Journal of Scientific & Industrial Research

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- High pressure ammonolysis of ethylene dichloride
- Mineral matter separation in coal by froth flotation

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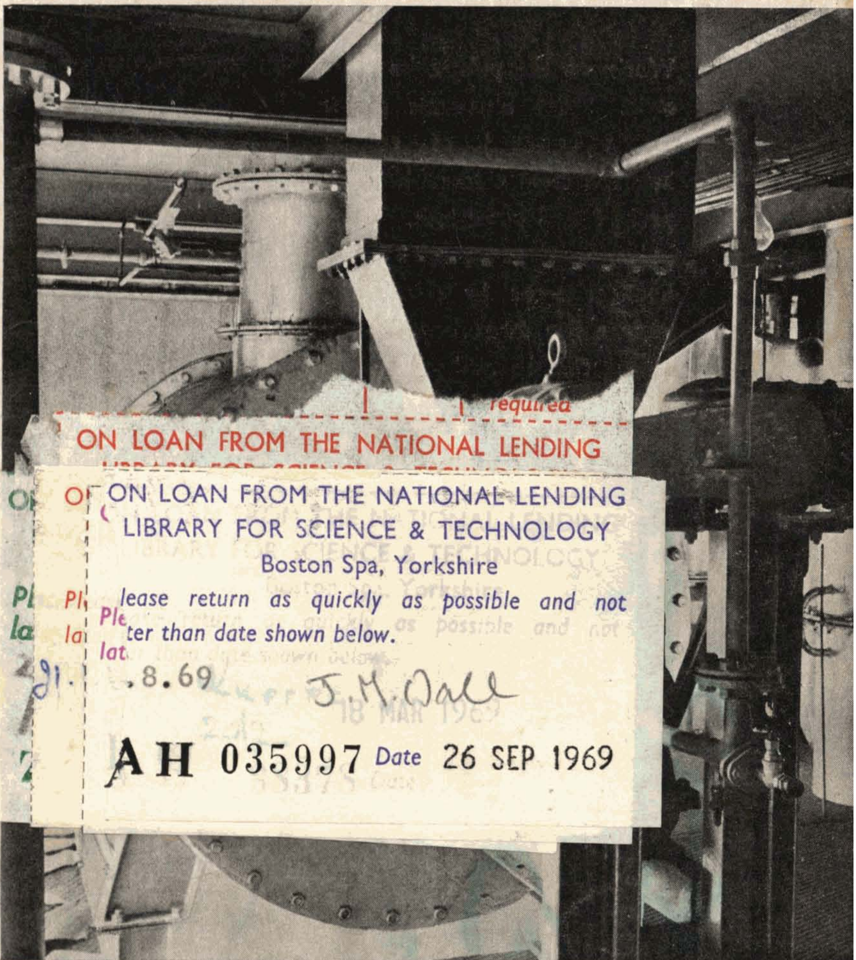

THE COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, NEW DELHI

J. sci. industr. Res., Vol. 17A, No. 7, Pp. 259-306 JULY 1958

Journal of Scientific & Industrial Research

Vol. 17A, No. 7, JULY 1958

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COVER PICTURE

Economic generation of synthesis gas, which accounts for about 70 per cent of the cost of production of synthetic fuels and chemicals, from inferior coals is engaging the attention of the Central Fuel Research Institute, Jealgora. The cover picture shows a section of the Kopper-Totzek coal gasification plant, one of the four types under trial, erected at the Institute at a cost of about Rs. 2·5 lakhs. The capacity of the plant is 100 kg. coal per hour. Parts of the plant have been generously supplied, free of cost, by Messrs H. Koppers of Germany.

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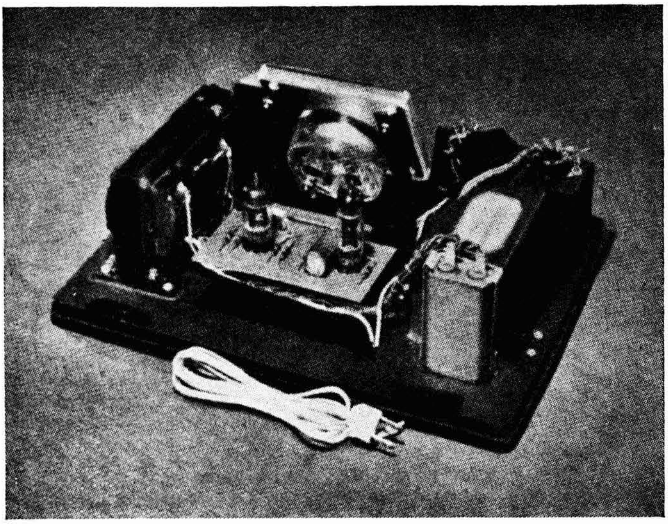

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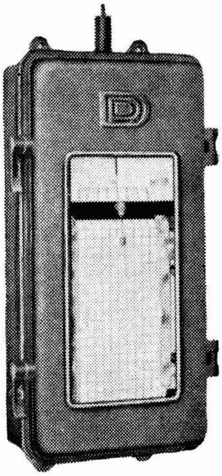

