated, and there is a brief explanation of heat transfer by radiation and by forced and natural convection. The usefulness of this contribution is enhanced by the heat transfer data assembled in the last few pages of the book.

Finlayson and Taylor in the " Design of Regenerators with special reference to Coke-ovens " have written an essentially practical essay of value to coke-oven manufacturers and all concerned with design in this field. " Tubular Metallic Recuperators " by Critchley and Fehling embraces practical, experimental, and theoretical aspects of its subject. Corrosion is not overlooked, but the treatment here is none too thorough.

W. Gregson's chapter on " Waste-heat Boilers " is a general review, largely descriptive, and contains little revolutionary except a plea for the use of the gas turbine. Nevertheless, it constitutes a balanced statement in the body of the book : it will be valued more especially by the non-technical reader.

In " The Utilization of Waste Heat in the Carbonizing Industries ", King and Dent give a comprehensive description of their investigations into waste heat in coal carbonization practice. The improvements possible with horizontal gas retorts, continuous vertical retorts, intermittent vertical chambers, coke ovens, and water-gas plant are indicated. The essay is lengthy, but will repay careful attention by all concerned with the manufacture of coal gas or water gas. The brief appendix by Dr. S. Pexton is an admirable statement on the balance of fuel, steam and power in carbonizing plants.

Chapter VII, by several authors, deals with " Waste-heat Recovery in the Metallurgical Industry ". A large section is devoted to the iron and steel industry including coke-oven plant, blast furnaces and cupolas, melting furnaces, and reheating and heat-treatment furnaces. The dry-cooling of coke receives full and reasoned consideration. The second " half " of the chapter is devoted to the non-ferrous metal industry, and the future development of waste-heat boilers in connection with metallurgical industries. Reference is again made to the driving of gas-turbines by means of high pressure waste gas from boilers burning compressed blast-furnace gas and air.

In " Waste-heat Recovery in the Glass-container Industry ", W. A. Moorshead shows the application of the theoretical principles enumerated in chapters I and II to the problems of the glass industry. The case for the use of waste-heat boilers with glass-melting furnaces is so strong as barely to need argument.

The book, therefore, constitutes a good general guide to modern practice in the recovery of waste heat from industrial furnaces. The usual line of procedure at works is to utilize the sensible heat in the hot gases either for steam raising or for heating the air to be supplied for the combustion process, employing in the latter case regenerators or recuperators. Mention has already been made of a further possible development, namely the utilization of the hot gases in gas-turbines.

The problem of waste heat, however, exists in rather larger context. Waste heat may result not only from hot flue gases, but also from incompletely burnt fuel or hot rejects in any form. The present treatise leaves one with the feeling that a first-class comprehensive volume on the utilization and/or reduction of waste heat still remains to be written. The treatment of fundamentals given in the present volume certainly needs expanding to cover the field. But this expansion must not be cumbersome and at the expense of clarity. The subject is an intensely practical one, and the book must have its appeal essentially to engineers of all ranks. Carbon balances, however, must not be overlooked, and the losses due to incomplete combustion or to rejection of hot fuel or even hot solid or liquid matter from furnaces should be considered. No doubt we have stepped outside the field envisaged as " Waste Heat ", but the line of demarcation is artificial.

It remains to say that the appearance and printing of the book are good and that in its present form it will be of great service to engineers and fuel technologists concerned with waste-heat recovery. But may we not ask the *Institute of Fuel* to use it as a first step towards the compilation of further treatises on fuel economy ?

J. W. W.

**Television Production Problems,** by J. F. Royal ( NBC-Columbia University Broadcasting Series ) ( McGraw-Hill Book Co. Inc., New York & London ), 1948, pp. xii+179. Price $2.50.

THE COLUMBIA UNIVERSITY IN CO-OPERATION with the National Broadcasting Company started a scheme of instruction in all phases of radio programming and operation with as much workshop experience as possible. This book is based on an extension of the scheme to the art of television. It is really a course of lectures delivered by different authors touched up by the editor.

The following are the main topics dealt with : (i) Introduction by J. F. Royal ; (ii) The Science of Television by F. A. Wankel ; (iii) Television Writing Problems by R. P. McDonough ; (iv) Television Production Facilities by N. R. Kelly; (v) Settings for Television by R. J. Wade ; (vi) Production of Dramatic and Variety Programs by E. Sobol ; (vii) Opera in Television by H. Graf ; (viii) Problems in the Studio by F. Coe ; (ix) Television Mobile Unit Broadcasting by B. Crotty ; (x) Legal Problems in Telecasting by I. E. Lambert; and (xi) Television Advertising by R. Kraft.

The above "titles" indicate clearly the wide variety of topics dealt with. All the authors are experienced persons engaged by the NBC. The book covers a complete course of instruction that need be given in this field and is *typically* American. We do not believe yet in this type of courses in the universities. In the States there is considerable demand for specialization and the book is a clear indication of the way in which the Americans feel the need. Television is yet to make its way into India and when it comes in, the book will be most useful to the staff-training schools. But there is another way in which the book can find its utility. It is a very valuable addition to the general section of college libraries and as a book for general reading to our communication engineering students. As one reads through the book, one finds little that is new and there is little that does not occur as a matter of course to a systematic thinker. But we must remember that originality consists not in thinking differently from others but in thinking for oneself !

The printing and get-up of the book are excellent. The numerous photographs are aptly chosen and beautifully reproduced. The book can as well go in as a model to those engaged in book production problems !

S. V. CHANDRASHEKHAR AIYA

**An Introduction to Comparative Biochemistry,** by Earnest Baldwin ( Cambridge University Press ), 1948, 3rd edition, pp. xiii+164. Price 7*s*. 6*d*.

THIS EXCELLENT LITTLE VOLUME NEEDS NO introduction to students of biochemistry. The fact that the book has gone through 3 editions ( once reprinted ) in the course of a decade bears ample testimony to its excellence and to the popularity which it enjoys with those interested in biochemistry.

The author states that the aim of the book is twofold : first, to provide an elementary text-book of this special subject suitable for students of biochemistry, and, secondly, to provide a starting-point for those who, for any reason, may find themselves attracted to the subject. These two objectives have been more than fulfilled in the short space of 164 pages. The subject-matter has been selected carefully and the exposition is lucid. As Professor Hopkins has stated, the author has " illustrated very adequately, in relatively few pages, many of the main lines of current progress, leaving in the mind of the reader no doubts concerning their significance. Above all, he has displayed his own interest in the subject, an interest, intense enough to be infectious."

This edition has been revised, considerably enlarged, re-set and brought up to date. A new section on the transport of carbon dioxide has been added to chapter VI, and a new chapter ( VIII ) has been introduced to cover the comparative aspects of nutrition, digestion and metabolism, adding materially to the usefulness of the book.

A number of useful books and reviews has been added to the list of references. A classification chart is included to provide the student, who is not familiar with zoological nomenclature, with a ready means of deciding to what phylum or class a given animal should be assigned.

A. K.

**Supersonic Flow and Shock Waves,** by R. Courant & K. O. Friedricks ( Interscience Publishers Inc., New York & London ), 1948, pp. xvi+464. Price $7.00.

THIS BOOK IS THE FIRST OF THE SERIES OF books and monographs on pure and applied mathematics edited by H. Bohr, R. Courant and J. J. Stoker. The first two names are familiar to all students of mathematics, the latter of them being the author of the celebrated *Methoden*. The book under review appears to be an off-shoot of the scientific work done in connection with the last war and is, therefore, relevant to problems of national defence. The book is a model of clear and accurate exposition of a mathematical subject.

The book consists of six chapters. In the first the fundamental equations of hydrodynamic flow with proper thermodynamic considerations, but omitting viscosity and heat conduction, are obtained. In the second the differential equations of the hyperbolic type involving two variables are thoroughly discussed by the help of characteristic curves. In the initial value problem, the concepts of domain of dependence and range of influence are introduced. Propagation of discontinuities alongc haracteristic lines is then considered and a discussion of the hodograph transformation is given. Generalizations to more than two variables are indicated.

In the third chapter specific problems of one dimensional flow are taken up. Shocks are regarded as irreversible processes described by sudden jump discontinuities occurring across sharply defined surfaces. The jump conditions and theorems relating to shock transitions are established. Treatment is given of reflection of a shock by a rigid wall, interaction of a shock, Riemann's problem, detonation and deflagration waves, Chapman-Jouguet processes, Jouguet's rule and wave propagation in elastic-plastic material.

In the fourth chapter the treatment of special flows by the hodograph method is given, characteristics, Mach lines and Mach angle are introduced and then follow flows round a bend, along a bump and in a two-dimensional duct, phenomena in a jet by gas streaming in parallel supersonic flow out of an orifice and an account of shock polars, oblique shocks and flow round an air foil by perturbation methods. The boundary conditions for steady flow are critically examined and scope for further work is indicated. A qualitative discussion of flow in nozzles and jets follows in the fifth chapter. Cylindrically symmetric steady flow, steady conical flow and non-steady flow with spherical or cylindrical symmetry are treated in the last chapter of the book.

An excellent comprehensive bibliography is given at the end of the book. But the regret is that some of the important references come under unpublished war work. References to Chandrasekhar, the distinguished astrophysicist, Bethe and Gamow, the distinguished nuclear physicist and Weyl and Neumann, the famous top-ranking mathematicians, are found in this work. Chandrasekhar's reports to the Aberdeen Proving Ground on the normal reflection of a blast wave, on the decay of plane shock waves and on the condition for the existence of three shock waves have been referred to in the bibliography. This shows the amount of interest taken by the leading mathematicians and physicists on problems relating to national defence in a time of emergency.

N. S. NAGENDRA NATH

**Electrical Accidents,** by K. V. Karantha ( Harsha Printery & Publications, Puttur, S. Kanara, Madras ), 1948, pp. iii+136. Price Rs. 3/8.

THE BOOK IS THE FIRST OF ITS KIND IN INDIA. The subject-matter is dealt with in great detail with a number of illustrations from practice which can be understood by a reader with a preliminary knowledge of electricity. The book is written from the author's own experience and is based on the general practice followed in the province of Madras. It would have been better if information about the practice in other provinces was also incorporated and it is hoped that this will be done in the future editions to enhance its usefulness and make it a standard book of reference all over India.

The author has given in detail the causes of accidents in houses and outside, and has suggested the enforcement of Rule 48 of the I.E. Rules, 1937. In the Bombay province, the statutory provisions regarding the electric installations in houses, factories, places of public entertainment, etc., are strictly enforced and a licencing board grants licences to electrical contractors who are authorized to take up such works and conducts examinations for electricians and wiremen. It would be interesting to know the methods adopted in other parts of India. If the regulations in force have not actually prevented accidents, they will have to be modified in the interest of safety to the public and extension of electric power to rural areas.

The author has clearly described the physiological effects of the passage of electric current through the human body and explained the circumstances under which the electric shock is likely to prove injurious and fatal. The chapter regarding artificial respiration should have followed this, and it should have included other methods such as mechanical respiration aids. Recommendations of a few medicines commonly used in cases of electric burns would have been useful.

The chapter on the causes of electric accidents in dwelling houses, factories and in public utility concerns are very exhaustive and show conclusively how most of these accidents could be avoided by proper observance of the prescribed regulations under the *Indian Electricity Act and Rules.* The author has given details of clearances from the live conductors and the safety measures such as fencing, anti-climbing devices, etc., prescribed by the Madras Government. It would be better if these are standardized on an all-India basis and prescribed under the authority of the Central Electricity Board.

Provision of an adequate " earth " being essential for the operation of all safety devices such as fuses or earth leakage relays of circuit breakers, etc., it is very essential that this question should be properly understood. The author has considered this aspect and described in detail as to how proper earth connections should be made and how very low earth resistances should be maintained. This information would be very useful for all public utility engineers and engineering contractors.

The author has devoted a special chapter for the multiple earth neutral system and has shown both the advantages and disadvantages of the same and its application in the Madras province. The adoption of this system would be very useful for other provinces.

The causes of fire accidents by the use of electric appliances in houses and factories are discussed in detail and helpful suggestions to avoid them are given. Discussion and implications of some extracts from the regulations for the installation and maintenance of electric installations prescribed by the " fire insurance companies " would be very useful and should be included as an appendix.

On the whole, the book is very instructive and useful as a text-book for technical institutions and for examinations conducted by the Provincial Licencing Boards and as a reference book for the public utility concerns and factories. The author must be congratulated for publishing such a valuable book.

B. K. R. PRASAD

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**The Indian Sugar Industry, 1947-48, Annual,** edited by N. P. Gandhi (Gandhi & Co., Bombay), 1948, Vol. XIII, pp. xlviii+136+32. Price Rs. 6.

THE PRESENT ANNUAL REVIEW OF INDIAN Sugar Industry, like its fore-runners, contains an exhaustive account of the progress of the sugar industry during 1947-48, all the relevant data of the previous years being brought into well-compiled tables for ready comparison.

The author's preface is a succinct account of the present problems of the sugar industry and his own views on them. There are 41 highly informative tables given at the very beginning of the book. The review covers the progress of not only the sugar industry, but also the other allied subjects like power alcohol and cane cultivation. The subject is dealt with in all its aspects, and the world position is reviewed. An appendix giving an up-to-date list of sugar mills of India and Pakistan is included.

The author lays special stress on the futility of an elaborate tariff enquiry on the continuance of protection to sugar at a time when the industrial conditions, both in India and abroad, are very unstable, costs of production violently fluctuating, and the future of production, consumption and world trade in sugar cannot be predicted owing to the prevalence of insecure conditions. Protection to sugar industry must be maintained at the present juncture in the interest of the agricultural economy of India. The author has stressed the importance of intensified agricultural research and, therefore, the necessity of restoring the grant agreed to by the Government of India in 1947. This grant is estimated to be about Rs. 50 lakhs per year. A wider sales organization, on the same lines as of *NIWAS* of Java to cover every factory in India, has been advocated. Among other points stressed by the author are : improving the manufacture of *gur* and encouraging the manufacture of palm *gur* and the necessity for reducing the price of sugar by reducing the price of cane and the sugarcane cess to give an impetus to increased production and consumption of sugar.

The Annual cannot be missed by anyone closely associated with, or interested in, the Indian sugar industry.

G. G. RAO

**Plants Diseases : Their Causes and Control,** by Sudhir Chowdhury (Kitabistan, Allahabad & Karachi ), 1948, pp. 106. Price Rs. 4/8.

AS ONE GOES THROUGH THIS LITTLE VOLUME, the impression that the book has not been planned properly and that the subject-matter has been inadequately and poorly treated becomes confirmed by the time the end is reached. The author states that at the request of his students he has published his lectures in the form of a booklet. It may be that in the form of lectures the subject-matter was interesting enough to students, but, to publish the material in the form of a book, the author should have given greater attention to precision in terminology and proper sequence in the presentation of the subject.

The book is divided into six chapters. After a short introduction ( which forms chapter I ), " definitions and symptoms of diseases " are discussed in the second chapter. The material is evidently based on Heald's Manual of plant diseases, and the author's attempt at brevity and simplicity has resulted in a large number of inaccurate statements and generalizations which, in the opinion of the reviewer, is not beneficial to students for whom the book is intended. Chapter III should have formed the introduction as it is very necessary for a beginner to know the structure, life history, etc., of the causal organisms before making an attempt to study the diseases they produce and the symptoms which are manifested.

The book abounds in grammatical, spelling and typographical errors. There is little in the volume which the reviewer can commend to readers. The publication is priced high ; a third of the price would have been reasonable.

V. S. RAO

**German Primer for Science Students,** by Haragopal Biswas ( University of Calcutta ), 1948, 2nd edition, pp. xiii+250+iii. Price Rs. 7/8.

THIS PUBLICATION IS FAMILIAR TO STUDENTS in this country, and particularly to those who have attempted to gain a working knowledge of scientific German by the "self-taught" method. The reviewer himself had occasion to use the book ( 1st edition ), and he can unhesitatingly recommend it as a useful aid to the learning of German.

The present edition has been revised and a number of fresh exercises have been added. The sub-sections of exercises in physics, physical geography, pharmacology, chemistry and mathematics have been enlarged. An interesting feature of the new edition is the inclusion, in the last chapter, of passages of general scientific interest, biographical sketches of men of science, and a selection of well-known Indian short stories and extracts from the works of Kalidasa, Goethe, Shakespear, Schiller and Tagore.

A. K.

**Elastomers and Plastomers, Their Chemistry, Physics and Technology, Part III,** edited by R. Houwink ( Elsevier Publishing Co. Ltd., New York, Amsterdam, London, Brussels ; Cleaver-Hume Press Ltd., London ), 1948, pp. 174. Price 25*s*. net.

THE PUBLICATION OF PART III BEFORE PARTS I and II of the volume on elastomers and plastomers might appear a bit unusual as would, for instance, serving first the last dish of a meal while the others are still in preparation. But the reason for it might very well be, as in the case of Elsevier's 20 volume *Encyclopedia of Organic Chemistry*, whose publication started off with the 14th volume, the practical consideration of immediate public demand for the sort of information contained in this particular part. In fact, whereas parts I and II are to be entitled, respectively, " General Theory " and " Manufacturing Properties and Applications ", part III, under review, deals with the methods of testing and analysis of elastomers and plastomers in a manner which will be much appreciated by those who have had up to now to refer for such information to scattered publications in journals and to the *American Society of Testing Materials* ( *A.S.T.M.* ) *Standards*, the *British Standard Specifications* ( *B.S.S.* ), or even the *German Verband Deutscher Electrotechniker* ( *V.D.E.* ), among which, however, there has not been much uniformity in terminology or test specifications.

Chapter 1 is a page and a half introduction by Dr. R. Houwink of Wassenaar ( Netherlands ) on the general nature and significance of the physical and chemical tests and numerical data presented in the succeeding chapters. Dr. J. H. Teeple of *Celenese Corporation of America* treats, in chapter 2, the standard methods of testing the mechanical thermal, optical, chemical and electrical properties of plastics and rubbery materials, explaining clearly and concisely the scientific principles, specific test conditions and inherent limitations involved in each type of tests and furnishing diagrams of testing machinery and tables of typical experimental data. This chapter is an excellent introduction and a helpful guide to the use of the American, British and German standard specifications of individual plastomers and elastomers.

Chapter 3, of particular interest even to practised organic chemists not well-versed in the analysis of high polymeric materials, is contributed by Dr. A. G. Epprecht of Zurich, in whose laboratory several of the analytical methods were either modified or developed. Starting with the method of preparing the material for analysis, since any given plastomer or elastomer consists of many other ingredients than the basic resin or latex, the author details successively the procedure for preliminary examination, systematic analysis along with confirmatory tests and a scheme of qualitative analysis of the more common plastics and rubbers, based on their solubility in organic solvents and resistance to both organic and inorganic chemicals, and finally gives several examples of quantitative analysis of individual polymers and plasticizers. Chapter 4 by Dr. B. B. S. T. Boonstra of the *Rubber Foundation*, Delft ( Netherlands ), contains, in tabular form, valuable numerical data of the properties of elastomers, a large part of which was obtained in his own laboratories, with explanations of the terms and standards prevalent in U.S.A. and other countries. Chapter 5 by J. W. F. van't Wout of Delft and Dr. R. Houwink presents similar tables of numerical data of the properties of plastomers. A detailed table of contents and a comprehensive index complete the volume.