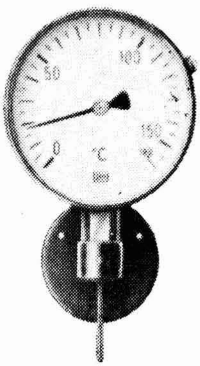

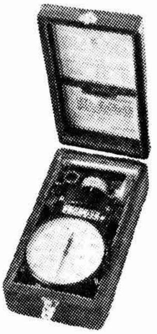

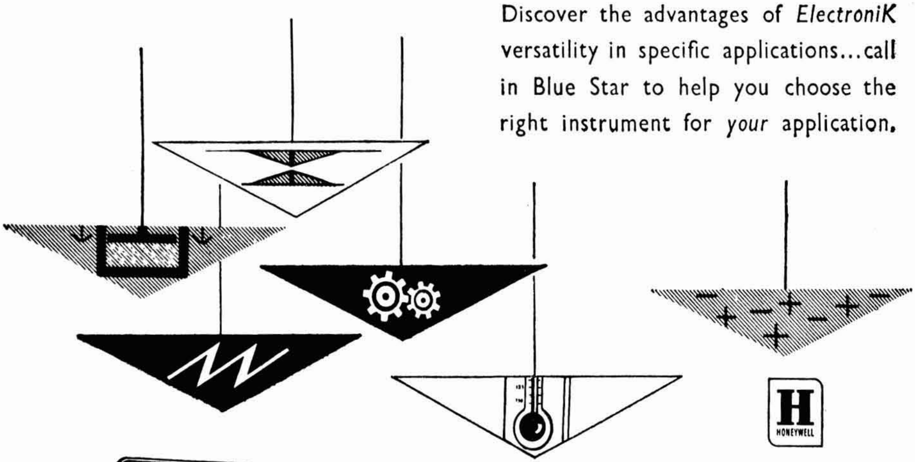
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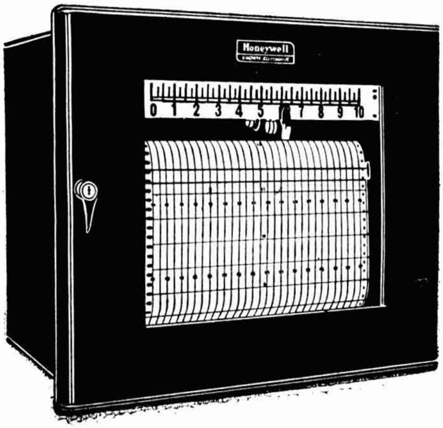
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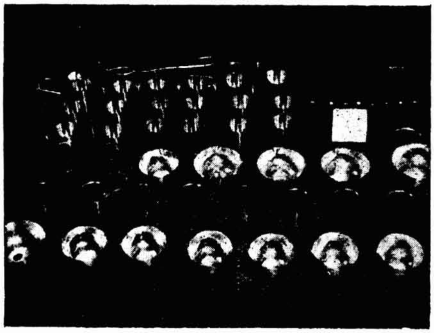

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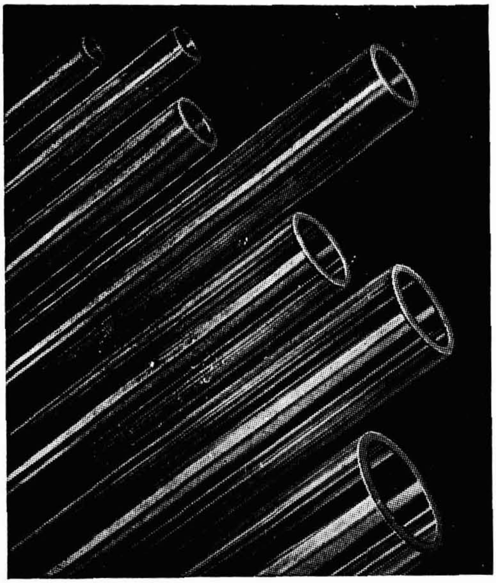

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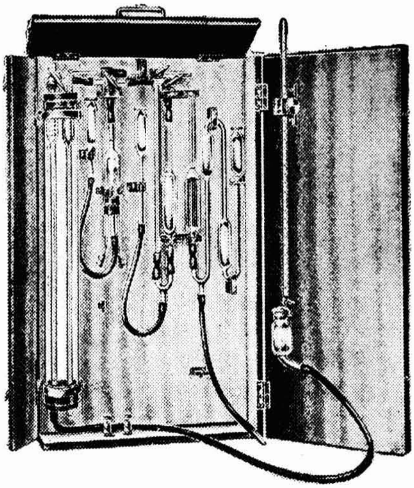

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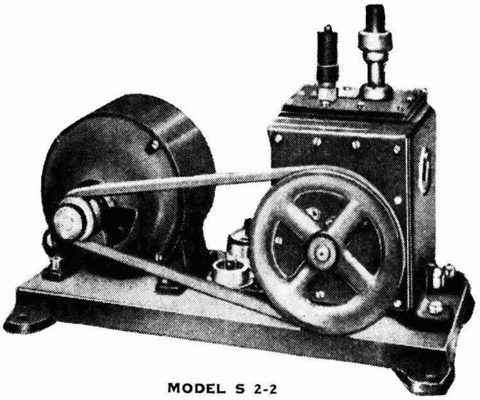
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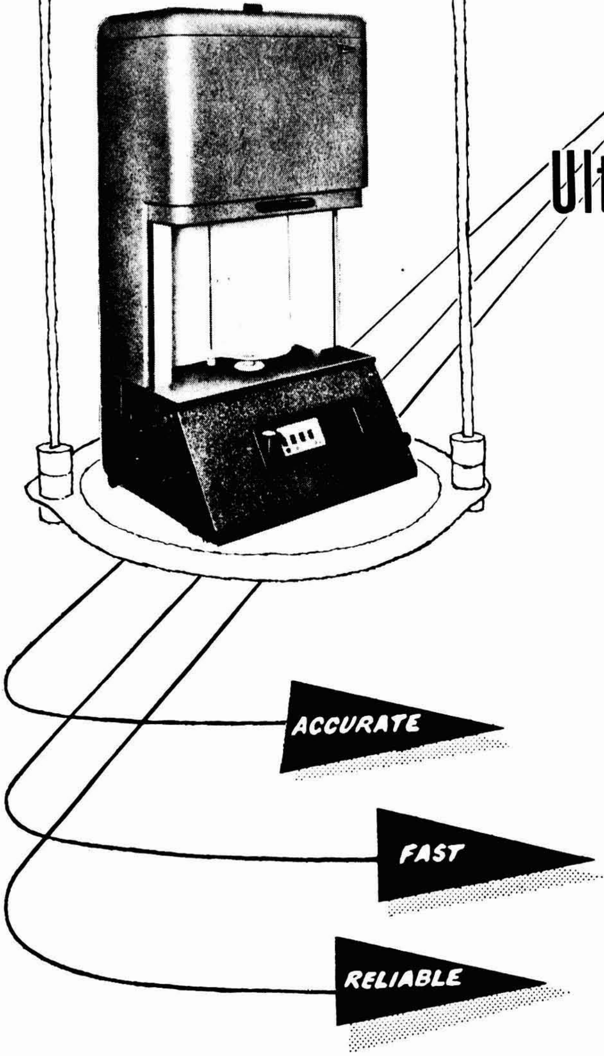
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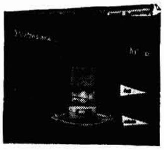