Written by competent authorities and comprising within a comparatively short space a great deal of information on elastomers, the book is recommended, without reserve, to research workers in high polymers. The printing is good and no typographical mistakes were noticed. The only complaint which could be made is that the price is a little forbidding, almost two pence a page, although books of this type are nowadays generally expensive.

L. M. YEDDANAPALLI

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**Plasticity as a Factor in the Design of Dense Bituminous Road Carpets,** by L. W. Nijboer ( Elsevier Publishing Co. Ltd., New York ; Cleaver-Hume Press Ltd., London ), 1948, pp. xv+184. Price 28*s*. net.

AMONG THE ROAD MATERIALS IN USE, BITUMINOUS road mixtures play an important rôle in the development of roads designed to meet the exacting demands of modern traffic with respect to load, speed and safety. For gauging the potentialities of this road material and putting it to the best use, it is essential to have a detailed knowledge of its mechanical properties.

This book deals with methods of investigating the plastic properties of bituminous road materials and the practical applications of bituminous compositions.

The subject-matter is divided into 8 chapters. Chapter I gives a survey of road-building methods through the ages with particular stress on bituminous roads and functions and properties of bitumen. Properties of bituminous mixtures are studied, their mechanical properties analysed and existing testing methods reviewed.

Chapter II deals with the theory of the triaxial shear test method, testing apparatus and preparation of test specimens. The validity of the theory and the accuracy of the method are discussed, and a clear idea is presented of the resistance of bitumin-aggregate mixture to plastic flow as indicated by the 3 physical constants: coefficient of friction, viscosity and initial resistance.

Chapter III on mechanical properties at plastic deformation includes the quantitative assessment of the influence of changes on the composition and properties of mixtures subjected to different test conditions. The comparison of properties under standard conditions is thus rendered possible and the properties of mixes of variable composition can be evaluated when the properties of the standard mix are given. This is an important practical aspect of the subject, as mixes can be designed to meet the traffic requirements of the road.

In chapters IV and V, bearing capacity and compaction by rolling are discussed from the theoretical and experimental viewpoints, and the test apparatus specially developed for these investigations are described.

Chapters VI and VII contain tables and graphs relating to the grading and design of mixtures. These are of great help to research engineers.

In chapter VIII are to be found many observations on mechanical properties of bituminous road mixtures — a new line of approach based on the principles of soil mechanics. This has resulted in the clarification of the rôle of many variables in the bituminous road mixtures. The results obtained are not conclusive enough to formulate specifications for the composition of mixtures to meet any given requirement. These results have, however, led to the conclusion that road carpets might be designed to possess a " Prandtl Bearing Capacity " equal to the inflation pressure of pneumatic tyres. This study was restricted to hot mixtures, and research on cut-back mixtures and emulsion mixtures have still to be carried out.

The author's endeavour has been to bring the knowledge on mechanical properties of bituminous road materials to a level comparable to that of steel and cement concrete in order to obtain the same degree of safety in construction. In this attempt he has succeeded to a great extent, and this book will be of much use both to research workers and field engineers engaged in the construction of bituminous roads.

R. K. N. IYENGAR

**Photography in Crime Detection,** by J. A. Radley (Chapman & Hall Ltd. London), 1948, pp. 186. Price 21*s*.

CRIME PLAYS, AND HAS ALWAYS PLAYED, AN important rôle in the life of mankind since time immemorial. In the current century, it is said, crime has become a refined scientific pursuit. Those who fight against crime are, therefore, facing the need for utilizing all the advances of modern science for detection and prevention of crime.

In this book, *Photography in Crime Detection*, the author has managed to pack, within 186 pages, a staggering amount of information on the various uses of modern photographic processes in detection, analysis and proof of crimes. While keeping within limits of popular appeal, the author has made the chapters intensely practical and valuable alike to the working detective or legal adviser as to the general reader who finds interest and diversion in greedily imbibing detective fiction.

After a theoretical introduction concerning the rôle of photography in crime detection, the author goes on to discuss cases involving mechanical vehicles, identification of persons, criminal cases such as murder, house-breaking, counterfeiting coins, gems and precious stones and betting offences. The examination of documents which are disputed on the ground of forgery and the photographing of such documents by normal and oblique lighting for purposes of comparing handwriting, identification of inks, detecting alterations, erasures or additions, etc., are dealt with in detail in a long chapter covering nearly 30 pages. The use of ultra-violet light and fluorescence analysis of documents, photographing documents, fingerprints, etc., by fluorescent light, ultra-violet light and infra-red light are covered in a very practical manner under separate chapter headings. The place of photomicrography and X-ray photography in crime detection is rightly emphasized and exhaustive practical information given under relevant chapter heads.

On the whole the book is an admirable condensation of very useful and important information on all aspects of the subject, and at the end of each chapter there is a bibliography which helps the reader to refer to other literature for additional information.

No less than 104 reproductions of photographs, actually used in various criminal cases, illustrate the book.

The author deserves special commendation in keeping one main principle in view which he states in his preface : " Every effort has been made to present the material in the simplest manner possible and the examples have been selected for their value in *illustrating the various techniques* rather than for their sensational value ".

Professional men in the police departments and lawyers and judges will find much of real value in this book, while photographers, both professional and amateur, will realize that their fellow-photographers, who do specialized work in police laboratories or in forensic science laboratories, rank among the best exponents of photographic technique and ingenuity.

S. LAKSHMINARASU

# Central Advisory Council of Industries

ADDRESSING the first session of the Advisory Council ( Jan. 24, 1949 ) the Hon'ble Dr. Syama Prasad Mookerjee, Minister for Industry and Supply, reviewed the industrial and economic problems of the country at the present juncture and the measures taken by the Government in this context.

The Government have taken preliminary steps for setting up or investigating into the possibility of establishing a number of important industries. The schemes include : the establishment of new steel works for increasing indigenous steel production by 1 million tons, construction of one or more ship-building yards, manufacture of synthetic petrol from coal, of heavy electrical equipment, of dry core, paper-insulated telephone cables, radio equipment, machine tools, diesel engines, tractors, penicillin, paludrine, sulpha drugs and explosives.

For increasing agricultural production, the Ministry of Agriculture has a number of schemes, the most important of which is the reclamation of waste land by mechanized cultivation. An area of 150,000 acres has already been brought under cultivation. The multi-purpose projects of the Ministry of Works, Mines and Power, when completed, will add over 27 million acres of irrigation to India's existing 48 million, and about 9 million kW. of hydro-electric energy to the present half a million.

Plans to develop rail and road transport, telephone and telegraph and schemes for port development and civil aviation are being executed in some cases and others are under investigation. The State-owned telephone factory, recently established at Bangalore, is designed to make India self-sufficient in regard to her requirements of automatic telephone equipment and the factory near Asansol for the manufacture of locomotives is expected to be completed by the end of 1950.

The policy of the Government for raising the living standards of people is to encourage fruitful enterprises bringing quick results. To encourage cottage and small-scale industries, an All-India Cottage Industries Board has been set up and an all-India policy laid down by the Board is being implemented.

The Hon'ble Minister referred to the work of the Indian Standards Institution in 3 major fields of industry — cotton textiles, engineering and chemicals. He referred also to lack of statistical data and the measures the Government propose to take for the preparation of statutory annual census of manufacturing industries and compilation of monthly statistics.

As regards the nationalization of industries, the Government have assumed primary responsibility for the development of new undertakings in 6 industries other than arms and ammunition, railways and the production and control of atomic energy, viz. coal, iron and steel, air-craft manufacture, ship-building, manufacture of telephones, telegraph and wireless apparatus and mineral oils. The policy of the Government is specific in that the existing undertakings will not only remain with private enterprise for at least 10 years, but that they will be assisted to increase their efficiency and expand their production. The question of their acquisition by the State will be a matter for review at the end of 10 years in the light of the circumstances then prevailing.

The appended table gives the statistics of production for certain important industries.

## PRODUCTION TRENDS DURING 1947-48

*Note* — The 9-hr. day was generally in force till August 1945 as against the 8-hr. day now obtaining

| SER. No. | INDUSTRY | INSTALLED CAPACITY | PEAK PRODUCTION IN BEST YEAR | PRODUCTION IN 1947 | PRODUCTION IN 1948 | REMARKS |
|---|---|---|---|---|---|---|
| 1. | Coal | 30 million tons | 30 million tons ( 1947 ) | 30 million tons | 29·73 million tons | ...... |
| 2. | Steel | 1,264,000 tons | 1,166,200 tons ( 1943 ) | 893,296 tons | 854,000 tons | Decrease in production due to labour troubles, transport difficulties and a temporary breakdown at Steel Corp. of Bengal ( from April to Sept. ) |
| 3. | Salt | 48 million md. ( approx. ) | ...... | 49·6 million md. | 59·3 million md. | Increase is due to the 10-acre concession for small-scale producers and favourable weather conditions |
| 4. | Electric lamps | 14·35 millions | 9·13 millions ( 1948 ) | 7·62 millions | 9·13 millions | Increase due to new units |
| 5. | Dry cells | 142 millions | 122·73 millions ( 1948 ) | 87·91 million | 122·73 millions | Increase due to new units |
| 6. | Motorcar batteries | 268,000 | 110,000 ( 1948 ) | 70,028 | 110,000 | Increase due to new units |
| 7. | Electric motors | 150,000 h.p. | 60,000 h.p. ( 1948 ) | 38,000 h.p. | 60,000 h.p. | Increase due to new units |
| 8. | Transformers | 175,000 kVA. | 80,000 kVA. ( 1948 ) | 23,000 kVA. | 80,000 kVA. | Increase due to new units |
| 9. | Electric fans | 250,000 | 180,000 (1948 ) | 160,000 | 180,000 | Increase due to new units |
| 10. | Cables and wires | Wires ( bare and covered ) 24,350 tons Cable and Flexibles 50 million yd. | ...... | ...... | 6,684 tons wire 23,913 million yd. ( cables, etc. ) | ...... |
| 11. | Electric black copper | 24,000 tons | 6944·1 tons (1946) | 6,889 tons | 3,630 tons | Decrease due to off-take of rubber insulated cable and bare copper wire |

## PRODUCTION TRENDS DURING 1947-48 ( *contd.* )

| Ser. No. | Industry | Installed capacity | Peak production in best year | Production in 1947 | Production in 1948 | Remarks |
|---|---|---|---|---|---|---|
| 12. | Belting | 1,600 tons | ...... | 615 tons | 661 tons | |
| 13. | Diesel engines | 1,200 | 964 ( 1948 ) | 761 | 964* | Increase due to new units |
| 14. | Iron castings | 400,000 tons | 272,454 tons (1948) | 168,333 tons | 272,454 tons | Increase due to improved supplies of raw materials |
| 15. | Machine tools | 3,000 ( 1 graded ) | 2,900 ( 1945 ) | 1,400 | 1,691* | Increase due to improved availability of raw materials and facilities for production |
| 16. | Bicycles | 60,000† | 51,688 †( 1948 ) | 48,827 | 51,688† | Increase due to expanded capacity of existing units |
| 17. | Sulphuric acid | 100,000 tons | 62,550 tons (1945) | 60,000 tons | 80,000 tons | Increase due to new plants |
| 18. | Superphosphates | 75,000 tons (1948) | 21,358 tons (1948) | 5,000 tons | 21,358 tons | Low production in 1947 due to restricted supplies of imported rock phosphate which are now coming on a liberal scale |
| 19. | Caustic soda | 13,500 tons in 1948 | 4,383 tons (1948 ) | 3,314 tons | 4,383* tons | Increase due to certain units coming into production in 1948 |
| 20. | Soda ash | 54,000 tons | 28,200 tons (1948) | 13,624 tons | 28,200* tons | Increase due to improved methods of production and availability of raw materials |
| 21. | Chlorine ( liquid) | 2,100 tons | 1,825 tons (1945) | 1,706 tons | 1,800* tons | Increase due to improved off-take |
| 22. | Bleaching powder | 6,000 tons | 2,836 tons (1948) | 2,550 tons | 2,836* tons | Production likely to increase further if greater supplies of good quality limestone are available |
| 23. | Drugs ( tinctures and galenicals ) | 750,000 gal. | 600,000 gal. (1944) | 500,000 gal. | 522,000 gal. | Gap between capacity and production due to non-availability of the required quantity of spirit |
| 24. | Metals | | | | | |
| | (i) Aluminium | 5,000 tons | ...... | 3,215 tons | 3,354* tons | Increase due to expansion in existing units |
| | (ii) Antimony | 700 tons | ...... | 235 tons | 370 tons | |
| | (iii) Copper | 7,000 tons | ...... | 5,981 tons | 6,031 tons | Normal fluctuation |
| | (iv) Lead | 7,200 tons | ...... | 230 tons | 545 tons | Industry not yet definitely established |
| | (v) Alloys | 30,000 tons | ...... | 15,558 tons | 15,732 tons | |
| | (vi) Semi-manufactures | 50,000 tons | ...... | 22,545 tons | 26,511 tons | Increase due to expansion in existing units. |
| 25. | Rubber ( raw ) | 17,174 tons (1944) | 17,174 tons (1944) | 16,449 tons | 15,400 tons | Fall due to exhaustion of trees by slaughter tapping and stoppage of production on some estates due to unattractive prices |
| 26. | Tyres and tubes | | | | | |
| | (i) For cycles | 4·5 million sets | Tyres 3·34 millions (1948) ; tubes 4·32 millions (1947) | Tyres 3·23 millions; tubes 4·32 millions | Tyres 3·34 millions; tubes 3·82 millions | |
| | (ii) Other than cycle ( motor, giant, etc.) | 1 million sets | Tyres 0·83 millions (1947) ; tubes 0·80 millions (1947) | Tyres 0·83 millions (1947); tubes 0·80 millions | Tyres 0·78 millions ; tubes 0·74 millions | |
| 27. | Alcohol | | | | | |
| | (i) Industrial | 20 million gal. | ...... | 4·82 million gal. | 4·9 million gal. | Increase due to additional plant |
| | (ii) Power | ...... | ...... | 2·20 million gal. | 3·54 million gal. | |
| 28. | Raw cotton | Restrictions are in force over cultivation in view of food position | | 28 lakh bales | 28* lakh bales | |
| 29. | Cotton textiles | | | | | |
| | (i) Yarn | 10,158,909 spindles ( including 35,000 spindles installed during 1948 ) | 1,642,831,000 lb. (1943) | 1,314,524,000 lb. | 1,442,342,000 lb. | Increase due to better use of capacity in mills during 1948 |
| | (ii) Cloth | ...... | 4,735,750,000 yd. (1944) | 3,816,088,000 yd. | 4,337,502,000 yd. | |
| 30. | Woollen manufactures ( excluding cottage industries ) | 30 million lb. | 30 million lb. (1945) | 24 million lb. | 23,668,074 lb. | |
| 31. | Rayon weaving | 150,000 hand looms | 180 million yd. (1937) | 85 million yd. | 114 million yd. | Increase due to improved supplies of yarn |
| 32. | Rayon weaving | 15,000 power looms | 38 million yd. | ...... | ...... | |
| 33. | Cement | 2,115,000 tons | ...... | 1,441,335 tons | 1,516,226 tons* | Increase due to expansion of an existing unit |
| 34. | Sugar | 1,400,000 tons | 1 million tons* (1948) | 925,000 tons | 1 million tons* | Increase due to higher price for sugarcane and sugar during 1948 |
| 35. | Paper and boards | 110,000 tons | ...... | 93,090 tons | 99,765 tons | |
| 36. | *Vanaspati* | 220,000 tons | 145,000 tons* (1948) | 95,000 tons | 145,000 tons | Increase due to new units |
| 37. | Tea | 551 million lb. | 551 million lb. (1947) | 551 million lb. | 550 million lb. | Decrease due to climatic and labour conditions |
| 38. | Coffee | 15,040 tons (1947-48) | 25,200 tons (1945-46) | 15,040 tons (1947-48) | 17,000 tons (1948-49; estimated ) | Fall due to climatic conditions. Coffee has a 3 to 4 year cycle of good, medium, and poor crops |

* Approximate figures. † Complete bicycles + spare parts equivalent to complete units.

PRODUCTION TRENDS DURING 1947-48 ( *contd.* )

| SER. No. | INDUSTRY | INSTALLED CAPACITY | PEAK PRODUCTION IN BEST YEAR | PRODUCTION IN 1947 | PRODUCTION IN 1948 | REMARKS |
|---|---|---|---|---|---|---|
| 39. | Cigarettes | 30,000 millions | 9,658·16 millions | 18,879·77 millions | 29,658·16 millions | Increase due to improvement in production and larger availability of raw materials |
| 40. | Matches | 800,000 cases ( 1 case = 50 gross boxes of 50 sticks each ) | 618,000 cases (1947) | 618,000 cases | 499,586 cases | For three-quarters only. |
| 41. | Leather ( production of organized units ) | | | | | |
| | Buffalo hides ( veg. tan ) | 2,000,000 | …… | 800,000 | 979,000 | Decrease is due to shortage of tanning materials and transport |
| | Cow hides ( chrome )<br>Buffalo hides E.I. tanned | 3,000,000 | …… | 1,200,000 | 1,087,000 | |
| | Cow Hides Tanned | 9,300,000 | …… | 6,420,000 | 4,602,300 | |
| | Goat skins<br>Sheep skins | 19,080,000 | …… | 17,500,000 | 15,250,000 | |
| 42. | Plywood | 63 million sq.ft. in 1948 | 50 million sq.ft. in 1944 | 28·6 million sq.ft. | 38·63 million sq.ft. | Increase due to additional units |
| 43. | Soap | 250,000 tons | 190,000 tons (1948) | 80,000 tons | 190,000 tons | Marked improvement due to rapidly increasing availability of raw materials |
| 44. | Glass | 150,000 tons | 120,000 tons (1945) | 80,000 tons | 70,000 tons | Decrease due to shortage of raw materials and fuel |
| 45. | Ceramics | 30,000 tons* | 25000 tons (1945) | 20,000 tons | 23,000 tons* | Increase due to additional plants |
| 46. | Refractories | 225,000 tons (1948) | 185,511 tons (1948) | 175,376 tons | 185,511 tons | Increase due to 3 firms going in for tunnel kilns |
| 47. | Enamel ware | 25 millions | …… | 8·532 millions | 6·763 millions | Fall despite increased capacity due to shortage of raw materials, particularly mild steel sheets and difficulties of transport |
| 48. | Insulators | 4,200,000 | …… | …… | 2,529,000 | |
| 49. | Abrasives | 121,680 reams | 40,600 reams ( 1947 & 48 ) | 40,600 reams | 40,600 reams | |

* Approximate figures.

# Obituary — Prof. Birbal Sahni ( 1891-1949 )

IN THE DEATH OF PROF. BIRBAL SAHNI ON April 9 at Lucknow, India has lost one of her eminent sons, and science a brilliant savant.