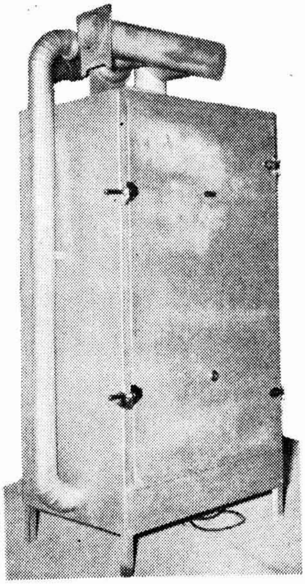

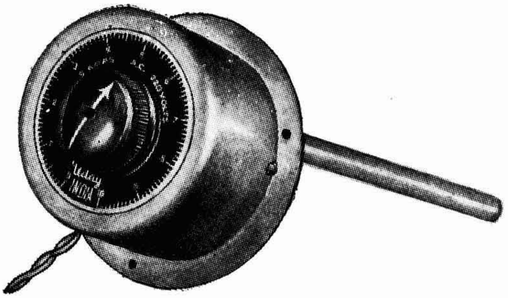
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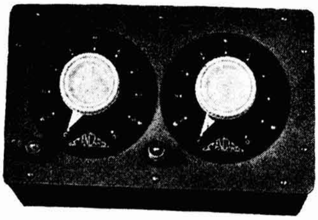

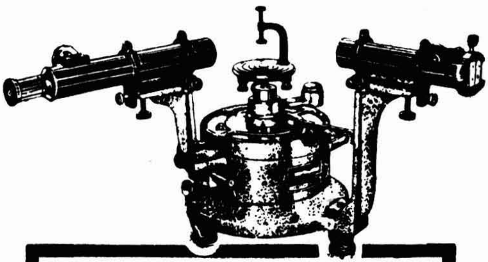

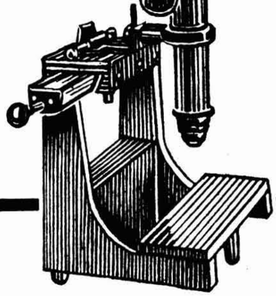

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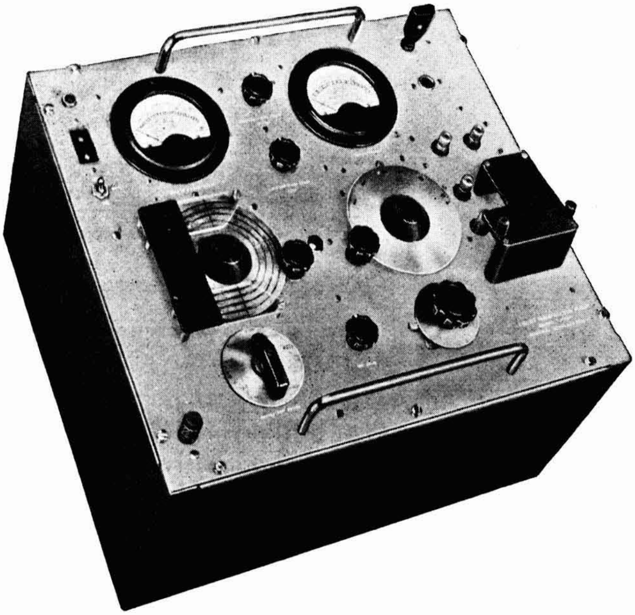

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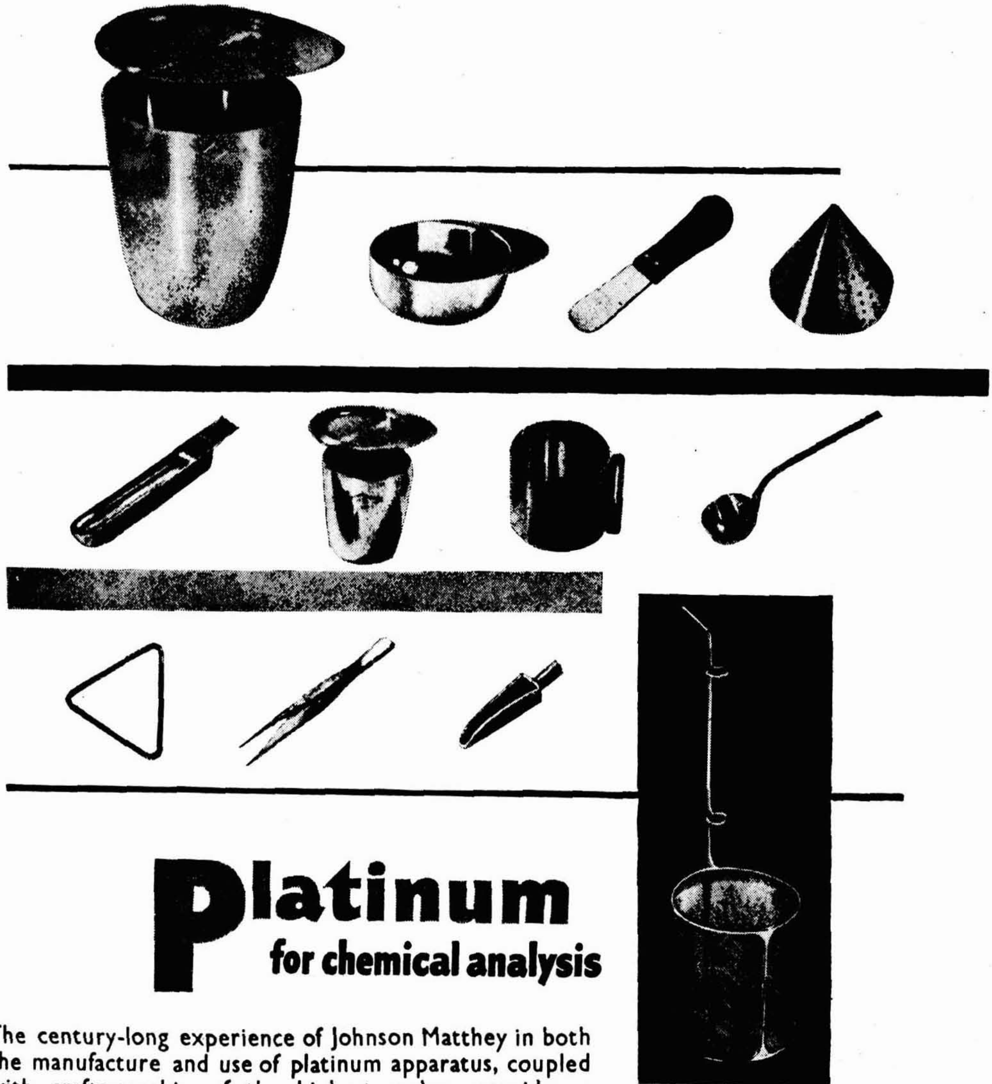

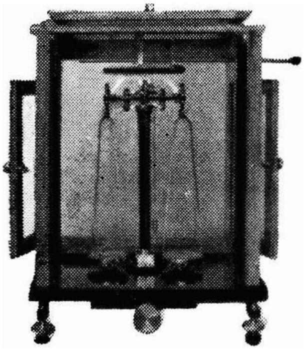

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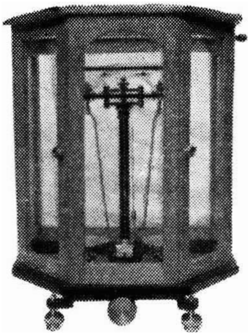

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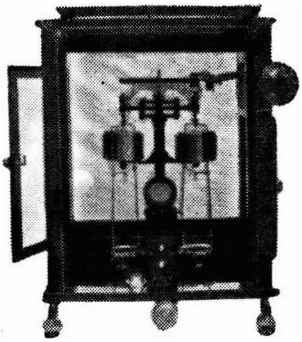

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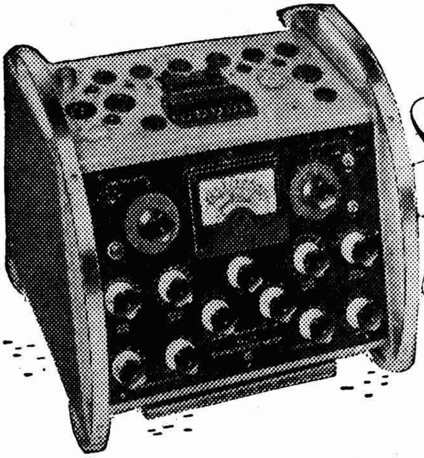

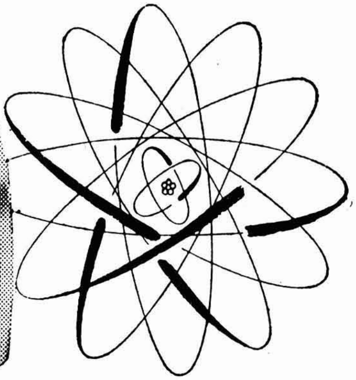

Literature Citations in Scientific & Technical Periodicals — A Survey*

AN investigator is often confronted with a variety of problems in his literature search because of the diverse ways literature citations are made in scientific and technical periodicals. This is mainly due to divergent instructions issued to authors by editorial boards of periodicals specifying the manner in which literature citations are to be made. The problems created by the lack of a standard practice in this regard have been receiving the attention of editors, bibliographers, documentationists and others.

A survey of a representative selection of 200 Indian and foreign scientific and technical periodicals was recently undertaken with a view to obtaining information on the patterns of literature citations. The results presented in the following paragraphs reveal the diversity in existing practices and emphasize the need for standardization of a vital aid for literature search.

Components of a citation

The component parts of a citation are: Author; Title; Name of Periodical; Year; Volume (Number); and Page(s).

Author — The name(s) of author(s) occupies the leading position. In the case of author entries, the surname is used first, followed by the initials. Of the 200 periodicals surveyed, 156 periodicals follow this system; the *Journal de Physique et le Radium* gives initials within parenthesis. When there are more authors than one, the initials are followed by surnames in the case of the second or subsequent author entries. This practice is followed by 27 periodicals. Where there are three or more author names, all the names are cited in 183 periodicals; in the case of 10 periodicals, only surnames are given; and 7 periodicals use *et al.* after the first author's name.