The 58-year old scientist was born at Bhera, Punjab, on 14th November 1891 and was educated at the Government College, Lahore and at the Emmanuel College, Cambridge. He was appointed Professor of Botany, University of Lucknow, in 1921, and this position he filled with conspicuous distinction all these years. He was the Founder and Honorary Director of the Institute of Palaeobotany, the foundation-stone of which was laid just a week before his death.

Among his numerous publications may be mentioned an excellent text-book of botany, original contributions on extinct plants, fossil flora and their geological bearings, and Indian numismatics. He was the Editor of the Lucknow University Studies, and Palaeobotany in India. He was a brilliant teacher esteemed alike by his numerous students, colleagues and friends both for his personal charm and scientific eminenece.

He was elected a Fellow of the Royal Society, London, in 1936, President of the 27th Session of the Indian Science Congress, Madras ( 1940 ), and a non-official member of the Indian Delegation to the Royal Society Scientific Conference held in London in 1946. He was invited to preside over the Palaeobotany Section of the 7th International Botanical Congress to be held at Stockholm in July 1950. Prof. Sahni was awarded the Barclay Medal of the *Asiatic Society of Bengal* in 1936, and the Nelson Wright Medal of the *Numismatic Society of India.*

Prof. Sahni dedicated his private funds, his immovable property, a reference library and his valuable fossil collections for the promotion of research in fossil botany at the Institute of Palaeobotany at Lucknow. The cutting short of a fruitful career, at a time when the prospect of further activity on a large scale was opening out, will be deeply felt by all those interested in the progress of science in India.

# NOTES & NEWS

## A New Horizontal Electron Microscope

A HORIZONTAL ELECTRON MICROSCOPE with several distinctive features has been designed in the University College of Science, Calcutta (*Ind. J. Phy.*, 1948, **22**, 497). The instrument is essentially a research unit, flexible in design and highly suited for investigations on electro-optical problems. The different elements of the unit are mounted on 2 stainless steel rods held in position by brass sleevings which can slide over the rods. It is thus possible to dismantle any part of the microscope without disturbing the rest. The distance between any 2 elements can be varied, and it is also possible to interpose an extra element between any 2 of the existing ones. Each microscope element is approachable from all sides, and the image formed on the fluorescent screen can be demonstrated to a number of people simultaneously. The instrument is designed for a maximum electronic magnification of 20,000 dia.

The electron gun, the condenser, the objective and the projector lenses are supported on separate carriages consisting of 4 pairs of horizontal brass plates. Any of the pairs of brass plates can slide together in a horizontal plane perpendicular to the axis of the microscope on a pair of stainless steel guide rods fixed to the frame of the instrument. The upper plates supporting the microscope elements can also be raised or lowered with respect to the lower plates by a set of screws. These 2 motions at right angles to the optical axis can be given to any element of microscope. By means of transmission gear arrangement, the operator, sitting at the control table near the final fluorescent screen, can move any of the lenses or the gun for proper alignment. Each of the 3 lenses has 4 levelling screws by means of which the lens may be slightly inclined to the axis so as to allow for any asymmetry of the pole pieces. In addition to lateral motions, the gun can be slightly tilted about the horizontal and vertical axes.

The length of the microscope column from the filament tip to the objective is 59 cm. and the length from the objective to the fluorescent screen is 79 cm. For evacuating the microscope column, an oil-diffusion pump and a *Cenco Hypervac* 20 are used. The mechanical pump is housed in a specially designed underground chamber a little distance away from the microscope in order to reduce noise and vibration. A thermo-couple gauge measures the fore-vacuum while the high vacuum within the microscope is indicated by an ionization gauge.

The electron gun assembly is demountable and is made vacuum tight by means of the rubber gaskets and gasket tightening screws. For a change of filament, the filament unit together with the cathode shield can be taken out.

The object stage can be rotated through a small angle and the tilting of the stage for stereophotography purposes is also possible. 2 viewing ports provided on the upper side of the specimen chamber allow a view of the specimen stage through all operations. 4 hydraulic sylphon bellows, fitted at 90° to each other, provide movement for the object stage in 2 perpendicular directions at right angles to the optical axis. By compressing and expanding the hydraulic sylphons it is possible to move the stage in a plane perpendicular to the microscope axis and thus explore different parts of the specimen. It is also possible to move the stage while looking at the image on the final fluorescent screen. A unit is provided on the control desk with both coarse and fine adjustments, so that the specimen can be placed accurately in any desired position.

The specimen changing is accomplished by means of an air lock.

The photographic unit consists of a plate magazine holding 20 photographic plates and is demountable. The plates are moved by a pressure pad, the pressure being maintained by the vacuum in the unit. A mechanism is also provided by means of which the plate carrier and the shutter mechanism are swung out of the path of the electron beam and the total area of the fluorescent screen can be utilized for visual observation of the micrograph.

The microscope filament is heated by r.f. current of about 150 kc./s. This reduces the problem of electrostatic shielding and simplifies high voltage insulation.

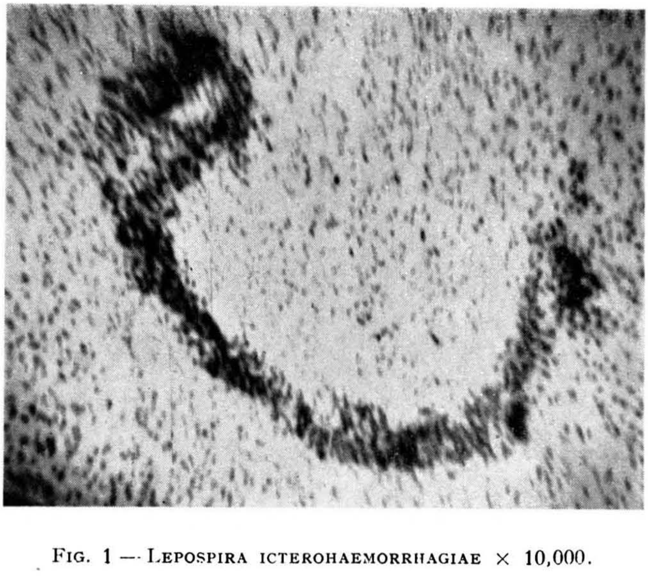

FIG. 1 — LEPOSPIRA ICTEROHAEMORRHAGIAE × 10,000.

## Mounting Medium for Textile Fibres

AN IMPROVED PERMANENT MOUNTING medium for textile fibres is described ( *Textile Res. J.*, 1948, **18,** 756 ). A colourless permanent mount with the desired refractive index can be made by the combination of a synthetic resin of a moderately low refractive index with a compatible plasticizer of a much higher index. By varying the composition of the mixture, it is possible to adjust the refractive index to a value suitable for a particular fibre. A mounting medium with a refractive index that is approximately half-way between the maximum and minimum indices of common fibres has been found to be most useful.

*Iso*butyl methacrylate polymer ( *Du Pont* ) of refractive index 1·48 and Aroclor 1242 ( *Monsanto* ) of refractive index 1·62 are, respectively, the resin and the plasticizer used for the mount medium. Both materials are colourless and quite miscible with xylene. The proportions of all the 3 materials are adjusted until a viscous liquid with a refractive index 1·52 is obtained. This index is half-way between the indices of cotton or wool and acetate rayon.

The following is a formula for a permanent mounting medium ( after heating ) with a refractive index of 1·516 and 1·520 :

| | |
|---|---|
| Xylene ( filtered ) | 21 c.c. |
| Aroclor 1242 | 9 c.c. |
| *Iso*butyl methacrylate polymer | 18 gm. |

The *iso*butyl methacrylate polymer should be finely ground and free from lumps. Into the filtered xylene Aroclor 1242 is first added and then the methacrylate polymer and the contents stored. The mix is allowed to stand overnight until all the bubbles produced due to stirring have passed off. The resulting colourless liquid has a viscosity very close to that which is usual with solutions of Canada balsam. The material is used in the same manner as Canada balsam.

A slight excess of material is employed and the slide after preparation should be heated in an oven at 70°C. for at least 2 hr. The excess material around the cover glass can be removed by a razor blade.

The 2 tables below give the approximate refractive indices of various mixtures, and data for obtaining photomicrographs of fibres using the mixtures.

TABLE I — APPROXIMATE REFRACTIVE INDICES OF VARIOUS MIXTURES

| *Iso*BUTYL METHACRYLATE POLYMER gm. | AROCLOR 1242 c.c. | XYLENE c.c. | REF. INDEX OF FINISHED MOUNT |
|---|---|---|---|
| 18 | ... | 30 | Between 1·495 and 1·500 |
| 18 | 3 | 27 | „ 1·495 and 1·500 |
| 18 | 6 | 24 | „ 1·505 and 1·510 |
| 18 | 9 | 21 | „ 1·515 and 1·520 |
| 18 | 12 | 18 | „ 1·520 and 1·525 |

TABLE II — DATA FOR PHOTOMICROGRAPHS OF FIBRES

| FIBRE | REF. INDEX | MOUNTING MEDIUM | REF. INDEX | VISIBILITY |
|---|---|---|---|---|
| Cotton | 1·56 | Canada balsam | 1·54 | Poor |
| Cotton | 1·56 | Formula with 9 c.c. Aroclor 1242 | 1·515-1·520 | Good |
| Cotton | 1·56 | Formula with 3 c.c. Aroclor 1242 | 1·495-1·500 | Good |
| Acetate rayon | 1·48 | Formula with 3 c.c. Aroclor 1242 | 1·495-1·500 | Very poor |
| Wool | 1·55 | Canada balsam | 1·54 | Poor |
| Wool | 1·55 | Formula with 9 c.c. Aroclor 1242 | 1·515-1·520 | Good |
| Wool | 1·55 | Formula with 3 c.c. Aroclor 1242 | 1·495-1·500 | Good |

## Manufacture of Anhydrous Hydrofluoric Acid

THE CONSTRUCTION AND OPERATION of the first plant to produce anhydrous hydrofluoric acid by a continuous process at the *Imperial Smelting Corp.*, Avonmouth, England, are described as a landmark in chemical history ( *Ind. Chem.*, 1948, **24,** 801 ). The anhydrous acid is for the first time transported in rail-tank cars. The plant is producing 6 tons of 80 per cent acid per day.

The major problem in the manufacture of anhydrous hydrofluoric acid is corrosion, and the process is, therefore, of particular interest from the point of view of materials of construction employed. At strengths of 80 per cent or over of the acid, little difficulty is experienced from corrosive attack so that the section of the plant in which acid at these high concentrations are handled can be constructed of mild steel. Below 80 per cent corrosion is very severe and it has been necessary to employ plastics in various forms of Karbate, a form of non-porous carbon.

Fluorspar, a flotation product of acid grade and mostly passing −200 mesh and analysing to 97·0 per cent $CaF_2$, and less than 1 per cent $SiO_2$ and $CaCO_3$ and concentrated sulphuric acid are heated in a reaction kiln at about 430°C. and the gas evolved is first absorbed in 80 per cent acid and subsequently in 60 and 30 per cent acid. The reaction kiln is a cylindrical mild-steel structure ( 6′ dia. × 40′ long ), supported on 2 steel tyres running on rollers and rotated at about 1 r.p.m. All extraneous air is sealed off from the kiln by means of pressure oil seals.

The gas stream is washed in counter-current with sulphuric acid in the " drip acid tower " constructed of mild steel and then absorbed in 3 stages as described above. The absorption towers are of mild steel and of the disc and plate type and are cork-lagged. The circulating acid is cooled by passing it through brine coolers of mild steel in the first stage of absorption. The circulating acid is diluted in a bronze pot. The second absorption tower containing 60 per cent acid comprises a Keebush tower, a circulating pump of Karbate and water cooler constructed of Karbate pipes. Keebush has been selected as the material for the tower, as it is capable of withstanding 70 to 75 per cent acid satisfactorily. The 30 per cent acid tower is similar to the 60 per cent one but is made of Plastimold.

Anhydrous acid is obtained by distilling 80 per cent acid in a simple bubble-cap type distillation column of copper. The vapour stream is cooled in a mild-steel brine cooler and stored in mild-steel tanks.

The anhydrous acid is delivered directly to rail cars ( 6·5 tons capacity ) and is effected by means of compressed air.

For pipe lines Saran has been employed and in some cases polythene has shown considerable promise. 60 and 30 per cent acids are stored in tanks lined with Neoprene. For the concentrated acid, valves with phosphor bronze bodies and Ilium seats have proved adequate. A satisfactory pump-packing material has been the only difficulty so far.

## Measurement of Radioactive Isotopes

THE DETERMINATION OF THE NUMber of radioactive atoms in a sample of a radioactive isotopes has become a matter of importance, because of their expanding use in biological and medical studies, and in the clinical treatment of diseases. Uniform quantitative results can be obtained only by using procedures that will give the same result whenever the determination is made. The *National Bureau of Standards*, U.S.A., has, therefore, issued standard samples of radio-isotopes whose radioactivity has been accurately determined and is planning to issue samples of additional radio-isotopes as they become available ( *J. Franklin Inst.*, 1948, **246**, 419 ).

Two factors involved in the preparation and use of the samples are : (i) methods and standards that are correct in principle must be used ; and (ii) a reasonable degree of accuracy must be attained in all steps of the process both of producing and of using the standards.

Most of the difficulties in making quantitative determinations of radio-isotopes can be eliminated if standard sources of the isotope are available. Such a standard source consists of a preparation of the isotope in a form convenient for use with the detector of radiation and for which the disintegration rate measured is the same as that from which the standard is made. Reliable results may be secured by observing only a few simple precautions. Isotopes which have short half-life periods and a known disintegration scheme may be measured with a fair accuracy by means of $\gamma$-ray standards, if it is known that a $\gamma$-ray or a positron is emitted for every disintegration, and if the maximum energies of $\gamma$-ray specific of the standard and of the specimen are not too widely different. In the case of those isotopes for which a disintegration scheme is lacking, or for which the mode of disintegration does not permit measurement of disintegration rates, an alternative method of comparing the activities of sources is available if the isotope emits $\gamma$-rays. This method is, however, limited to comparisons of the same isotope. An ionization instrument properly designed to measure Roentengen units will satisfy the requirements for a standard instrument. To determine the strength of a radioactive source the Roentengens per unit time must be measured at a standard distance. On the basis of these considerations, the *Bureau* scientists have proposed that the unit of time be 1 hr. and the distance 1 m., giving as a unit for comparison of $\gamma$-ray sources the *Roentengen per hour at a meter* ( *r.h.m.* ).

The name *Rutherford* ( *rd* ) has been proposed for that quantity of radio-isotope which disintegrates at the rate of a million disintegrations per sec. This unit which is smaller than the *Curie* can be specified exactly and independently of natural constants such as the rate of decay of radium, which is the basis of the *Curie*. The *Bureau* has adopted an arbitrary value of $3{\cdot}700 \times 10^{10}$ disintegrations per sec. for the *Curie* when applied to isotopes other than the members of the radium family. The use of *Rutherford* in data presupposes that a disintegration rate has been measured and that this rate is expressed in disintegrations per sec. This procedure removes all confusion regarding units and renders data from several laboratories comparable on an absolute basis.

## Radio-cardiography

A NEW METHOD FOR STUDYING blood flow through the chambers of the heart has been developed by recording graphically the passage of radioactive substances through the cardiac chambers with the aid of a specially constructed, ink-writing Geiger-Muller counter ( *Science*, 1948, **108**, 340 ). The procedure makes possible the investigation in human beings of certain haemodynamic functions not previously accessible to study.

The method consists in placing a carefully shielded Geiger-Muller counter over the pericardium, rapidly injecting 0·1 to 0·2 mc. of radio-sodium ( $Na^{24}$ ) into one of the antecubital veins, and recording the counts by means of the newly devised direct-writing counter. Of the 2 waves recorded on the radio-cardiogram, the first wave relates to the entrance of the blood into superior vena cava, right auricle and the right ventricle and the pulmonary circulation and the second wave represents the period during which the left cardiac chambers are being emptied of the labelled blood.

The tracings taken in subjects with enlarged hearts and failure do not appear to differ materially from those in cardiac enlargement without failure. This observation arouses interesting speculation concerning the role of the heart in failure. Other observations of interest emerging from these studies are : the rate of venous return from a lower limb is much slower than that from an upper extremity, which helps to explain the greater tendency of venous thrombosis to develop in the lower extremities. The recording counter has also been used to estimate the time required for a substance to be absorbed from the side of a local infection ; in preliminary studies on congenital heart disease ; and in revealing certain sources of error in the usual clinical methods of determining circulation time and provides a more accurate method of determination.

## Improved Seismic Method of Locating Oil

AN IMPROVED METHOD OF locating underground petroleum deposits has been reported by Dr. Thomas C. Poulter of the *Institute of Inventive Research*, San Antonio, Texas. The method employs " shaped charges " of explosive compositions which are set off above the ground, thus eliminating the cost of drilling shot holes. The " shaped charge " is based on the principle that if a hollow is made in an explosive cartridge on the side toward the object to be blasted, the effect is greatly increased. The method can be used in regions where boring would be exceedingly difficult as in mountainous country or in a search for oil under sea.

Charges are set close to the ground and spread in a hexagonal design. Depending upon the type of record sought, comparatively light charges are placed from 5′ to 85′ apart and detonated simultaneously. The procedure may be used to produce an essentially flat wave front of low amplitude over a relatively large area (*Science Newsletter*, 1948, Nov. 13, p. 307).

### Growth Factor in Coconut Milk

THE STRIKING NUTRITIONAL effect of coconut milk on the growth of explants of carrot root has been demonstrated both in liquid media and on nutrient agar (*Science*, 1948, **108**, 655). This nutritive fluid endosperm has already found application in the culture of bacteria, fungi, orchids and immature plant embryos. It has been claimed by a number of workers that it contains both a specific factor for plant embryo development and a thermo-stable general growth factor, probably indole-3-acetic acid. The evidence, accumulated so far, favours the presence of an unidentified active principle in coconut milk which can foster rapid growth of carrot cultures. Qualities which promote growth of carrot cultures can be demonstrated in heat-sterilized, filtered, water-clear preparations of coconut milk (*p*H, 5·6-5·9) obtained from mature nuts. Other experiments have shown that 1 per cent by volume of coconut milk added to an otherwise complete organic and inorganic nutrient medium causes a marked increase in growth; at about 15-20 per cent there is an optimum with marked decrease at higher concentrations. No growth occurs in coconut milk alone. In the presence of indole acetic acid alone a mean increase in weight observed was 8·2 mg. in 21 days and with added coconut milk a mean weight of 184 mg. was observed.

The active principle of coconuti milk is not a constituent of the ash; nor is it any of the other known growth factors: thiamin, niacin, pyridoxine and glycine. It seems also to be different from any of the normal constituents of yeast, malt, tomato, carrot or liver extract when used at *p*H 5·6. Though coconut milk may contain indole acetic acid, it is clear that this is not the factor responsible for increased growth. The substance is stable to prolonged autoclaving and, since its activity decreases on dialysis, it has relatively small molecules.