Type variations are not unusual. While 101 periodicals use caps and small caps for printing authors' names, 90 adopt caps and lower case. The names are printed in boldface type in 6 periodicals and in italics in 3.

Title — Is the title of an article a necessary component of a literature citation? Some argue that a citation without the title is defective in so far as it deprives the reader of the means by which he can immediately ascertain for himself its relevance to his field of interest. Others, however, are of the opinion that the omission of the title would reduce the space occupied by the citation and also the cost of printing. The survey has revealed that 98 periodicals give the titles and 88 do not. Of the group of periodicals dealing with biological sciences 75 per cent give the title in full, while only 23 periodicals in the group of general and physical sciences follow this practice. In the mathematics and engineering periodicals, however, the title forms a part of the citation as a rule.

The title follows the author's name in the citation and is printed in italics or in the type used in the text; in the latter case, it is often inscribed within inverted commas. Three periodicals, namely *Journal of Applied Mechanics*, *Electrical Engineering*, and *Transactions of the American Society of Mechanical Engineers* give title first and the author's name next.

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*From a paper presented by S. Dutta and T. S. Rajagopalan, Indian National Scientific Documentation Centre, New Delhi, to the Indian Standards Convention held at Madras in December 1957.

Name of the periodical — The name of the periodical is invariably abbreviated in the citation. The abbreviations used, however, are very varied. The recommendations of the International Organization for Standardization (ISO R4-1953) are not followed by periodicals in many countries. Identification of a periodical is thus made difficult by the diverse ways in which the title of a periodical is abbreviated. Other variations met with are: where a reference is made to the very periodical in which the article appears, some give the full name of the periodical, e.g. the *British Medical Journal*; some use *this* Journal or *this* Bulletin. Instances of periodicals following the latter practice are the *Bulletin of the Chemical Society, Japan*; *Journal of the Electrochemical Society*; and *Journal of the American Chemical Society*. Some periodicals use such skeleton abbreviations as DAN for *Doklady Akademii Nauk SSSR*; *J* for the *Journal of the Chemical Society*, London; BER for *Berichte der Deutschen Chemischen Gesellschaft*, Berlin. This practice is misleading; there are many German periodicals the titles of which begin with Berichte.

The names of periodicals are usually given in italics, but quite a large number of periodicals (80 out of 200) use the same type face as the text. *La ricerca scientifica*, Rome, is the only journal which uses inverted commas for the title of the periodical.

Year, volume and page — The sequence followed in this respect varies widely. While 64 periodicals follow the sequence ' volume-page-year ', the sequences ' year-volume-page ' and ' volume-year-page ' are followed by 24 and 25 periodicals respectively. In those periodicals which follow the alphabetic arrangement in the list of references at the end of the article, and where the citations are indicated in the text by author and year (82 periodicals), year is given immediately after the author, followed by the name of the periodical, volume and page; in such cases the year is invariably given within parenthesis. Five periodicals do not follow any definite sequence.

The year is printed in Arabic numerals and is generally given within parenthesis. In 141 periodicals, the volume number is given in bold face, and in 16 others, in italics. When printed in the same type as the text, some use the abbreviation v. or vol. before the volume number given in Arabic numerals. Roman numerals are seldom used to indicate volume number.

The types used for page numbers usually conform to the types used for the text. In 17 cases, however, the page number is preceded by p. or pp. The practice of giving inclusive pages is followed by 92 periodicals regularly and by 5 occasionally. *Structural Engineering* seldom gives the page number while furnishing other relevant information including the title.

Placement and arrangement of references

The survey has revealed varying practices in regard to the location of references in an article. References may be given at the end of an article, at the foot of a column or a page and in the body of the text. When citations are given at the end, the arrangement is usually alphabetic according to authors' surnames; they may be also arranged serially in the order in which they are referred to in the text. In some cases, particularly in review articles, the chronological order is favoured. When citations are given at the foot of a column or page, they are indicated by serial numbers in the numerical order mentioned in the text.

Citations are placed at the end of articles in 179 publications, at the foot of a column or page in 18, and in both ways in two. The *Journal of the Indian Chemical Society* is the only instance in which references are incorporated in the text.

Among the 181 periodicals in which citations are listed at the end of articles, 105 follow the alphabetic arrangement, 68 follow the serial arrangement, 7 adopt both alphabetic and serial arrangements and one the chronological order. Eighty per cent of the periodicals in the general and physical sciences group, in which the references are given at the end of the article, follow the serial arrangement; 90 per cent in the biological sciences group follow the alphabetical arrangement.

Citations are indicated in the body of the text by one of the following ways: (i) by author's name and year within parenthesis; (ii) by author's name and serial numbers; and (iii) only by serial number. Serial numbers are given either in parenthesis in the same line, as superscripts in parenthesis, or simply as superscripts. Of the 200 periodicals analysed, 82 follow (i). The number of periodicals using the other two methods are respectively 21 and 94; three others do not follow any of the methods consistently.

The problem of adopting a standard for literature citations in scientific periodicals was discussed by a Joint Committee set up in 1950 by the Council of Scientific & Industrial Research and the National Institute of Sciences, India. The Committee recommended that citations should be given in the following manner: DAKSHINAMURTY, P., RAO, G., RAGHAVACHARY, M. V. & VENKATARAO, C., *J. sci. industr. Res.*, **16B** (1957), 340-44. The reference is indicated in the text by numbers placed above the line (superior), the references being cited in a standard form against the appropriate number in the order in which they occur in the text. The sequence of volume, year and page as given in the above citation is followed by a few journals only. With respect to abbreviating the names of periodicals, it is recommended that the *World List of Scientific Periodicals* be followed. The practice of giving the year along with the volume number is adopted because it readily gives the reader an idea as to the period of publication of the paper which the volume number alone fails to convey. Some periodicals do not use a volume number and the only way of identifying a particular volume is by the year. Since the majority of periodicals give the volume number, inclusion of the year provides additional information, and is, therefore, given in parenthesis.