The work done so far point to much wider problems than those which merely affect growth, and the following points suggest themselves:

(i) An outstanding difference between the carrot cultures which do not grow in the absence of coconut milk, or which sluggishly expand in the presence of indole acetic acid, and those which burst into very rapid growth must surely be in their ability to harness their respiratory energy in protein synthesis, salt and water uptake, i.e. to maintain the process in the cells which require the use of energy. It would be reasonable, therefore, to seek this coupling mechanism in some effect of the coconut milk which may promote not only carbohydrate breakdown, but also the linkage of respiratory energy to useful work; (ii) the possibility exists, however, that the active principle in coconut milk is not peculiar to carrot tissue and is at least specific for cells which contain carotenoids.

The literature on vitamin A also suggests a possible connection between a constituent of the coconut milk and vitamin A or carotene. Apparently coconut milk, as a partial source of protein in the basal vitamin A deficient diet, causes the vitamin A to be more effectively used when it is supplied. It has been suggested that there is a factor in coconut milk which, combined with vitamin A, makes the vitamin more effective or which facilitates the transformation of carotene to vitamin A.

### Geochemical Prospecting

AMONG THE NEWER METHODS OF investigation to discover mineral deposits is the geochemical method which is being increasingly employed in Australia (*Chem. Age*, 1949, **60**, 163). Australia is particularly suited to such methods of investigation because the mature residual soils of parts of the country and possibly some non-residual soils are likely to contain geochemical anomalies in mineralized areas, such anomalies being related spatially and genitically to economically important ores. It is likely that these methods will be most useful in areas where neither geological nor geophysical methods can indicate ore bodies.

Most of the claims in this field have come from Russia where deposits of tungsten, molybdenum and tin were claimed to have been discovered by examining spectrographically soils up to 9′ thick. A geochemical unit in U.S.A. is engaged in field, laboratory and greenhouse research, chiefly on copper and lead contents of soils, plants and waters in areas of known mineralization.

The poorly "dispersed" elements such as tin, lead, mercury, etc., may produce well-defined dispersion halos at no great distance from their source, both in semi-arid and in humid environments. The "dispersed" elements such as zinc, nickel and others may form large but poorly defined halos; in areas of ample rainfall these "dispersed" elements may travel long distances.

Four methods are employed: (1) the soil samples are tested by colour reactions to indicate abnormal concentrations; (2) spectrographic analysis of soils, which has proved of limited value; (3) the role of plants as guides to prospecting which takes into account the fact that certain plants grow best when there is a high concentration of a metal, and the accumulation by plants in their tissues of metals such as zinc, nickel, copper and cobalt in the leaves and lead and arsenic in roots; the available experience in U.S.A. indicates that plants, in general, are not precise indicators of geochemical anomalies; (4) testing of surface and ground waters in order to locate unusually high concentrations of heavy metals. The examination of water as a procedure of general exploration in a virgin country may be a great time-saver.

### A Rapid Laboratory Method for DDT

DDT, AS PREPARED BY THE Bayer condensation method using chloral and fuming sulphuric acid, is expensive and requires complicated apparatus. Both chloral and fuming sulphuric acid are unpleasant and hazardous to handle, especially by inexperienced workers and students. A short and simple method for preparing small quantities of DDT without the use of either chloral or fuming sulphuric acid has been worked out to overcome the above drawbacks (*Science*, 1948, **108**, 339). The entire process can be carried out within one laboratory period and has been used by graduate students taking courses in insecticides.

The method is based on the theoretical reaction of 1 mole of chloral hydrate and 2 moles of chlorobenzene (sp. gr., 1·107) in the presence of about 4 to 5 times their combined volumes of concentrated sulphuric acid.

17 gm. of chloral hydrate crystals and 23 c.c. of chlorobenzene are placed in a glass-stoppered, 500 c.c. pyrex reagent flask and kept in an electric oven at 60°-70°C. for about 20-30 min. with occasional shaking or until all the crystals have dissolved. The contents are cooled to room temperature and about 180 c.c. of concentrated sulphuric acid added, the flask stoppered and contents vigorously shaken until precipitation starts. This operation requires about 1 hr. The contents are let stand for 15 min. with an occasional shaking or until precipitation is complete. The mixture is poured into a glass jar containing about 4 litres of cold tap water and allowed to stand for 15 min. or until the solids have settled down. The precipitate is filtered through 3 layers of cheese cloth, washed several times with water and transferred to a glass jar or wide-mouthed bottle to which 50 c.c. of either 2 per cent sodium carbonate or 4 per cent sodium bicarbonate solution are added to neutralize the acidity. The product is finally filtered over a Buchner and washed with distilled water until the filtrate is neutral to litmus. The residue is triturated with 100 c.c. of ethanol in a mortar and pestle for about 10 min. and filtered in a Buchner and dried in an electric oven at 70°-75°C. for about 2 hr. or until all the alcohol has volatilized. An average yield of about 16 gm. of pure DDT is obtained and analyse to an average of 96·82 per cent *p,p*'DDT with an m.p. ranging from 106°-108°C.

## Fluorine Substituted DDT

ANOTHER INSECTICIDE, DIFLUORO-diphenyl-trichloroethane (DFDT), a compound obtained by substituting 2 chlorine atoms in DDT by fluorine, has proved to be better than DDT in killing house flies and certain other insects. Extensively employed in Germany during the war, the new insecticide is less toxic to warm-blooded animals and fish. Although it kills fewer insect species than DDT, it has quicker knock-down power against those it is effective (*Sci. Newsletter*, Jan. 1, 1949, p. 9).

## Estimation of Rotenone

A RAPID AND SIMPLE METHOD for evaluating the toxicity of *derris* roots is based on the absorption of light by the toxic constituents of the roots dissolved in acetone and measuring it in a spectrophotometer at 360 mμ (*J. Agr. Res.*, 1948, **77**, 271).

The method is as follows: 1 gm. of *derris* root, ground to pass an 80-mesh sieve, is taken in a 125 c.c. Erlenmeyer flask with 50 c.c. of purified acetone. The contents are swirled for a few seconds and set aside for 1 hr. or longer with occasional shaking. The contents are cooled in an ice-box for 30 min. and then rapidly filtered taking every precaution to reduce the evaporation of the solvent. When the filtrate reaches room temperature, a 10 c.c. aliquot is made up to 100 c.c. in a volumetric flask using pure acetone. The percentage of transmittance at 360 mμ is measured in a spectrophotometer using pure acetone as reference solution. The rotenone equivalent is determined from a standard curve prepared from transmittance values of 2 biologically assayed *derris* roots prepared as above.

When acetone extracts of *derris* samples are adjusted to equal rotenone contents and the percentage transmittance determined, there is close agreement between the known content of other toxic constituents (rotenoids) and the transmittance values. Likewise, transmittance values of graded rotenone solution in acetone determined at 360 mμ follows the Beer's-Lambert law. The transmittance values of total acetone extracts of *derris* roots are in close agreement with the rotenone equivalents as determined biologically on house flies. Transmittance values of *Lonchocarpus* root do not correlate with the biological rotenone equivalent indicating that some of the toxic constituents in these roots are different from those in *derris* roots.

## Humidity Test Apparatus

A "DIVIDED FLOW, LOW TEMPERATURE test apparatus", developed at the *National Bureau of Standards*, now makes available an instrument for research, calibration and testing of hygrometers at temperatures below freezing (*J. Franklin Inst.*, 1948, **246**, 421). The use in recent years of radiosondes for the meteorological sounding of the atmosphere has created a marked need for information on the performance of the humidity-sensing element in the radiosonde under conditions of flight.

The function of the humidity test apparatus is to produce air of known relative humidity at temperatures from 0° to −40°C. In this instrument, a current of dry air is divided into 2 streams, one of which is maintained dry while the other is saturated with respect to ice; finally the two are recombined. A proportioning value divides the flow of moisture-free air in a known ratio. One part passes through a saturator, containing ice-trays till saturated, then mixed in a mixing chamber with the other dry part and finally allowed to exhaust through a test chamber into the atmosphere. The saturator, mixing chamber and the test chamber are kept immersed in a constant temperature bath. The hygrometer to be tested is inserted into the test chamber for testing. The overall performance of this apparatus has been established by a series of gravimetric moisture determinations. For the temperature range of 0° to −40°C. and for air velocities up to 1500′ per min., this instrument produces air whose relative humidity is known within 3 per cent.

While particularly suited for investigation and calibration of hygrometers, psychrometers and development apparatus at temperatures below 0°C., this equipment may find further application in general research and testing where air of a known low dew-point or moisture content is desired. With such apparatus, devices placed in the test chamber can be subjected to discrete and almost instantaneous changes in relative humidity. It may also find useful application in refrigeration and air-conditioning industries.

## Flax Retting with Aeration

THE NORMAL FLAX RETTING process, which is anaerobic, gives rise to a large volume of polluting waste waters which present a serious disposal problem. In a factory with a capacity of 32 tons of flax straw per week, an average of 40,000 gal. a day of waste waters are discharged from the tanks which is equivalent to about 170,000 gal. of settled domestic sewage. Further, the disposal of waste waters from retting present far greater difficulty than would be encountered in the disposal of an equivalent volume of domestic sewage because of their acid nature and the polluting matter is in colloidal dispersion and cannot be removed by simple sedimentation. *Technical Paper No.* 10, published by the *Water Pollution Research Laboratory, D.S.I.R.,* London (*H.M.S.O.*, price 3*s*.), gives an account of the investigation, of which the final stage was the development of a new process in which the retting liquor is aerated during retting and re-used.

The process may find application in the retting of other fibrous materials like jute, coir, etc.

In laboratory-scale experiments, it was shown that the waste waters from anaerobic retting treatment could be treated economically to give an effluent of a high quality. In small-scale experiments good results were obtained when successive batches of flax were retted in the same liquor which was treated continuously by passage through percolating filters or through a separate aeration tank or which was aerated in the retting tank itself during retting. The fibre produced by all the 3 methods was of good quality; the method of using biological filtration produced a fibre of a better quality than that from other processes, but the area of filters required would be large. For a rettery with 4 tanks, each holding 4 tons of deseeded flax, an area of about 4,050 sq. ft. of filters, 6′ deep, would be necessary.

During all the stages of the work, bacteriological examinations were made of the retting liquor and of the flax during retting, and much information on the bacteria responsible for retting was obtained. It was found that, when the liquor was aerated, bacterial growth resulted largely in the organic matter being oxidized away instead of being converted to acids as in the anaerobic process. Conditions in the flax stems themselves, however, were still anaerobic and the organisms mainly responsible for retting, even under conditions of considerable aeration, were found to be spore-forming anaerobes.

A number of flax factories in England are operating plants employing retting with aeration. The tanks usually are 4 in number, of 4 tons capacity of deseeded flax each, rectangular and, in its internal dimensions, 19′ 8″ long, 13′ wide and 9′ 3″ deep. The top is open and is fitted with transverse cross beams 8″ apart to retain flax. On the floor are diffuse units through which air is bubbled through the retting liquor, and above are wooden grids on which flax is packed. Equipment for injecting steam to heat the liquid in the tank is provided.

A storage tank is provided, a third more in capacity than that of the retting tank, into which liquor from the retting tanks can be run in. The heating here is by steam injection.

Compressed air is supplied to the tanks by 2 compressors, each driven by a 6 h.p. electric motor and rated to deliver 100 cu. ft. of air per min. against a pressure of 4·5 lb. per sq. in. Each diffuser unit has a separate air pipe and the distribution of air can be adjusted by a valve on each pipe.

Before the start of a ret, water is run into the tank to a depth of about 6″ above the diffuser plates, and the air supply is turned on and adjusted to give even distribution. Beets of deseeded flax are packed into the tank in 2 tiers. Fresh water, which has been heated in the storage tank to 30°C., is run into the retting tank to within about 3″ of the top and the air supply is increased during the filling to about 30 cu. ft. per min. The temperature of the liquor in the tank is maintained at about 30°C. and warm fresh water or retting liquor is added during the ret to maintain the level of liquid in the tank.

Retting is generally complete in 2 to 3 days at 30°C. Samples are withdrawn at frequent intervals after the first 24 hr. for examination; when the fibre is found to be separating from the straw, samples should be withdrawn at least every hour for examination.

When a ret is finished, the liquor is run off into the storage tank, leaving enough in the retting tank to cover the diffuser plates and retted flax removed. The retting tank is then packed with fresh straw, the liquor is pumped back from the storage and fresh water is added to make up the original amount.

Samples of retting liquor are analysed 3 or 4 times a day for the acidity or alkalinity. If acidity increases, the air supply is increased; if the liquor becomes alkaline, the air supply is reduced.

Aeration should be maintained between rets so long as the diffuser plates are covered with retting liquor. At least once a week all liquor should be run off and the wooden grids removed so that material deposited from the flax can be forked and hosed out.

The bulletin has a useful chapter on review of literature on retting of flax and on the various aspects of retting and methods employed in several countries. The 3 appendices relate to bacteriological investigations and methods of analysis.

## Continuous Steel-casting

ONE OF THE FAR-REACHING developments in steel-casting, which promises to revolutionize the steel industry, is the continuous steel-casting method developed by Prof. Sachs, Director, National Metallurgical Laboratory, prior to his coming to India. The new method dispenses away with the reheating and rolling process of the conventional process and saves space and expense.

In this process the steel is poured into an oval-shaped mould, where it starts to solidify uniformly and quickly. The steel flows from the mould through a sleeve-like insulated chamber below the mould, which equalizes the temperature of the steel as it further hardens. As the metal is withdrawn, it is cut into billet-size length by an automatic oxy-acetylene torch. The steel, then in a semi-finished shape, is ready to be sent to the finishing mill.

One of the most difficult tasks in the development of the process was finding the correct oval shape for the mould. By the use of zinc and aluminium alloys the behaviour of steel in the oval mould was modified and the correct mould shape facilitating the freezing of steel was determined.

## Improved Baker's Yeast

A NEW PROCESS FOR THE MANUfacture of baker's yeast, the Deloffre Alcohol Process, named after the inventor, promises to improve world's yeast industry, according to a recent Australian report. The yeast, in this process, is made in one operation by inoculating a highly concentrated solution with a culture of pure yeast, which is then fermented to set up the required biological reactions under which yeast can absorb alcohol.

The advantages of the process are many. Due to the high alcohol concentration in the fermenting liquid, fermentation takes place under sterile conditions without any bacterial contamination. The manufacturing process is simplified, the production cost cut by 30 per cent, yield increased by 20 to 25 per cent, and the yeast produced has exceedingly high keeping qualities because high alcohol concentration reduces the protease content of yeast, and has better baking properties with substantially greater leavening power.

The entire process is automatic and a special feature of the plant is that the pipe lines conveying the materials are kept to a minimum length.

Australia uses about 8,000,000 lb. of yeast each year for baking purposes and 80 per cent of it is produced by the Deloffre Alcohol Process ( *Agricultural Newsletter, Australia* ).

### New Varieties of Fish in Rihand River

AN UNKNOWN SPECIES OF FISH and several other specimens of exceptional zoological interest have been discovered in a fish survey of the Rihand river in the Mirzapur district ( U.P.) recently carried out by the *Zoological Survey of India*. 42 varieties of fish were found in the neighbourhood of the site of Pipri dam. 16 of these are of economic importance, though all of them are edible. The South Indian *Khudree Mahseer* found in the river grows to a large size and may be found suitable for large-scale cultivation in the dam reservoir. The temperature is also likely to suit the culture of Indian carp. It is estimated that if scientifically stocked and judiciously exploited, the reservoir may yield over 1,300,000 lb. of fish annually.

### Wind Power for Agriculture

THE *Monthly Weather Review* published by the Government of India shows average and normal wind velocity at 8 a.m. and 5 p.m. at hundreds of weather stations throughout the country. Unfortunately these hours are unsatisfactory for measuring windmill possibilities, because wind velocity is then much less than during the other hours of the day. At Belgaum ( Bombay Province ), for which hourly data are available, the monthly averages are 2 to 3 times as great as the average velocity at 8 a.m. and 5 p.m. recorded in the Review. Nevertheless, these data give a rough indication of the places where more wind is obtained and months when winds are strongest. Possibly part of the seasonal variation is due to the fact that wind strengths are greater at an earlier hour and dies down at a later hour when days are longer.

The normal wind velocities recorded at 3 stations in India ( TABLE I ) give a fair idea of the wind velocities.

The records at other stations show that India has enough wind power to operate certain types of American windmills almost everywhere except in the Gangetic plains. The best places for windmills are in the draught-stricken Saurashtra and almost anywhere between Karachi and southern Bombay province. To this should be added parts of coastal region along the Bay of Bengal and much of Deccan plateau and South India.

A pamphlet published by the Director General of Observatories, *Wind Data for Windmills*, reports experiments conducted in Madras which shows that a steady breeze of about 6 miles an hour would keep a windmill in continuous operation pumping water from a depth of 25′. A 4-mile breeze at Coimbatore was shown to pump enough water for domestic purposes.

In more detailed basic data available for 12 points in India with wind velocity recorded every hour, several other favourable factors are revealed ( TABLE II ). Wind velocity is greater during day-time when it is convenient to irrigate the crops. There is generally more wind during the hot months preceding and at the beginning of the monsoon rains when it is most important to get the crops sown and growing, and there is scarcely a month without sufficient wind power not to justify a windmill except at Agra, Jubbulpore and Calcutta.

The data show that in a year a windmill pumping water for 2,000 hr. will be pumping water for more hours than a pair of bullocks do. A windmill of the American type described in the United States Department of Agriculture, *Farmer's Bulletin No.* 1857 ( December 1940 ), costs just a little more than a pair of bullocks and can easily be worked on wind velocities of 4 to 6 miles an hour. A windmill pumping

TABLE I — NORMAL WIND VELOCITY : MILES PER HR. AT 8 A.M. & 5 P.M.

| | RAJKOT | SHOLAPUR | BANGALORE |
|---|---|---|---|
| January | 4·1 | 5·7 | 5·1 |
| February | 4·8 | 5·5 | 4·7 |
| March | 6·0 | 5·7 | 4·5 |
| April | 8·1 | 6·4 | 4·3 |
| May | 11·3 | 8·2 | 5·5 |
| June | 11·7 | 8·2 | 8·2 |
| July | 12·0 | 10·1 | 8·4 |
| August | 10·2 | 8·7 | 7·3 |
| September | 7·0 | 6·4 | 5·8 |
| October | 4·2 | 5·9 | 4·1 |
| November | 3·6 | 6·1 | 4·4 |
| December | 3·6 | 5·7 | 4·8 |

TABLE II — WINDY DAYS & HOURS DURING A YEAR

(*Average of 5 years* )

| STATION | DAYS WITH WIND BLOWING AT LEAST 6 MILES PER HR. FOR 10 HR. OR MORE | HR. OF WIND BLOWING AT LEAST 7 MILES PER HR. |
|---|---|---|
| Dhubri ( Assam ) | 91 | 2,190 |
| Sagar Island ( Calcutta ) | 263 | 5,782 |
| Calcutta ( Alipore ) | 40 | 1,051 |
| Allahabad | 116 | 2,628 |
| Agra | 40 | 1,139 |
| Deesa ( Rajputana ) | 226 | 4,643 |
| Jubbulpore | 69 | 1,752 |
| Bombay | 223 | 4,468 |
| Belgaum | 307 | 6,132 |
| Bangalore | 248 | 5,081 |
| Madras | 182 | 3,767 |
| Kodaikanal | 212 | 4,380 |

500 gallons a day per hour for 13 hr. a day will deliver 2′ of water to an acre of land within a week.