The problems connected with bibliographic references were discussed at the Indian Standards Convention held in Madras last December and it was agreed that the ISI Committee on Documentation should go into the matter and standardize the method of presentation of literature citations. The formulation of a standard practice and its implementation by all organizations concerned with the handling and publication of scientific literature will help authors, literature consultants and documentationists alike.

Fourth Indian Standards Convention

THE FOURTH INDIAN STANDARDS CONVENTION will be held in New Delhi from 24 to 29 November 1958. The convention will comprise the following 8 sessions: Consumer Standards and Certification; Metric Conversion; Standardization and Productivity; Standardization and Export Promotion; Safety and Health Standards; Sampling for Quality Evaluation; Steel Economy and Alloy and Special Steels; and Modular Planning, Design and Construction in the Building Industry.

Original papers (not exceeding 3000 words) to be presented at the Convention for discussion should be sent to the Organizing Secretary, Indian Standards Convention 1958, Manak Bhavan, 9 Mathura Road, New Delhi 1, by 15 August 1958.

Study of the Atmospheric Radio Noise at 27 & 100 kc/s. at Delhi

D. K. SACHDEV
Radio Propagation Unit, National Physical Laboratory, New Delhi

(*Manuscript received* 8 *May* 1958)

A preliminary report of the observations on VLF atmospheric radio noise being carried out by the Radio Propagation Unit, National Physical Laboratory of India, New Delhi (28·5°N, 77°E), is presented. Measurements are currently made at two frequencies, namely 27 kc/s. and 100 kc/s. Diurnal and seasonal variations of the radio noise indicate appreciable ionospheric attenuation during daytime, and a summer afternoon maximum, possibly associated with local thunderstorms. Noise intensity falls rapidly during the early morning hours, the 'sunrise time' differing greatly from one day to another. The sunrise fall is sharper at 100 kc/s. than at 27 kc/s. Long period (~2 hr.) fading is observed on certain nights, and is believed to be associated with disturbed conditions in the ionosphere.

Particular attention has been given to the study of sudden enhancement of atmospherics (S.E.A.) for which the observations were initially undertaken. It is found that enhancement is observed at both frequencies at the time of a solar flare; the effect at 27 kc/s. is normally larger and earlier than at 100 kc/s. However, there is no one-to-one correspondence between the S.E.A. and the solar flare. Further, cases have been observed when the S.E.A. at 100 kc/s. is found to be larger than at 27 kc/s. From the present observations it is suggested that transition from enhancement to fadeout occurs somewhat beyond 100 kc/s.

AT frequencies below 100 kc/s., known as the very low frequency region, it is becoming increasingly popular to use atmospheric radio noise for studies on ionospheric propagation. The advantages of instrumentation are obvious, and, although interpretation is not always easy, worthwhile results can be achieved in the study of VLF propagation and of the ionospheric effects of solar flares. In the former sphere, extensive investigations have been carried out at Cambridge by Gardner[1], Bowe[2], Pierce[3] and others over frequencies of 3·5 kc/s. to 50 kc/s. These observations have yielded a frequency-dependent attenuation factor with a maximum around 3 kc/s. In the latter sphere, a great volume of data has accumulated over the past years, indicating the possibilities of using VLF noise observations as a sensitive tool for keeping a continuous flare patrol.

Bureau[4] was the first to notice that atmospherics in the VLF region increase in intensity simultaneously with a flare. He found maximum enhancement at 27 kc/s., a result later verified by Ellison[5] and others. The enhancement, however, turns to fadeout at frequencies below 10 kc/s.[1] and in the higher frequency side similar transition must necessarily occur between 27 kc/s. and 500 kc/s. The latter transition region is still unidentified.

In addition to the propagation aspect of such measurements there is also a meteorological aspect: this consists mainly in locating thunderstorm areas either from sferic fixes using two or more direction finding stations or by a single station system studying the variation of the waveform of an atmospheric pulse with distance and with frequency (supplemented by D-F observation). The former method is extensively used and gives reliable results. The latter is still in the investigational stage, and requires further study.

In addition to these, there is the major (and the more obvious) use of such observations in providing a practical index of noise levels in various regions of the world. While reliable world maps are available in the HF band, those in the VLF and LF regions are highly tentative and mostly incomplete. Studies in this field by the National Bureau of Standards and by individual workers elsewhere[6] with the help of the atmospheric noise recorder have provided in recent years some useful values in this part of the spectrum. However, no survey in this field exists in this part of the world.

Atmospheric noise measurements were undertaken by the Radio Propagation Unit towards the end of 1956 with the following objectives:

(1) to study the ionospheric propagation characteristics of VLF waves;

(2) to investigate in detail the possibility of obtaining a reliable method for flare patrol, and to investigate other aspects of solar-terrestrial relationship;

(3) to examine its existing uses in meteorology and to explore critically the possibility of a single station sferic network; and

(4) to measure the noise levels at Delhi at LF and VLF regions.

The frequencies chosen were 27 kc/s. and 100 kc/s.; the former, because existing knowledge indicates maximum S.E.A. effect at this frequency and the latter, because somewhere near this frequency S.E.A. effect should vanish (Fig. 1). The 100 kc/s. equipment was set up in November 1956 and the 27 kc/s. equipment in May 1957.