The factory price of an American 40′ windmill tower in U.S.A. is about $150 and the price of a wheel and accessories range from $100 for the 6′ size to about $300 for a 12′ size.

Too much should not be expected from wind power. If the required flow of water is over 400 cu. ft. per hr. or if the lift is much over 50′, it is the electric power or fuel power that can do the job ( *USIS* ).

### Non-ferrous Metals Development Committee

THE COMMITTEE HELD ITS SECOND meeting during the last week of January and, after reviewing the general overall position of the non-ferrous metallurgical industries in the country, fixed the following target figures for the non-ferrous metals and alloys with the ultimate objective of attaining self-sufficiency in respect of semi-manufactured metals and alloys ( TABLE below ).

| | | |
|---|---|---|
| Copper ( fire refined ) | 6,000/7,000 | tons per year |
| Copper ( electrolytic, from copper-base scrap ) | 20,000 | ,, ,, |
| Copper sheets | 6,000 | ,, ,, |
| Brass sheets | 35,000 | ,, ,, |
| Copper and brass tubes and shapes | 5,000 | ,, ,, |
| Lead | 6,000 | ,, ,, |
| Antimony ( depending upon imported ore ) | 7,000 | ,, ,, |
| Aluminium ingots | 15,000 | tons, short-term target |
| ,, sheets | 50,000 | tons, long-term target |
| ,, sheets | 10,000 | tons, short-term target |
| | 25,000 | tons, long-term target |
| ,, foil | 1,500 | tons per year |
| ,, powder | 1,000 | ,, ,, |
| ,, wire and cables | 5,000 | ,, ,, |
| ,, tubes and shapes | 2,000 | ,, ,, |

As regards white metal and copper-base alloy ingots and castings, it was stated that there was already adequate capacity in the country to meet any demand including exports.

To attain the above target figures, the Committee recommended to the Government the need to take immediate steps to have a comprehensive project about the setting up of an electrolytic copper refinery to treat copper-bearing scrap, as at the present moment India had to depend entirely on imports for her requirements of electrolytic copper. This move is deemed essential not only on account of the strategic importance for the defence of the country but also for peacetime industrial development. The Committee also recommended the need for Government assistance for the development of the Zawar lead-zinc mines, and for the procurement of antimony ore to the smelter at Bombay, which had now to depend entirely on imported ore. India has ample resources for increasing aluminium production and, therefore, the Committee emphasized the need to formulate a definite policy regarding this industry. But as any scheme of expansion badly needs finance, which at the present moment is difficult to raise, the Committee felt that it was most essential for the Government to create favourable conditions to encourage investment in industry.

### Committee on Factory Data Reporting

A SPECIAL COMMITTEE OF THE *International Society of Sugarcane Technologists* on uniformity in reporting factory data has been formed with the following members : Dr. F. W. Zerban ( New York ), Chairman, and Messrs Alegandro S. Alvarex ( Argentina ), Anibal R. de Matos, Hoao da Feiga Fromiga, Helio Morganti ( Brazil ), J. F. Williams, J. C. Davies ( West Indies ), Mario A. Mascaro, Raul E. Daigo, Ceasar Forn, Julio C. Gonzalex Maiz, Rafael Pedrosa Puertas, Jaun C. Salines ( Cuba ), Y. T. Chou ( Formosa ), W. L. McClerry ( Hawaii ), P. Honig ( Java ), K. C. Banerji ( India ), A. G. Keller ( Lousiana ), Louis Baissae ( Mauritius ), Jorge A. Aolano ( Peru ), M. A. Del Valle ( Puerto Rico ), J. L. du Tiot ( South Africa ), and Walter Scott ( Venezuela ).

### U.S. Technical Information Committee for Military Establishments

THE FORMATION OF A SPECIAL Committee on Technical Information to promote effective exchange of research and development information among the departments of the National Military Establishment was announced recently by Dr. Vanevar Bush, Chairman of the Research and Development Board, National Military Establishment, U.S.A. The Committee will study the problem of collecting, correlating, reproducing and disseminating technical information potentially useful in the research and development programme of the National Military Establishment. Study and application of new methods and techniques to the problem of technical information organization and promotion of active research in this effort are to receive special attention by the group.

### Herbage & Fodder Plants

BULLETIN 39 OF THE *Australian Commonwealth Bureau of Pastures & Field Crops*, entitled *Five Hundred Varieties of Herbage and Fodder Plants* ( edited by M. Hall ; Price 18*s*. 9*d*.), is a descriptive catalogue of recognized species and varieties of fodder plants. Under each variety details are given of its origin, the authority responsible for its development, its characteristics, adaptation, use and certification, together with notes on the market for seed. Species are arranged alphabetically and varieties are grouped according to the country of origin or use. Australian species are covered better than that of other countries.

The Bulletin will be of particular interest to persons concerned with the introduction and breeding of pasture plants and forage crops, and will also be a useful document for agriculturists and seedsmen who give advice on the choice of varieties for specific purposes. Copies of the publication can be had from the Liaison Officer, Commonwealth Agricultural Bureaux, 314 Albert Street, East Melbourne, C. 2, Australia ( *J.C.S.I.R.*, 1948, **21**, 223 ).

### Wool Research, 1918-48

*The Wool Industries Research Association*, Torridon Headingley, Leeds 6, England, proposes to publish a series of 11 volumes on wool research in which the results of 30 years of scientific investigation of wool and its processing have been incorporated

The first volume, published already, is volume 4 on " Carding " and will be followed by volumes 5 and 6 on " Combing and Tops " and " Worsted Drawing and Spinning " respectively. Volumes 5 and 6 are expected to be ready during this year.

## Grant for Calcutta University

THE GOVERNMENT OF INDIA have sanctioned a capital grant of Rs. 20 lakhs to the University in addition to the recurring grant of Rs. 2·19 lakhs every year.

## Announcements

*First International Congress of Biochemistry* — A new section entitled " Industrial Fermentations " has been included in the programme of the Congress to be held at Cambridge from 19th to 25 August 1949.

*Sir Ben Lockspeiser* has been appointed Secretary, Department of Scientific & Industrial Research, Britain, in succession to Sir Edward Appleton. He will be taking up his new duties in May.

*Dr. C. V. Raman* has been elected corresponding member of the French Academy of Sciences, Paris.

*Dr. Birbal Sahni*, Director, Indian Institute of Paleobotany, has been elected honorary President of the 7th International Botanical Congress to be held at Stockholm in July 1950.

*Grading of Wool* — The Indian Textile Division Council of the *Indian Standards Institution* has drawn up a Draft Indian Standard Specification for grading of wool for export. This has been done with a view to help the industry to grow on sound lines by standardizing the various grades of exportable wool and to ensure that the exported product conforms to those standards. The draft standard is being circulated among wool producers, exporters and manufacturers and others. Comments on the subject will be received till the end of May 1949 by the Director, Indian Standards Institution, Block 11, Old Secretariat, Delhi 2.

*The Central Board of Irrigation Medal* — This medal has been instituted by the *Board* to encourage research relating to, and the technique and development of, water resources of India and all devices and matters connected therewith. The medal will be awarded every year for an outstanding original contribution of the preceding year, and will be awarded at the annual meeting of the *Board*. The contribution may be in the form of a paper presented to the annual meeting of the Research Committee of the Central Board of Irrigation or to any other engineering institution in India or may be an article published for the first time in the *C.B.I. Journal* or any other technical journal published in India. Applications for the award, accompanied with 5 copies of the paper or article, should be addressed to the Secretary, Central Board of Irrigation, Kennedy House, Simla 4, to reach him before March every year.

*Russian Technical & Scientific Publications* — The Research Information Service, 509 Fifth Avenue, New York 17, N.Y., have introduced a new and much-needed service by cataloguing and preparing translations of articles from current Russian technical and scientific publications. The subjects dealt with are : petroleum, metallurgy, aeronautics, machinery, medicine and a few others. The publications can be had from the publishers at the above address ( *Curr. Sci.*, 1949, **18,** 53 ).

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## ERRATA

### Vol. VIII, No. 3, Article : " Chemical Control for Power Alcohol Distilleries "

Page 91, R.H. col., item 8 : *for* " 12 to 13 " *read* " $\frac{12}{13}$ "

,, 92, R.H. col., item 20 : *for* " Wash + Steam = Spentwash + Equilibrium vapour = Wash + Steam – Equilibrium vapour " *read* " Wash + Steam = Spent wash = Wash – Steam – Equilibrium vapour "

,, 93, L.H. col., item I : *for* " gal. of spirit " *read* " gal. of proof spirit "

,, 93, L.H. col., item V : *for* " $\frac{\text{Alcohol \% wash (9)}}{\text{Possible recovery (I)}}$ " *read*

" $\frac{\text{Alcohol \% wash (9)}}{\text{Molasses \% wort (II)} \times \text{sp. gr. (5)}}$ "

,, 93, L.H. col., item IX :

*for* " $\frac{\text{Alcohol made (24)}}{\text{Distilled wash (17)} \times \text{Alcohol wash (9)}} \times 100$ " *read*

" $\frac{\text{Alcohol made (24)}}{\text{Distilled wash (17)} \times \text{Alcohol \% wash (9)}} \times 10{,}000$ "

,, 93, R.H. col., item X : *for* " gal. of spirit " *read* " gal. of proof spirit "

,, 94, R.H. col., item (f) 5 : *for* " md./gal. " *read* " lb./gal."

# Reports from States & Provinces

## BIHAR

### Mica Industry

THERE ARE ABOUT 600 MICA-mining centres in the mica belt of the province. Out of these a 100 are worked by mechanical means and the rest by manual labour. The average depth in the mechanized mines is 200′-250′ and the deepest ones range from 600′-700′. The average depth of mines worked manually varies from 5′-50′.

About 15,000 labourers are employed in these mines and over a lakh are engaged in mica-processing factories. Mica-splitting is carried on as a cottage industry, over a lakh of women providing almost all the labour. About 250 firms hold licence under the *Bihar Mica Act* for mining and out of them 15 firms produce nearly 80 per cent of the total mica.

There are over 2,500 licensed dealers in mica in the province. The export of mica is in the hands of 60 exporting firms of which 8 are responsible for most of the export. The 2 biggest exporters of mica in Bihar are: *Messrs Chatturam Horliram Ltd.*, and *Messrs Chrestein Mica Industries Ltd.*, whose combined exports amount to over 50 per cent of the output. The mica industry is the chief dollar earner for the country. Small holdings, large number of small exporters and absence of any standardization of mica for export purposes are some of the chief factors which hinder the proper organization and expansion of the industry.

Over 50 per cent of the mica exports during 1948 was for the American market. The mica exports for 1948 are shown below:

| *Variety* | *Quantity* Cwt. | *Value* Rs. |
|---|---|---|
| Block | 8,497 | 64,30,626 |
| Splittings | 1,63,426 | 4,44,07,669 |
| Ground and scrap | 1,23,895 | 7,40,299 |

The serious threat to this industry in the shape of synthetic mica can only be countered by putting the industry on a sound footing by introducing improved methods of mining, standardization of material for export, better marketing practices, and by providing training facilities and amenities for the labour employed in the industry. (*Contributed*)

## WEST BENGAL

### Locomotive Factory

THE FOUNDATION-STONE OF THE first Indian locomotive factory was laid by the Minister for Railways and Transport during January at Mihijam in the Burdwan district. The factory, with an out-turn of 120 steam locomotives and 30 spare boilers annually, besides standardized spare parts, will be set up at a capital cost of Rs. 8,50,00,000. The workshop will cover an area of 3,000 acres and will be equipped with over 1,000 assembling and manufacturing machines. The total cost of machinery will be about Rs. 3,00,00,000. Besides, the foundry, furnaces and minor machine tools will be obtained in India at a cost of Rs. 1,00,000. The Maithon Dam across river Barakar, six miles from Mihijam, will supply the necessary power for the factory, which is expected to go into production in January 1950. Standardized locomotive parts will be produced during the first year.

## MADRAS

### Mineral Resources of Sandur State

THE STATE OF SANDUR NEAR Bellary is reported to have rich deposits of iron and manganese, according to a recent survey by the *Geological Survey of India.* Smaller deposits of copper and gold. are also reported. The extent of the deposits of iron and manganese ores are estimated at 2·5 and 170 million tons respectively. The report recommends the installation of pilot plants for the production of cast iron and ferro-manganese steel.

### Cotton Growing

THE *Indian Central Cotton Committee* has sanctioned a 5-year scheme for improving the yields of cotton varieties grown in the province, viz. “Northerns”, “Westerns”, “Karunganni”, and “Cambodia”, and for trying out “Sea Island” cotton in Malabar and South Kanara districts. This scheme is expected to result in an increase in the cotton acreage in the province by at least 1 lakh acres. The area under cotton is expected to increase further by 1952, when the Lower Bhavani Project is expected to be complete.

## UNITED PROVINCES

### Sugarcane Development

TO IMPROVE THE YIELD PER ACRE of sugarcane in the factory zones of the province, the U.P. Government have inaugurated the Cane Development Competition Scheme which comes into force in the current cane season. The target is a minimum of 700 md. per acre which has been proved to be within the means of the average cultivator using approved methods of cultivation. The provincial Government has sanctioned a sum of Rs. 19,000 for this purpose.

The competition is held under 3 categories: (1) Among the cultivators — “Cane Development Zonal Competition”; (2) among the cane development zones in a range — “Inter-zonal Range Competition”; (3) among all the ranges in the province — “Inter-range Provincial Competition”.

The province is divided into 4 ranges, viz.: (1) Western Range; (2) Rohilkhand Range; (3) Central and Banaras Range; and (4) Mideastern and Eastern Range. The varieties of cane recommended for cultivation in these areas are: *Co.* 313, *Co.* 385, *Co.* 421, *Co.* 353, *Co.* 453, *Co. S.* 76, *Co.* 356, *Co.* 386, *Co.* 527, *Co.* 456, *Co.* 395, *Co.* 393, *Co. S.* 109 and *Co.* 370.

# INDIAN PATENTS

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

### Inorganic Chemicals

40400 Steinschlaegerl : Manufacture of carbon black : *Cracking carbonaceous material by mixing with hot gas.*

37641. The National Smelting Co. Ltd. : Removal of sulphur compounds from gases : *Contacting the gases with alkaline earth metal carbonate on a large porous support at* 100°C. *to* 850°C. ( Addition to No. 37640. )

38945. Seailles : Method of extracting alumina from bauxite or similar aluminous ores and simultaneously obtaining a residue available as ore rich in iron : *Roasting bauxite with calcareous material, lixiviating roasted product to extract alumina, mixing residue left from lixiviation with further bauxite and repeating the cycle till the residue reaches desired iron content.*

38947. Seailles : Improved method of extracting magnesia from aqueous solution containing magnesium salts decomposable by lime and alkalis : *Reacting successively magnesium salts with lime and alkali.*

37623. Dorr Co. : Calcining lime-bearing sludges : *Bringing solids and upflowing gaseous stream into calcining chamber under condition of suspending the solids, and calcining them to yield nodules of lime.*

39691. De Directie Van De Staatsmijnen in Limburg : Method of manufacturing a compound fertilizer from an acid solution which contains fluorine as well as calcium and phosphoric acid : *Maintaining the* pH *of the medium of the neutralizing and vaporization processes between* 1·5 *and* 2·5.

39937. I.C.I. Ltd. : Improvements in ammonium nitrate compositions : *Maintaining a mixture of ammonium nitrate and potassium nitrate between* 32°C. *and* 100°C.

### Organic Chemicals

40084. Stanolind Oil & Gas Co. : Process and catalyst for synthesis of hydrocarbons from carbon monoxide and hydrogen : *Contacting hydrogen and carbon monoxide with fluidized iron catalysts.*

39213. Les Usines De Melle : Saccharification of amylaceous materials in acid medium : *Through acid hydrolysis, neutralization, alcoholic fermentation and distillation.*

40072. N.V. De Bataafsche Petroleum Maatschappij : Method of producing ethyl alcohol : *Passing a mixture of ethylene and water vapour over a catalyst comprising of diatomaceous earth pellets soaked with phosphoric acid at high temperature.*

### Food & Kindred Products

37242. Ludin : Method of producing fat by means of micro-organisms : *Cultivation of yeast on carbohydrates and the extraction of the fat produced by a solvent.*

### Medical Research & Practice

37328. Elderfield & Head : Anti-malarials : *Condensing* 5, *6-dimethoxy-8-amino-quinoline with the hydrohalide of an alkylaminoalkyl halide.*

39032. Ortho Pharmaceutical Corp. : Production of compounds having vitamin A activity : *Subjecting cyclohexene derivative to hydrogenation, dehydration and acid or alkali isomerization.*

### Metals & Metal Products

39636. Carnegie-Illinois Steel Corp. : Electrodeposition of tin : *Electrolyte comprising acid solution of tin with an addition agent consisting of monobutyl-phenylphenol-sodium-monosulphonate or of diphenyl-p-phenylene-diamine or both and a sulphohone of phenol, cresol, resorcinol, or naphthol.*

38890. Carnegie-Illinois Steel Corp. : Electrolytic tin-plating baths and addition agents there for : *Addition agent for tin-plating bath consists of a polymerized reaction products of aniline or toluidine with acetone or methyl ethyl ketone.*

39302. The Mond Nickel Co. Ltd. : Cast iron : *Graphitic cast iron containing retained magnesium.*

39498. Chaudary : Manufacture of metallic sheets, strips, rods and wires : *Roller rotated in molten metal which adheres to roller as scale or sheet which is passed between two smooth rollers.*

### Stone, Clay & Glass Products

37338. Champman : Bricks : *Protruding portion of one brick, adjacent a mortar-retaining recess, projects into the adjacent recess of another brick.*

### Miscellaneous

40513. Brake & Osborne : Improvements in the treatment of water for the purpose of reducing the effects due to hardness : *Inducing electronic charges of high frequency from a valve oscillator having means for its frequency control.*

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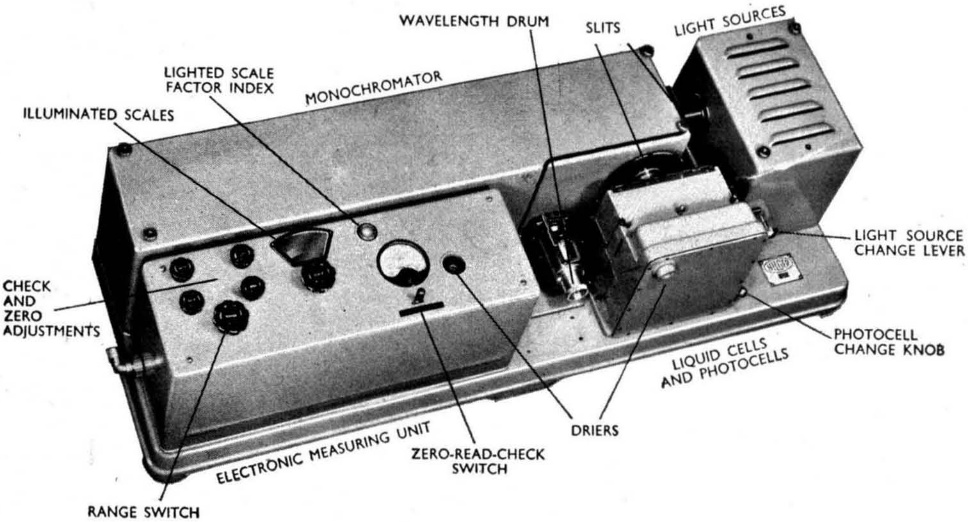
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RANGE SWITCH

# Hypochlorite Oxidation of Cotton Cellulose in Presence of Mixtures of Certain Vat Dyes — Part I

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THE ability of certain vat dyes to accelerate the oxidation of cellulose by air and by chemical oxidizing agents in presence of light is well established[1,2]. It has been shown that nearly all yellow, orange and several red, pink and brown vat dyes behave in this manner. None of the blue and green vat dyes exhibit this behaviour. In the dark, however, the oxidation of cellulose by the oxidizing agent has been found to be independent of the colour of the dye present on the fibre. This behaviour of vat dyes was explained by Scholefield and Turner[3] on the basis of a chemical theory, which may be summarized as follows: Firstly, illumination is necessary for the acceleration of the oxidation of the cellulose substrate and active dyes are more or less restricted to certain colour groups only. Secondly, the active yellow and orange vat dyes show a strong absorption of light of short wave length — between 3,600 to 4,000Å — while the non-active blues and greens do not show strong absorption in this region[4]. Scholefield and Turner[3] assumed that the yellow and orange vat dyes, by virtue of their ability to absorb light in the near ultra-violet region, where large light quanta are available, bring about the necessary chemical change. They further assumed that the initial chemical change brought about by the absorbed light is to reduce the vat dyestuff to its leuco compound and at the same time to cause an equivalent oxidation of the cellulose substrate. The leuco vat dyestuff thus formed then gets oxidized by air according to circumstances, also bringing about the auto-oxidation of cellulose, as suggested by Engler[5]. Scholefield and Turner[3], therefore, deduced that the chemical production of leuco compounds of vat dyes dyed upon the fibre should be capable of accelerating the oxidation of cellulose by oxidizing agents in the absence of light and irrespective of the optical properties of the vat dye.

This point was examined in considerable detail by Turner, Nabar and Scholefield[6,7], who showed that when a number of tendering and non-tendering leuco vat dyes on cellulose were treated with dilute hypochlorite solutions in the dark, a considerable amount of the acceleration of the hypochlorite action on cellulose is brought about. The extent of the modification depended on the nature of the vat dyestuff present on the cellulose. With dyes which themselves underwent modification by the hypochlorite treatment, the extent of modification in the cellulose substrate was lower than that when the dyes were unaffected by hypochlorite. The acceleration of cellulose oxidation during the air oxidation of leuco compounds of vat dyes chemically produced in the dark has also been demonstrated by Breare and Turner[8].

Another interesting aspect of the behaviour of tendering action of active vat dyes is their behaviour in the presence of a non-active blue or green vat dye on cellulose. Scholefield and Patel[9] investigated the action of light on dyeings prepared from mixtures of active and non-active vat dyes. They found that in such mixtures the light-resistant blue was rapidly destroyed when an active yellow or an orange vat dye was present. They further showed that when mixtures of vat blues and active yellows were exposed in their leuco state to light and air, the blue is in certain cases wholly or partially destroyed. Ciba Blue 2B was found to be especially susceptible in this respect. The same behaviour was observed when the mixed dyeings were subjected to the action of chemical oxidizing agents in the presence of light. Similar observations have been recorded by Scholefield and Stafford[10], Konig[11] and Jones[12].

The present investigation relates to the behaviour of a mixture of a non-tendering dyestuff with an active orange or yellow towards the action of dilute sodium hypochlorite solution. From among the various tendering and non-tendering vat dyes, it was decided to select Ciba Blue 2B to represent the non-tendering class and Cibanone Orange R to represent the tendering class. Dyeings on cotton were prepared using various concentrations of the two dyestuffs individually and in mixture on cotton yarn. These dyeings, both in the reduced and unreduced state, were then subjected to the action of hypochlorite solution under controlled conditions of concentration of available chlorine, *p*H and temperature for 10 min. The available oxygen lost from the hypochlorite solution as a result of the treatment was estimated and the treated dyeings after thorough washing, drying and conditioning were examined for their cuprammonium fluidity and copper number. The degree of fading or destruction of the blue component was visually noted.

## Experimental

Sodium hypochlorite used in the investigation was prepared according to the methods of Musprat and Smith[18]. The stock solution had the following characteristics : free alkali, 2·0-2·5 N ; active chlorine, 150-160 gm./litre ; chlorate and carbonate, nil. For the oxidation experiments the dilute hypochlorite solutions were buffered to *p*H 9·1 using M/20 borax solution[19].

*Available Chlorine* — The concentration of active chlorine in buffered sodium hypochlorite solutions used for oxidation was adjusted, as near as possible, to 3 gm. available chlorine per litre. The *p*H of the solutions was determined by using the Morton type glass electrode system and a valve potentiometer[20].

*Cotton* — Fully bleached 18's yarn manufactured from Indian cotton was used for oxygen consumption experiments. The grey yarn was subjected to standard bleaching treatment ensuring more or less complete removal of impurities from the cellulose. After thorough washing, drying and conditioning, the cotton had the following properties : cuprammonium fluidity[13] ( 0·5 per cent solution ), 7·8 ; copper number[14] ( Heyes' method), 0·15 ; milliequivalents of $-COOH$/ 100 gm. of cellulose, 0·5.

*Method of Dyeing* — The dyestuff was pasted with a small quantity of monopol soap and dispersed in 2 litres of cold water. Calculated quantities of caustic soda and hydrosulphite were added while stirring the mixture. It was then gradually heated to 60°C. and then allowed to vat at this temperature for 30 min. 100 gm. of cotton yarn was wetted out with water containing a little alkali and hydrosulphite. It was squeezed and then worked in the dye solution for 45 min. The cotton, as far as possible, was kept immersed below the surface of the liquor during the dyeing. It was then removed from the dye-bath and excess liquor evenly wrung out. The dyeing was then kept immersed in cold running water till the oxidation of the dye was complete. This took about 2 hr. It was squeezed, allowed to dry, conditioned in the dark and stored away from dust and light.

*Reduction of the Dyeing* — 10 gm. of the dyed yarn was treated with cold 2 per cent sodium hydrosulphite solution for 3 min. ( The reduction of the dyestuff takes place within the first half minute after treatment with hydrosulphite solution. ) The dyeing was then removed from the hydrosulphite bath, excess solution wrung out rapidly and the reduced dyeing immersed at once in cold distilled water from which air had been expelled by previous boiling. It was kept stirred below the surface of water for 1 min. and then transferred to another similar bath. This was repeated 5 times. Finally, the yarn was squeezed between fingers to remove the excess water and at once transferred to a hypochlorite solution suitably buffered and kept ready in a stoppered flask. The repeated washings of the reduced dyeing were sufficient to remove the impurities carried from the reduction bath by the cotton.

*Hypochlorite Treatment of Reduced Dyeings & Determination of Oxygen Consumed* — The method is similar to the one used by Nabar, Scholefield and Turner[7]. 10 gm. of the reduced vat dyeing were used for oxidation

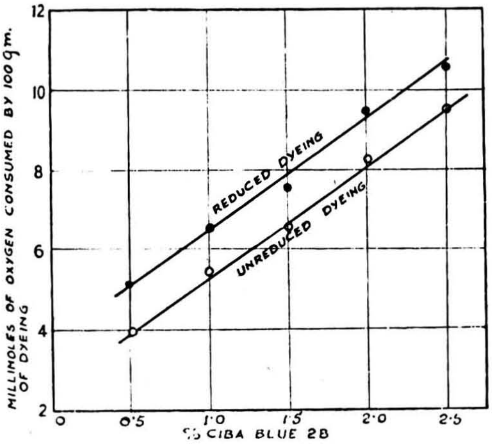

FIG. 1 — RELATION BETWEEN PERCENTAGE OF CIBA BLUE 2B USED FOR THE DYEING AND THE AMOUNT OF OXYGEN.

experiments. It was found that this weight of cotton approximately displaced 10 c.c. of water. It is impossible to get the reduced and washed dyeings in the dry condition and it is necessary to transfer the material to the hypochlorite solution in a wet state. The cotton, therefore, carries with it an undetermined weight of water and dilutes the hypochlorite. To allow for this dilution, the following procedure was adopted.

A flat-bottomed flask was graduated to hold 560 c.c. ( 550 c.c. for the liquor and 10 c.c. for cotton ). Into this flask were introduced 500 c.c. of buffered sodium hypochlorite solution and the wet dyeing. The volume was then made up to 560 c.c. with cold distilled water. This diluted the original 500 c.c. of the hypochlorite solution to 550 c.c. A control experiment in which 227·3 c.c. of the stock solution was diluted to 250 c.c. with distilled water was carried out. After the hypochlorite treatment ( 10 min. ), the liquor which remained was analysed for available chlorine and its final *p*H. From the difference in the concentrations of available chlorine in the control experiment and the solution after treatment, the net amount of oxygen used during the treatment can be calculated. The *p*H of the solution remained unaffected.

The reduction and oxidation of the dyeing was carried out in a dimly lit room. The hypochlorite solution used was standardized to contain 3 gm. available chlorine per litre. The time of treatment was 10 min. During the treatment, the dyeing was stirred below the surface of the liquor to ensure uniform contact.

The oxidized cotton was washed free of chlorine.

*Cuprammonium Fluidity* — The cuprammonium fluidity of the various oxidized samples was determined by the standard *B.C.I.R.A.* method[13].

*Copper Number* — The copper numbers were determined by the Schwalbe-Braidy method developed by the *B.C.I.R.A.*[16] and adapted for use with smaller quantities of the sample by Heyes[14].

### Experimental Results

A synopsis of the experimental results is presented graphically in Figs. 1-8.

In this series of experiments, the concentration of Ciba Blue 2B and of Cibanone Orange R was varied and the dyeings were treated in the reduced as well as in the unreduced state. The concentration of the dye, the oxygen consumption and the cuprammonium fluidity of the dyeing are given in Figs. 1-4. Corresponding values for the mixed dyeings are given in Figs. 5-8.

### Discussion

In discussing the experimental results, consideration has to be given to: (a) the effect of a non-active dye such as Ciba Blue 2B on the oxidation of cellulose when treated with hypochlorite solutions; and (b) its influence on the properties of a tendering dye such as Cibanone Orange R in bringing about the acceleration of the oxidation of cellulose.

A reference to results set out in Fig. 1 shows that Ciba Blue 2B dyed cotton, both in the reduced and unreduced state, when treated with sodium hypochlorite solution for 10 min., brings about a considerable

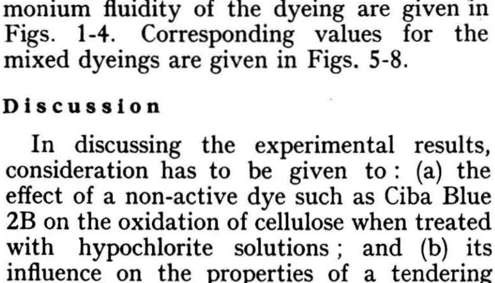

FIG. 2 — RELATION BETWEEN CUPRAMMONIUM FLUIDITY OF THE DYEING AND THE CONCENTRATION OF CIBA BLUE 2B.

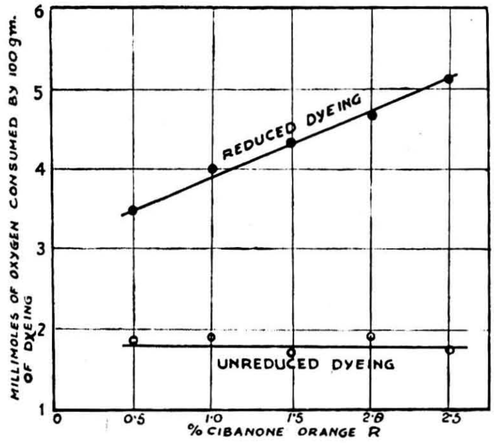

FIG. 3 — RELATION BETWEEN PERCENTAGE OF CIBANONE ORANGE R AND THE AMOUNT OF OXYGEN CONSUMED AND UNREDUCED DYEING.

amount of loss of active oxygen from solution. This amount of oxygen may be partly utilized in oxidizing the cellulose and partly in destroying the dyestuff. Close examination and comparison of the dyeings treated with hypochlorite solution both in the reduced and unreduced state with the untreated dyeings shows considerable loss of colour as a result of oxidation ; so, at least part of the oxygen lost from the solution must have been used up for the destruction of Ciba Blue 2B. It is known that when Ciba Blue 2B dyed cotton is exposed to light and air for sufficiently long periods, the main product of oxidation of Ciba Blue 2B is di-bromo-isatin[17]. From Fig. 2 it is seen that the amount of degradation produced in the cellulose substrate is small. For an oxygen up-take of 0·32 per cent, a cuprammonium fluidity ( 0·5 per cent solution ) of the order of 60 reciprocal poises may be expected, if all the oxygen is utilized for the oxidation of the substrate. It is found that the fluidity of the treated dyeings is of the order of 14 units and that of the untreated dyeings of the order of 10 units.

Applying the idea of auto-oxidation, numerous examples of which are collected by Engler[5], one would expect an extensive destruction of cellulose in the presence of an easily oxidizable substance. In so far as the results of the oxidation of cellulose in the presence of Ciba Blue 2B, both in the reduced and unreduced state, are concerned, this idea does not seem to be fully operative. In fact, in spite of an increased oxygen consumption with an increase in the concentration of the dyestuff, the fluidity of the substrate is not altered ( FIG. 2 ). The oxygen consumption appears to be simply related to the amount of dye present on the fibre. The difference of approximately 1 millimole between the reduced and unreduced dyeing ( FIG. 1 ) appears to be mostly due to that amount required for the conversion of the leuco-Ciba Blue 2B to the fully oxidized stage.

Sharply differentiated with these results are those obtained with Cibanone Orange R ( FIGS. 3 and 4 ). It will be seen that with an increase in the concentration of the dyestuff on cellulose the oxygen consumption also increases ( FIG. 3 ), as was found to be the case by Nabar, Scholefield and Turner[7]. At the same time, the cellulose substrate has been found to have undergone considerable chemical modification ( FIG. 4 ). For an oxygen consumption of about 3-5 millimoles ( 0·112 per cent ), the cuprammonium fluidity of the treated dyeing has been found to be of the order of 27 reciprocal poises, indicating a considerable chemical damage. Cibanone Orange R appears to be chemically inert to hypochlorite treatment and the oxygen taken up by the reduced dyeing is used up practically in oxidizing the cellulose substrate.

In Figs. 5-8 are given results of hypochlorite oxidation experiments carried out with mixed dyeings of Ciba Blue 2B and Cibanone Orange R. It has been pointed out[9] that when such a mixed dyeing is

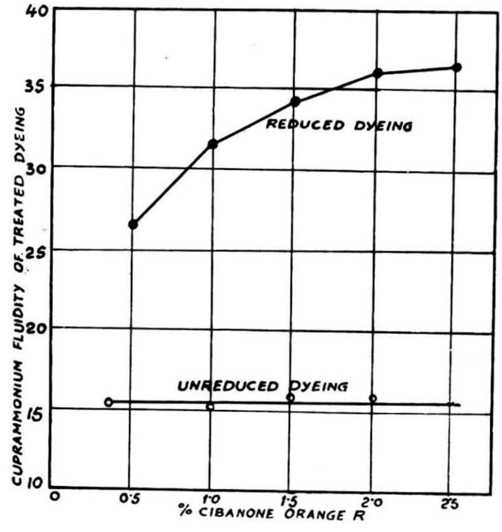

FIG. 4 — RELATION BETWEEN CUPRAMMONIUM FLUIDITY OF THE DYEING AND THE CONCENTRATION OF CIBANONE ORANGE R.

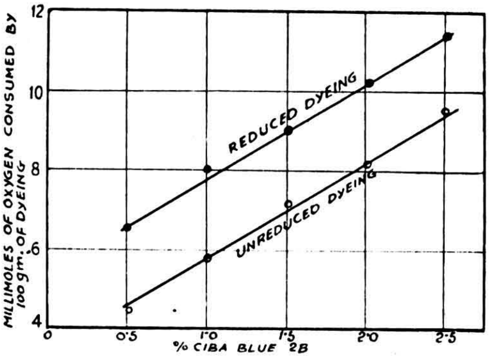

FIG. 5 — RELATION BETWEEN PERCENTAGE OF CIBA BLUE 2B AND THE AMOUNT OF OXYGEN CONSUMED BY REDUCED AND UNREDUCED DYEING IN THE MIXED DYEING.

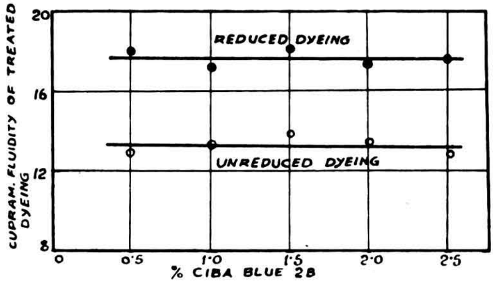

FIG. 6 — RELATION BETWEEN CUPRAMMONIUM FLUIDITY AND CONCENTRATION OF CIBA BLUE 2B IN THE MIXED DYEING.

exposed to light and air, the blue component gets rapidly destroyed giving protection to the cellulose substrate from the degrading action of Cibanone Orange R. It is interesting to find similar behaviour to exist when mixed dyeings produced from Ciba Blue 2B and Cibanone Orange R are treated in the reduced and unreduced state with hypochlorite solutions. From Fig. 5 it will be seen that when the amount of Cibanone Orange R in the mixed dyeing, is kept constant and the amounts of the Ciba Blue 2B are increased in regular amounts, the oxygen up-take also increases in the same manner. It is interesting to find that in the case of the unreduced mixed dyeings, when treated with hypochlorite solutions, the amounts of oxygen consumed are more or less the same as those consumed by the pure Ciba Blue 2B dyeings of similar concentration ( Fig. 1 ). The amount of oxygen consumed by the reduced mixed dyeings, however, are consistently higher by about 1 millimole than those for the reduced pure Ciba Blue 2B dyeing. This consistent difference must be due to the fact that the reduced mixed dyeing contains, in addition to Ciba Blue 2B, 2·5 per cent Cibanone Orange R, which has to be converted to the oxidized stage. Examining the cuprammonium fluidity of the treated mixed dyeings ( FIG. 6 ), it is found that in spite of 2·5 per cent Cibanone Orange R being present on the dyeing, the rise in cuprammonium fluidity is not at all significant. When dyeing with 2·5 per cent Cibanone Orange R alone is treated in the reduced state with hypochlorite solution, a cuprammonium fluidity of the substrate rises to 36 units ( FIG. 4 ), while the same for the mixed dyeing is of the order of 18 units ( FIG. 6 ). The presence of Cibanone Orange R, however, causes a very small but consistent increase in the cuprammonium fluidity of the mixed samples. When these results are compared with those obtained ( FIGS. 7 and 8 ) with mixed dyeings when the concentration of Cibanone Orange R was varied, keeping that of Ciba Blue 2B at 2·5 per cent, it is seen that the increase in the concentration of Cibanone Orange R does not make any difference in the amounts of

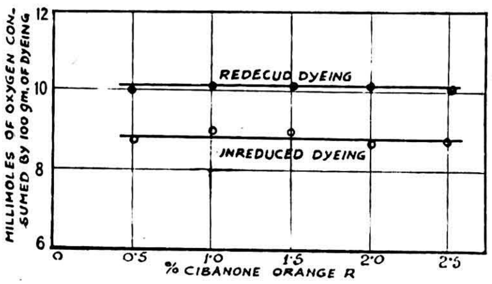

FIG. 7 — RELATION BETWEEN THE PERCENTAGE OF CIBANONE ORANGE R AND THE AMOUNT OF OXYGEN CONSUMED IN THE MIXED DYEING.