In this paper the results obtained so far at these two frequencies are presented and a preliminary analysis of the results has been carried out.

Method of observation and equipment

The details of the equipment used have already been described at length[7]. The aerials used are of inverted-L type. They feed into receivers having stabilized power supplies. The second detector output is amplified by linear differential amplifiers. The individual pulses of atmospherics are integrated by providing a time constant of the order of one minute. Since there is generally a considerable interval between individual pulses, any local VLF transmitter can charge the receiver. This is avoided by providing a suitable fixed bias to the second detector. The interference due to local strong medium-wave station is prevented by using wave-trap circuits at the input.

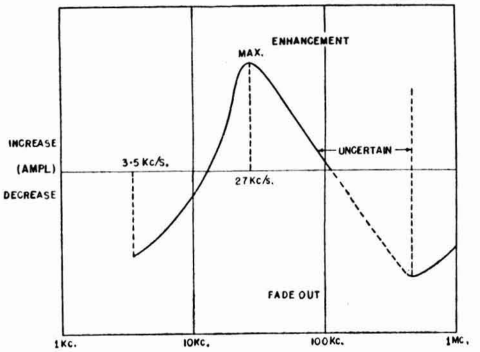

FIG. 1 — FREQUENCY DEPENDENCE OF ABSORPTION EFFECT DURING FLARES

Calibration procedure — It has been shown in the above-mentioned report that, in general, we have the relation:

$$E_f = \frac{E_g}{l_e} \times \frac{Z_A + Z_{in}}{Z_G + Z_{in}} \times \frac{1}{\sqrt{B}} \quad \ldots \text{(1)}$$

where

E_f = field intensity in μV./metre for 1 kc/s. bandwidth;

l_e = effective length of the antenna;

Z_{in} = terminating impedance of the receiver;

Z_G = terminating impedance of the calibrating signal generator;

Z_A = antenna impedance; and

B = bandwidth in kc/s.

At the very low frequencies used, the antenna impedance is almost purely capacitative. So while calibrating, Z_G is made nearly equal to Z_A by using a series capacitor of suitable value. This makes $(Z_G + Z_{in}) = (Z_A + Z_{in})$ which reduces (1) to

$$E_f = \frac{E_g}{l_e\sqrt{B}} \quad \ldots\ldots\ldots \text{(2)}$$

Results

Regular variations — The observations at both frequencies reveal a certain variation which repeats from day to day for one particular season. The noise level is greater at night than during daytime. Also, the average noise level throughout the day is greater during summer than during winter. The increase is mainly due to local storms during summer. The daily variation shows the more or less sudden decrease at sunrise except on days of local storms during morning hours.

Diurnal and seasonal variations — For 100 kc/s., data for about eight months have been obtained and analysed. Data for the months February, March and April 1957 have been taken with a different aerial, accurate measurements with the inverted-L type being not possible. For that period, therefore, only qualitative analysis has been done. For the remaining months (during which simultaneous observations have also been made at 27 kc/s.) a permanent aerial has been used. Figs. 2 and 3 give the diurnal variation of

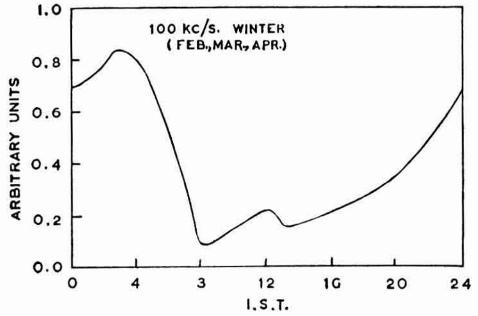

FIG. 2 — DIURNAL VARIATION AT 100 KC/S. (WINTER)

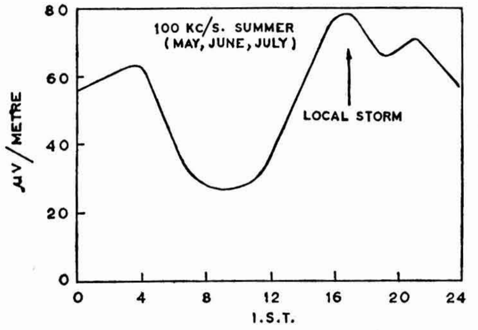

FIG. 3 — DIURNAL VARIATION AT 100 KC/S. (SUMMER)

mean atmospheric noise level for winter and summer at 100 kc/s. Two normalized curves have also been drawn. Fig. 4 gives the average value of the ratio I/I_0 for each month where I is the 24 hr. average for that particular month and I_0 is the maximum noise level in the diurnal variation for the same month. The curve shows an increase of nearly 50 per cent as we go from winter to summer. Fig. 5 shows the contribution of local storms. The diurnal variation curve for summer (Fig. 3) shows an afternoon peak at 1700 hr. which is due to local storms. Storms in the months of June and July are very frequent here and normally start towards noon reaching peak activity during early evening. Fig. 5 shows I_t/I plotted for the six months under consideration. I_t is the noise level at 1700 hr. and I, as before, is the 24 hr. average for each month. This shows a maximum during June. July has a lower value and the readings during August show that the downward trend is continuing. Fig. 6 shows the noise level at 27 kc/s. for the summer months. The field strength is much more than at 100 kc/s. as is to be expected. The local storm effect is equally prominent. Gardner[1] has studied the variation with frequency of the ratio of the mean noise level during daytime to that at night. Fig. 7 shows the curve obtained by Gardner; the X's are points obtained by him. Similar analysis by us give the points represented by O's. Whereas the ratio at 27 kc/s. agrees well with Gardner's value the one at 100 kc/s. could not be confirmed as his values are up to 50 kc/s. Two points are plotted for 100 kc/s., the upper one being for summer and the lower for winter. The summer value is obviously high due to local thunderstorms.