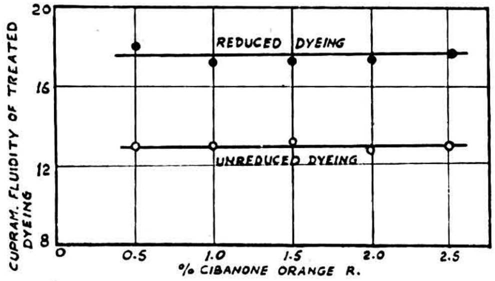

FIG. 8 — RELATION BETWEEN CUPRAMMONIUM FLUIDITY AND CONCENTRATION OF CIBANONE ORANGE R IN THE MIXED DYEING.

oxygen consumed by the mixed dyeings ( FIG. 7 ) as was found to be the case when Cibanone Orange R alone was dyed on cotton ( FIG. 3 ). The cuprammonium fluidity is also not altered with increase in the concentration of Cibanone Orange R. Both these observations indicate that in presence of Ciba Blue 2B, Cibanone Orange R, a pronounced tendering dye, both in the unreduced state in the presence of light and air and in the reduced state in the presence of oxidizing agents, loses completely its identity and its tendering properties appear to be lost in the presence of Ciba Blue 2B. It should be of interest to find out the mechanism by which Ciba Blue 2B renders Cibanone Orange R inactive in so far as tendering of cellulose substrate is concerned.

## Summary

1. The oxidation of cellulose dyed with Ciba Blue 2B, Cibanone Orange R and their mixture both in the reduced and unreduced state by dilute solutions of sodium hypochlorite is studied.

2. It is found that in the reduced state Ciba Blue 2B does not accelerate the oxidation of the cellulose substrate. Cibanone Orange R, on the other hand, under similar conditions causes a considerable acceleration of the oxidation of cellulose.

3. In the presence of reduced Ciba Blue 2B, reduced Cibanone Orange R loses its identity with the result that there is no acceleration of the oxidation of cellulose by hypochlorite solutions.

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# Lac-linseed Oil Varnishes
## Part III — Lac-linseed Oil-lime

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THE possibility of preparing lac-linseed oil varnishes using lead oxide and glycerine respectively as incorporating agents has been studied and reported in two previous papers[1,2]. It has been pointed out that about 15 per cent of litharge on the weight of lac is necessary to ensure complete dissolution of lac in the oil. It has also been pointed out that whereas baked films of the lac-linseed oil-lead oxide compositions are quite smooth and homogeneous, air-dried films are not so smooth and present an uneven surface. This was interpreted as being possibly due to the large amount of the " drying " metal necessarily present in the composition. It was hoped, therefore, that if the lead oxide could be partially or totally replaced by the oxide of another metal of the non-drying or at least less drying type, the defect might be eliminated. With this end in view, lime ( calcium hydroxide ) was investigated as a possible substitute for lead oxide.

The possibility of using lime as an incorporating agent for the preparation of lac-linseed oil combinations has been reported by Aldis[3] and by Fain[4]. The former, beyond reporting one particular proportion, does not appear to have studied the problem in all its aspects such as the film properties of the resulting compositions, while the latter has used the product only in conjunction with other synthetic resins. The problem has, therefore, been investigated anew in detail. As stated already, about 15 per cent of litharge on the weight of lac is necessary to bring about proper incorporation of lac in linseed oil. This, in terms of calcium hydroxide, will be equivalent to about 5 per cent on the weight of lac. Thus, to dissolve 50 gm. of lac in 100 gm. linseed oil, 7·5 gm. of litharge or its equivalent 2·5 gm. of calcium hydroxide are required, which was actually found to be the case. For the proportions given above, the temperature of incorporation is 240°-250°C. in the case of litharge, but when lime is used, a higher temperature ( 280°-290°C. ) is necessary. If less than 2·5 gm. of lime are used, a portion of the lac is left behind in granular form. Increasing the amount of lime lowers the temperature of incorporation but not to the same extent as in the case of litharge. Again, when 100 parts of lac are dissolved in 100 parts of linseed oil using 15 parts litharge, the temperature of incorporation is 220°C. But with these same proportions of lac and oil and the equivalent quantity of lime ( i.e. 5 parts ) in place of litharge, the product obtained as a result of reaction at the same temperature, namely 220°C., does not become clear immediately as in the case of litharge but only after 20-25 min. If, however, the temperature is raised to 290°C. the product becomes clear immediately. From the above data it may be inferred that as an incorporating agent lime is not so efficient as litharge.

It was observed in the course of our experiments that precipitated calcium linoleate may be used in place of lime for incorporating lac in linseed oil ( cf. lead linoleate ), and it was of interest to investigate whether any other soap of calcium would serve the same purpose or it should only be the linoleate. Accordingly, calcium stearate was tried, and was found to effect the dissolution quite well. Other metallic soaps, e.g. the stearates of zinc, aluminium and potassium as well as linoleates of manganese, sodium and potassium were tried, and it was found that all of them were more or less similar in bringing about the compatibility.

Among other compounds tried were sodium acetate and sodium oleate which, however, did not induce solubility, though the " life " of lac in the oil was prolonged considerably. The chemical reactions involved in these incorporations are being examined and will be reported later.

*Properties of Lac-linseed Oil-lime Compositions* — Though lime is not quite so efficient as litharge as incorporating agent, the products obtained by using either are very similar. Thus, lac-linseed oil-lime compositions are all soluble in the usual varnish solvents like white spirit or turpentine, and require, in addition, small proportions of alcohol to eliminate false body when the proportion of lac to oil exceeds 70-75 per cent. These varnishes are naturally slow-drying owing to the absence of lead, but when suitable driers are incorporated, the drying property can be considerably improved. The air-dried films are smooth and homogeneous. Even in the absence of a drier, the films dry when baked at 90°-95°C. in the course of 3-4 hr. and in shorter time at higher temperatures. The varnishes are non-skinning and the baked films are considerably more resistant to the effect of heat than the lead oxide compositions with regard to their ageing characteristics.

*Lac-linseed Oil-litharge-lime* — It was noted that though the lac-linseed oil-lime compositions could be made to air-dry in less than 24 hr. by using excess of lead and cobalt driers, the product could not be made to " surface-dry " in less than 6 hr. as required in some of the specifications. It was hoped that by substituting a portion of the lime with litharge, a composition might be obtained which would surface-dry in less than 6 hr. and retain all the other properties unaffected. Such a product could actually be made by using 3 parts of litharge and 1·5 parts of lime for incorporating 50 parts of lac in 100 parts of linseed oil.

A varnish based on the combined use of lime and litharge in the above proportions may be made under the usual conditions at a temperature of 270°-280°C. (as described for litharge). With the addition of 0·04 per cent cobalt in this varnish the surface-drying time is less than 6 hr. and the hard-drying time less than 18 hr. The resulting film in this case also was smooth. The acid value of the product is low and the colour satisfactory. Incidentally, this composition without the cobalt drier was found to be superior for use as a clear baking, insulating varnish to the one using only lime as the incorporating agent, as the drying was faster. This was also perfectly non-skinning in spite of the presence of lead. A sample of " empire cloth " prepared on a 5-mil cotton support by the application of 4 coats of this varnish (by dipping), each coat being baked in a tower at 125°C. for 1 hr. gave a product with the following characteristics :

| | |
|---|---|
| Thickness | 10 mil |
| B.D.V. at laboratory temperature after drying in a vacuum desiccator for 24 hr. | 980 volts/mil |
| B.D.V. at 90°C. | 700-750 volts/mil |

## Summary

1. Lac can be incorporated in linseed oil into which 5 per cent of lime on the weight of lac has been dissolved.
2. In place of lime the equivalent quantity of precipitated calcium linoleate or stearate may be used.
3. Other metallic soaps like zinc stearate, potassium linoleate, etc., also assist in the incorporation of lac in linseed oil.
4. Lime may be partially replaced by litharge to improve the drying characteristics of the composition.
5. The products obtained in either case are practically similar to those obtained using litharge alone except for the drying characteristics. They have, however, better ageing properties.
6. Cobalt or manganese together with lead may be used to improve the drying properties.

## Acknowledgement

The author wishes to acknowledge his grateful thanks to Dr. P. K. Bose, Director, Indian Lac Research Institute, for his interest and suggestions in the progress of the work.

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# Chemical Examination of the Dried Latex from *Euphorbia tirucalli*

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KARIMULLAH and N. L. Dutta[1] have reported the isolation of a crystalline substance from the dried latex of *Euphorbia tirucalli.* The present paper deals with the results of further work on the characterization of the crystalline substance, and also of the resin which constitutes the main bulk of the dried latex.

The crystalline matter was isolated in a crude form from the ether extract of the dried latex by dissolving it in a minimum quantity of hot alcohol and allowing the solution to cool. The yield of the ketone, which for ease of reference may at this stage of investigation be provisionally named *Euphoron,* varies from 20 per cent in fresh samples to 2 per cent in samples stored for about a year. In keeping with its ketonic nature, *Euphoron* gives an oxime (m.p. 194°-195°C.), a hydrazone (m.p. 149°-150°C.), and a semicarbazone (m.p. 178°-179°C.). It contains 2 double bonds as reported earlier (loc. cit.); but it takes only 2 hydrogen atoms on catalytic reduction with platinum oxide yielding a product which melts at 65·5°C. and is ketonic in character as it forms an oxime (m.p. 205°C.). Reduction with sodium and amyl alcohol, however, gave the corresponding alcohol in 20 per cent yield along with a large quantity of resinous material from which no crystalline matter could be isolated. The acetyl derivative of the alcohol, which will be referred to as *Euphorol,* melts at 107°-108°C., and is identical with the acetyl derivative of the alcohol isolated earlier (loc. cit.) from the mother liquor of *Euphoron.*

In the earlier communication, the formula $C_{27}H_{42}O$ was proposed for the ketone on the basis of the C, H values. Although the molecular weight determined by the Rast method (M.W. 390) agrees fairly well with this molecular formula (M.W. required, 382), the iodine value of the ketone and the nitrogen values of the oximes of the original and the hydrogenated ketones seem to be somewhat more favourable for formulations with a higher number of carbon atoms (cf. experimental). The exact formula for *Euphoron* cannot, therefore, be definitely fixed at the present stage of investigation.

The main component of the dried latex is a brittle, lustrous resin resembling rosin in appearance and melting at 65°-75°C. On distilling with superheated steam at 250°C. a portion of the resin distilled over as a viscous liquid, leaving behind a residue which melted at 125°-140°C. and was dark, lustrous and brittle in character. The linseed oil varnish prepared with this hardened resin gave films which were tack-free and glossy, indicating the possibility of its use in the formulation of paints and varnishes.

## Experimental

*Isolation of the Ketone* (*Euphoron*) — 5 kg. of the dried latex were powdered and exhaustively extracted with ether. The ethereal extract was concentrated to a thick syrup, which was dissolved in a minimum quantity of hot alcohol and left overnight for crystallization. The crystallizate was sucked, washed with alcohol and dried (yield, 1 kg.). On repeated crystallization of this product with chloroform, petroleum ether, ethyl acetate and alcohol, *Euphoron* was finally obtained as a colourless crystalline product melting at 119·5°C. The mother liquor yielded further quantities of the pure ketone, giving a total yield of 15 per cent on the weight of the dried latex. Euphoron is soluble in chloroform in the cold and ether, and in alcohol, acetone and petroleum ether in the hot and crystallizes from these solvents in star-shaped aggregates of needles.

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ANALYSIS*

| Required for | | C. % | H. % | M.W. | Iodine value ( Hanus ) |
|---|---|---|---|---|---|
| $C_{27}H_{42}O$ | | 84·80 | 10·99 | 382 | 132·9 |
| $C_{29}H_{46}O$ | | 84·87 | 11·22 | 410 | 123·8 |
| $C_{31}H_{50}O$ | | 84·92 | 11·41 | 438 | 116·0 |
| *Found* | (1) | 84·57 | 11·37 | 390 (Rast) | 110·0 |
| | (2) | 84·72 | 11·28 | | |

*Euphoron Oxime* — 1 gm. of hydroxylamine hydrochloride dissolved in 2 c.c. of water was added to 100 c.c. of alcohol containing 0·5 gm. of potassium hydroxide. The solution was then filtered and the filtrate refluxed for 1 hr. with 1 gm. of *Euphoron* on the water bath. The crystallizate obtained on cooling the solution at the end of the reaction was filtered, washed with water, and dried over a porous plate. On crystallization from a mixture of alcohol and chloroform, this product gave a nearly theoretical yield of the oxime ( 1 gm. ) which melted at 194°-195°C. and formed fine, colourless plates of crystals. *Euphoron* oxime is soluble in ether, chloroform, and ethyl acetate in the cold, and alcohol, acetone and petroleum ether in the hot.

ANALYSIS

| Required for | C. % | H. % | N. % |
|---|---|---|---|
| $C_{27}H_{43}ON$ | 81·60 | 10·83 | 3·53 |
| $C_{29}H_{47}ON$ | 81·89 | 11·06 | 3·29 |
| $C_{31}H_{51}ON$ | 82·12 | 11·26 | 3·09 |
| *Found* | 82·68 | 11·12 | 3·14 |

*Euphoron Semicarbazone* — To a hot solution of the ketone ( 1 gm. ) in alcohol were added semicarbazide hydrochloride ( 1 gm. ) and sodium acetate ( 1 gm. ) ; the mixture was refluxed over a water bath for 1 hr. and then poured into water. The precipitate was washed free from sodium acetate and crystallized from alcohol. The semicarbazone thus obtained melted at 178°-179°C. and was soluble in ether, alcohol, chloroform and petroleum ether.

*Euphoron Hydrazone* — To an aqueous solution of hydrazine hydrochloride ( 2 gm. ) was added 4 gm. of sodium acetate and 300 c.c. of alcohol and the resulting solution was refluxed with 1 gm. of ketone dissolved in a small quantity of hot alcohol for 4 hr. on the water bath. The crystals which separated on cooling the solution were filtered, washed with water and crystallized from a mixture of ether and alcohol. The hydrazone thus obtained formed pale-yellow plates readily soluble in ether, and chloroform, but sparingly so in other organic solvents ; m.p. 149°-150°C.

*Dihydro Euphoron* — A solution of 5 gm. of pure *Euphoron* in 100 c.c. of glacial acetic acid was shaken with 0·5 gm. of platinum oxide catalyst ( Roger-Adams ) in a long-necked flask connected to a graduated hydrogen reservoir. The absorption which was rapid in the beginning was complete in 8 hr. The glacial acetic acid was distilled off from the solution below 70°C. *in vacuo*, and the residue crystallized from alcohol. Dihydro *Euphoron*, thus obtained in nearly theoretical yield ( 5 gm.), formed triangular plates melting at 65·5°C. and was readily soluble in chloroform, ether, alcohol and acetone. It decolourized alkaline permanganate solution and bromine in chloroform, indicating its unsaturated character.

ANALYSIS

| Required for | C. % | H. % | Volume of H. absorbed for one double bond lit./100 gm. |
|---|---|---|---|
| $C_{27}H_{44}O$ | 84·37 | 11·47 | 5·83 |
| $C_{29}H_{48}O$ | 84·47 | 11·65 | 5·44 |
| $C_{31}H_{52}O$ | 84·53 | 11·81 | 5·09 |
| *Found* | 84·59 | 11·59 | 5·00 |

*Dihydro-Euphoron Oxime* — The oxime of the dihydro ketone was prepared by following the procedure described for the preparation of the oxime of *Euphoron*. It formed colourless flakes soluble in ether and chloroform, less so in alcohol and acetone, and nearly insoluble in petroleum ether ; m.p. 205°C.

ANALYSIS

| Required for | C. % | H. % | N. % |
|---|---|---|---|
| $C_{27}H_{45}ON$ | 81·19 | 11·28 | 3·51 |
| $C_{29}H_{49}ON$ | 81·51 | 11·48 | 3·28 |
| $C_{31}H_{53}ON$ | 81·75 | 11·65 | 3·08 |
| *Found* | 81·40 | 11·45 | 3·28 |

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* The micro analytical values recorded in the present paper have all been determined after drying the substances to constant weight at 50°-60°C. *in vacuo*.

*Euphorol** — A hot solution of the ketone ( 5 gm.) in 250 c.c. of amyl alcohol was fitted with a reflux arrangement and sodium added to it in small bits in the course of about 1 hr. After the completion of the reaction the amyl alcohol solution was washed free of alkali with water and the solvent distilled off *in vacuo.* The residue was then crystallized successively from petroleum ether, a mixture of methyl alcohol and ethyl acetate and from alcohol, when *Euphorol* was finally obtained in the form of colourless needles in a rather poor yield ( 1 gm. ). It melts at 109°C. and is more readily soluble than the ketones in ether, alcohol and other organic solvents.

*Euphorol Acetate* — To a solution of *Euphorol* ( 0·5 gm. ) in 10 c.c. of dry pyridine was added 5 c.c. of acetic anhydride. After warming over the water bath for half an hour, the solution was cooled, diluted with ether, and successively shaken out with dilute solutions of hydrochloric acid and sodium hydroxide, and finally with water. It was then dried over anhydrous sodium sulphate and the solvent removed on the water bath. The residue was repeatedly crystallized from alcohol when *Euphorol* acetate was obtained in the form of fine needles, readily soluble in ether and chloroform in the cold and in alcohol, acetone, and petroleum ether in the hot ; m.p. 107°-108°C. ( yield, 0·2 gm. ).

*Euphorol Benzoate* — A solution of *Euphorol* ( 0·25 gm. ) and benzoyl chloride ( 1 c.c. ) in dry pyridine ( 10 c.c. ) was warmed on the water bath for about half an hour, cooled and diluted with ether. The ethereal solution was shaken out first with a dilute solution of hydrochloric acid, then with dilute alkali, and finally with water. It was then dried over anhydrous sodium sulphate, filtered and freed from the solvent. On dissolving the residue in hot alcohol and leaving the solution overnight, *Euphorol* benzoate was obtained in colourless needles, fairly soluble in ether and chloroform, less so in alcohol, and melting at 114°C. ; yield 0·15 gm.

*Resinous Component of Dried Latex* — After separation of the total crystallizate ( crude *Euphoron* ) from the alcoholic solution of the ethereal extract of the dried latex, the solvent was removed from the mother liquor *in vacuo.* The residue, which constitutes the resin component of the latex, was a brittle, lustrous mass resembling rosin in appearance, and melting within the range of 65°-75°C. When distilled with superheated steam, at 250°C., it yields a small quantity of a viscous liquid. The main bulk of the resin, however, remained in the flask as a dark, brittle, lustrous mass melting at 125°-140°C. Both the steam distilled and the original resins are soluble in benzene, turpentine and linseed oil. The physical and chemical constants of the various resinous products obtained from the latex are given below.

**ANALYSIS**

| | ETHER EXTRACT | TOTAL RESIN | RESIDUE AFTER TREATMENT WITH SUPERHEATED STEAM | HIGH TEMPERATURE STEAM DISTILLATE |
|---|---|---|---|---|
| Yield on the wt. of the dried latex, % | 75 | 50 | 45 | 5 |
| Ash, % | 0·1 | 0·12 | ... | ... |
| M.p.,°C. | 55-62 | 65-75 | 125-40 | ... |
| Acid value | 21·6 | 17·7 | 12·3 | 9·1 |
| Ester value | 67·9 | 66·6 | 106·6 | nil |
| Iodine value | 113·0 | 131·0 | 55·9 | 62·9 |
| Carbonyl value* | 141·0 | 112·0 | ... | ... |

**Acknowledgement**

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

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* Further work on the purification of *Euphorol* will form the subject of a subsequent communication.

* Determined by the method described by Cottrell in *Paint Technology*, 1944, **9**, 73.

# An Ideal Encrustation of Lac

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DURING November 1923, a fresh piece of stick lac was collected at Bangalore. It belonged to the insect *Lakshadia communis* growing on *Acacia concinna*, which is a common plant there, but nevertheless only rarely attacked by the lac insect. Since no lac encrustation similar to it was collected before or subsequently, it has been the very best I ever collected. Even in the literature no record similar to it is mentioned.

I have shown elsewhere[1] that at the end of the monsoon season, in November, only small chunks of lac are found. From these pieces the larvae emerge as winged males. A few that get scattered, usually only 1 larva on a branch, change their sex, become crown shaped in form and give rise to a normal generation of young ones, about 13 females to 1 male. The progeny that emerges from single cells can never be very large and explains the occurrence of stick lac only as isolated chunks. The preponderance of males regularly following the monsoon season makes *L. communis* unfit for cultivation, a fact not recognized in the literature even to this day. A parallel occurrence of males in the Indian *Cochenille* insect likewise prevented its being utilized for cultivating the *Cochenille* insect in this country. I believe it is the insect originally introduced from South America where it was found wild and thus this species compares well with *L. communis* found all over South India.

When the young ones emerge from the crown-shaped cell, they settle down in the proximity of the dead mother insect. It is, therefore, commonly found that the empty cell gets incorporated with the fresh mass of encrustation produced by the living generation, as has been previously[1] illustrated. But the crown-shaped cell may remain apart from the main encrustation which has been likewise shown before[1]. The dry cell may even drop away, leaving no trace of the mother insect which has given birth to the generation forming chunk of lac. When we consider probabilities rather than possibilities, the piece of stick lac illustrated here was formed by a generation arising from a single crown-shaped mother cell which dropped off before it was collected.

When freshly collected, it was kept in a glass jar for breeding parasites. *L. communis* in Bangalore is most liable to the attack of *Eublemma amabilis* and next to it of *Erencyrtus dewitzi*. For about 8 days no parasite was observed. When the larval swarming became intense, the piece of stick lac was removed for infecting a branch of *Guazuma tomentosa* and left there for 24 hr. and after this period replaced in the breeding cage. The larvae that swarmed from the brood lac became all winged males which was expected. Not a single parasite was reared and as far as I know no such immunity has been established before. After 6 weeks the encrustation was removed when all insect life appeared to have been dead.

In May 1947 a photograph was taken of it and illustrations were also made which are being offered to supplement it. The dry encrustation with the twig weighed 40·425 gm. It was left overnight in alcohol which dissolved the lac resin and left the twig and individual insects free. The twig was dried at 80°C. and weighed (8·092 gm.). Insects were 1,335 in number. Thus the average weight of a dry lac cell, growing in a colony and feeding during the monsoon season, was 0·0272 gm. During the dry season more lac is secreted, likewise when insects are growing isolated. A single cell of *L. communis* collected in May 1947 from *Anona squamosa* in Hyderabad weighed 0·031 gm. For *Lakshadia nagoliensis*, feeding on *Schleichera trijuga*, during monsoon season, an average dry insect and its secretion weighed 0·0188-0·0194 gm. As compared with *L. communis* the individual insects of *L. nagoliensis* were rather small but when their body surface was considered, *Nagoli* lac insects appeared to secrete more lac.

The 1,335 insects that formed the colony-encrustation were examined carefully but indicated any injury, even in the past, from parasites. No shrivelled bodies of dead insects were observed such as would have indicated premature death. But even if 1,335 individuals had survived, it does not indicate that the progeny did not originally consist of more larvae. The generation that forms chunks of lac born of crown-shaped cells, generally consists of 13 females to 1 male. Thus for 1,335 female insects there must have been at least 126 males, so that the total larvae that issued are 1,461. Now, a normal female cell delivers 500 to 750 larvae ; the crown-shaped cell, on the contrary, has been known to yield 1,500 larvae ; in fact 1,505 have been actually counted. Taking 1,500 larvae as the maximum figure, 1,460 represent female and male larvae that survived to complete their life cycles, whereas 40 must have died prematurely. There is no record in the literature even to show that 1,335 individuals were derived from 1 mother cell. Such a large progeny can only come from a crown-shaped mother insect while no normal female cell can ever compete with it and literature is silent over crown-shaped cells.

The encrustation was photographed somewhat reduced and then enlarged to reproduce the object in natural size as shown in Fig. 1a. Fig. 1b gives a pen and ink outline to bring out its important features ; it also represents the encrustation ( natural size ). Fig. 1c shows the opposite or dorsal view of the same specimen. When a twig grows vertically, larvae settle all around it ; their final product, then, is a cylindrical encrustation of lac. If the twig is inclined, the population on the side facing the earth is more intense than on the other side of the twig ; the final encrustation is thicker on the side facing the earth than on the other half of the twig. If the twig is still more inclined or almost horizontal, then the larvae settle only on the lower half leaving the upper surface of the twig free from colonization. Now, all grades of inclination are found and, correspondingly, lac encrustations may be perfectly cylindrical sorrounding a vertical twig or may be a semi-cylindrical crust on the lower half of a horizontal one or may be any intermediate between these extremes. A glance at Fig. 1c shows that the upper end is approaching a cylinder with only a narrow line of partition between the two sides of the encrustation meeting together, while the lower end has a distinct separation with only three-fourths of the circumference of the twig surrounded with lac. In such a small twig degrees of inclination do not explain the difference noted above. Lac insects are phototropic so that colonization is often more intense towards the end receiving more light. Such a clear difference is not apparent on studying Fig. 1b representing the side of encrustation which was facing the earth. What is specially to be noted in Fig. 1c is that the encrustation formed by rows of insects on either side of the twig shows a partition between lac from either side which has not fused, a feature special to *L. communis*. It secretes lac poorly and thus the secretion does not easily fuse.

As early as 1567 Clusius also reproduced a wood engraving where stick lac envelopes a twig without forming a cylinder. It has been reproduced as Fig. 1 of a recent communication[2]. The same paper shows in Fig. 3 a stick lac of *L. communis* where the two sides of an encrustation meet but do not fuse. The object illustrated here ( FIG. 1c ) thus supports the illustration by Clusius.

When we compare an insect belonging to *L. communis* with that of *L. nagoliensis*, the former is larger in size but, compared with its body surface, the secretion of lac is less. In simpler words, *L. communis* has a thinner coat of lac to cover its larger body while *L. nagoliensis* has a much thicker one for a smaller body. When two rows of insects belonging to *L. communis* meet, their secretions, being thin, do not fuse, which easily happens in the case of *L. nagoliensis*. The difference in stick lac of these two lac insects has been previously explained[3].

It remains to add that the diameter of the twig of *Acacia concinna* in Figs. 1a, 1b, 1c was 0·6 cm. at the top, 0·7 cm. in the middle and 0·9 cm. at the basal end. Previous observations have shown that shoots with 0·75 cm. are ideal for lac infection. The present encrustation also confirms this standard.

Part of the work was done at the *Research Institute, Osmania University*, Hyderabad ( Deccan ) with the help of Mr. Kader Mohiuddin, to whom my thanks are due. Thanks are also due to Mr. Giriraj for the pen and ink drawings.

### Summary

A specimen of stick lac has been recorded from *Acacia concinna* as host, belonging to the insect *Lakshadia communis* found at

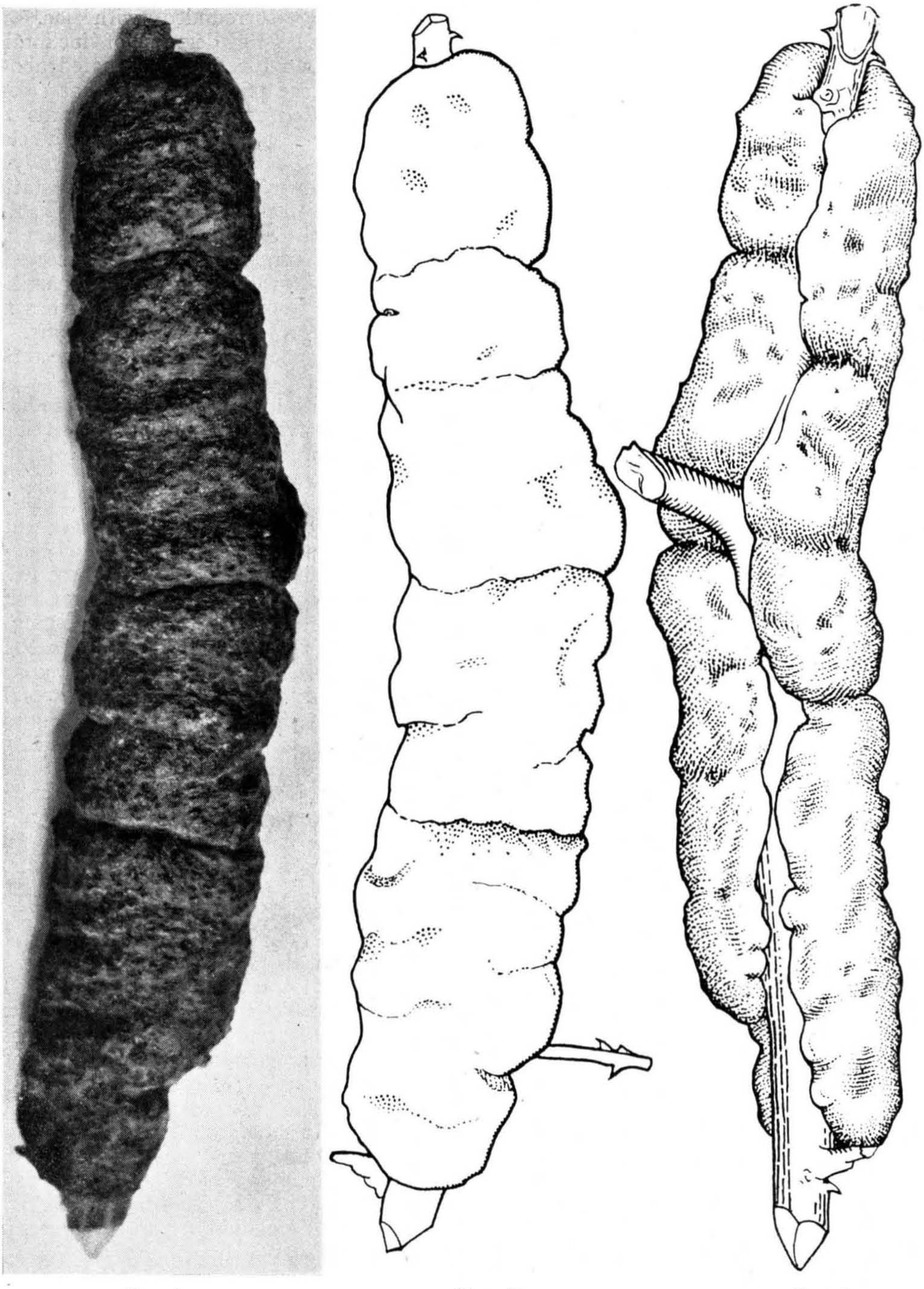

FIG. 1a FIG. 1b FIG. 1c

FIG. 1a — STICK LAC OF *Lakshadia communis* FORMED BY A COLONY OF 1,335 INSECTS BORN OF THE SAME MOTHER INSECT. NATURAL SIZE. FIG. 1b — SAME AS FIG. 1a ; ALSO NATURAL SIZE. FIG. 1c — THE SAME OBJECT AS IN FIG. 1a SEEN FROM THE OTHER SIDE. THE ENCRUSTATION AT ITS BASE SHOWS A DISTINCT NARROW GAP WHILE AT THE TOP LAC SECRETION FROM THE OPPOSITE ROWS OF INSECTS IS ACTUALLY TOUCHING EACH OTHER BUT HAS NOT FUSED. NATURAL SIZE.

Bangalore, growing there during the monsoon season. 1,335 individuals formed the encrustation which together weighed 32·3 gm. The entire generation was born of one crown-shaped mother cell. The same generation also comprised probably of 126 males so that the entire progeny, including those that may have died, consisted of about 1,500 larvae. The encrustation was ideal in so far as no bigger colony, born of one mother insect, has been recorded. It was again ideal in its being free from all parasites.

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# Letters to the Editor

## A DEPOSIT OF MASSIVE GARNET IN BIHAR SUITABLE FOR THE MANUFACTURE OF ABRASIVES & GRINDING MATERIALS

INDIA DEPENDS UPON FOREIGN COUNTRIES for the supply of materials required for cutting, polishing, crushing and grinding. Such articles are commonly required in almost all industries. Corundum sheets and papers are being manufactured on a small scale in the Mysore State. The raw material required is rare and hard to win, and a large-sized industry can be started only in a place where the raw material is available in assured commercial quantities.

A large deposit of massive garnet ( calderite-manganese-iron garnet ) of pinkish-black colour has been recently discovered on the bank of River Chandan near the villages Nawadih and Chandan in the Chandan sub-zemindary of the Banaili Raj, now worked by the *Mining & Industries Ltd.*, Bhagalpur. It is by the side of the Katoria-Deoghar Road and 10 miles east of Deoghar. An out-crop of $6' \times \frac{1}{4}$ mile is visible in bold relief surrounded by eroded fields. Its development awaits the installation of an improved garnet mill as employed on the continent.

The garnet is completely free from deleterious substances, and is the purest so far discovered on so large a scale in India. The field-test for garnet to be used as abrasive is that the particles should break off and offer a fresh surface after it has been once used. This deposit is extensive enough to warrant the establishment of a garnet mill.

Deoghar and Jasidih being the nearest railheads appear to be suitable places for this industry at present, but Jasidih being on the main line is more suitable. If the Sultanganj-Deoghar railway materializes, it will pass through this area.

Garnet-coated abrasives are used in the form of belts, discs and small sheets. 90 per cent of the garnet is used in the manufacture of abrasive-coated paper and cloth and the remainder is sold as low-priced material in the form of loose grains for surfacing and polishing marble, slate, soapstone and also in sand-blast operations. A small amount is used for the surfacing of plate glass. Garnet is also used for rubbing down painted surfaces of cars and for the manufacture of small discs employed in dental work. Garnet paper is used in leather, rubber and celluloid industries.

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## FISH POISON FROM THE WOOD OF *ADINA CORDIFOLIA*

IN THE COURSE OF A STUDY OF NATURAL and synthetic insecticides and fish poisons, adinin, the crystalline component of the wood of *Adina cordifolia*, has been examined for its insecticidal and fish-poisoning properties. Adinin and its methyl ether are found definitely toxic to fish, the methyl ether being more potent than adinin itself. It was, therefore, of interest to make a detailed study of their constitution.

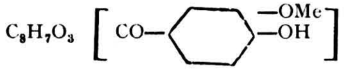
Lal and Dutt[1] reported the isolation of a yellow colouring matter from the wood of *Adina cordifolia*, which they named "adinin". It crystallized from glacial acetic acid as small bright-yellow prisms, which on heating darkened at 195°-196°C., shrunk at 200°C. and decomposed without melting above that temperature. The molecular formula of adinin was given as $C_{16}H_{14}O_7$.

In the present work, various modifications in the procedure for the isolation and purification of adinin have been examined. Extraction of the wood material with boiling methylated spirit ( repeated 3 times ) brought the adinin into solution. The semi-solid residue after the removal of solvent was washed with hot water, and dissolved in acetic acid in which adinin is sparingly soluble. Other coloured impurities were eliminated by washing with petroleum ether. Precipitation from absolute alcoholic solution by the addition of a large quantity of ether gave adinin as a pale-yellow solid which melts sharply at 235°C. Analysis gave the formula $C_{16}H_{14}O_6$ ( mol. wt., by Rast's method, 294 ).

Adinin contains 1 methoxyl group as determined by the micro-Zeisel method. Its methylether ( mol. wt. 328 ) has 2 methoxyl groups showing the presence of hydroxyl group. It also gives a mono-acetyl derivative. Boiling the methylether with acetic anhydride and pyridine does not bring about any change showing that adinin does not contain any other alcoholic hydroxyl group. It does not contain a sugar residue. Nor-adinin does not show any colour change in alkaline buffer solutions. The colour reactions in general seem to indicate that adinin does not belong to the flavone group.

Adinin readily forms a dinitrophenyl hydrazone on heating with a solution of 2 : 4 dinitrophenyl-hydrazine in hydrochloric acid. The ease with which this derivative can be prepared shows the existence of ketonic carbonyl group.

Out of the 6 oxygen atoms in the adinin molecule, 1 is present as a methoxyl, another as a phenolic hydroxyl and the third as a carbonyl group. The nature and function of the other oxygen atoms is not yet clear. Fission of adinin methyl-ether with 8 per cent absolute alcoholic potash and oxidation with neutral permanganate in acetone medium yielded veratric acid. This shows that the hydroxyl and methoxyl groups in the adinin molecule are present in *ortho* positions to each other in one benzene ring and this is possibly part of ketonic structure like



The location of the phenolic hydroxyl *para* to the carbonyl may account for the appreciable solubility of the compound in sodium carbonate.

Further work is under progress. The author's thanks are due to Prof. T. R. Seshadri of the Andhra University for suggesting the work and for his valuable guidance.

N. PRABHAKARA RAO

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*Waltair*
*June* 30, 1948

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