Sunrise effect — As is well known a sharp sudden decrease in noise level takes place near sunrise. The decrease is associated with the increase of absorption in the lower ionosphere. If the source of transmission is fixed at one particular point, and if we assume that the cause of the morning fall is simply

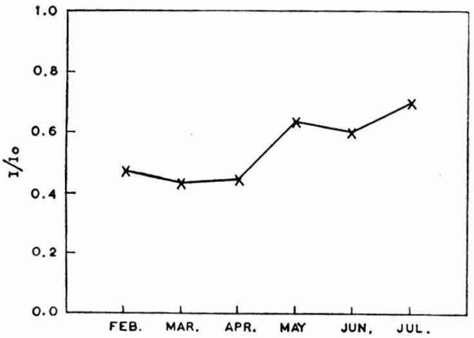

FIG. 4 — SEASONAL VARIATION AT 100 KC/S.: DAILY AVERAGE

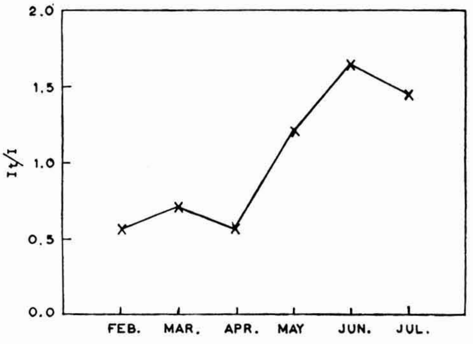

FIG. 5 — SEASONAL VARIATION AT 100 KC/S. AT 1700 HR.

the ionosphere, then the time difference Δt between the local sunrise time and the time of decrease of noise intensity should only vary with the height of the reflection level. Actual observations are far from being so simple. In fact, a difference as great as 2 hr. in the value of Δt has been observed on consecutive days. In order to find out the most probable value of Δt, a scatter diagram (Fig. 8) has been drawn for 100 kc/s., taking only those days into consideration where the sunrise effect was certain and there were no local storms during morning hours. The median of Δt has a value not much different from zero. The scatter indicates that the sources lie on both the east and west sides of Delhi. In order to compare the values of Δt with respect to sunrise at the ionospheric levels two additional scales are given, one for sunrise at 70 km. and the other for 100 km. above ground.

It may be mentioned here that whenever the night-time anomaly (discussed later) is present, the decrease at sunrise is very gradual.

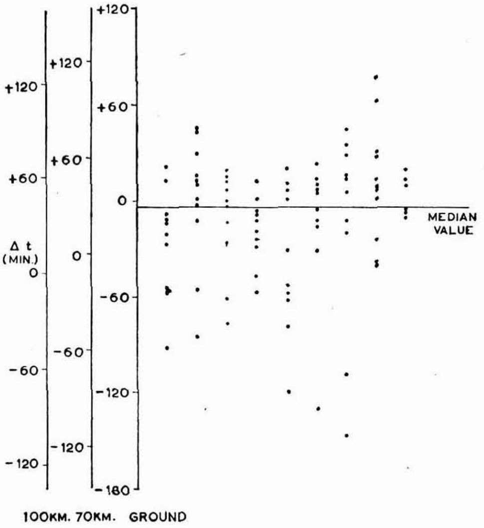

FIG. 8 — Δt AT 100 KC/S. FOR 0, 70 AND 100 KM. ABOVE GROUND LEVEL

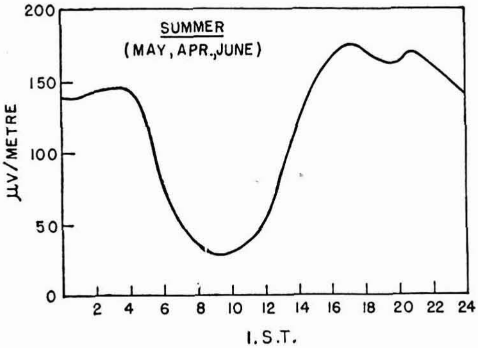

FIG. 6 — DIURNAL VARIATION AT 27 KC/S.

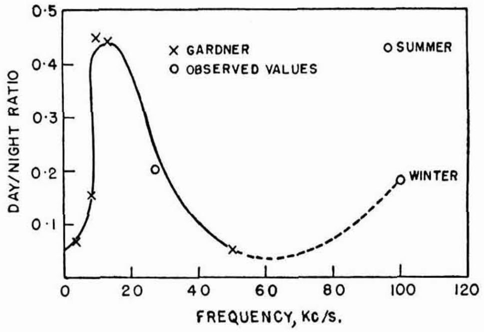

FIG. 7 — DAY/NIGHT RATIO AS A FUNCTION OF FREQUENCY

S.E.A.'s and solar flares — Considerable attention is being given to the study of S.E.A.'s and other effects associated with solar flares. The number of S.E.A.'s observed is not yet sufficient to make an analysis of their percentage association with the occurrence and size of solar flares. A complete list of the timings of the start, maximum and end of S.E.A.'s (and corresponding visual observations, whenever available) is given in the Appendix. This gives only the definite cases. Many uncertain cases have been left out and will be reported later if they are confirmed by solar data obtained from different observatories. Fig. 9(a) and (b) give the S.E.A. which was observed during the early hours of 21 June 1957. Fig. 10 shows the different shapes of S.E.A.'s observed so far for 27 and 100 kc/s. The records have been redrawn and the curves are smoothed reproductions of the original. Fig. 11 shows the S.E.A. following the big flare which occurred on 1 July 1957, the opening day of the International Geophysical Year. It is by far the biggest case observed by us since the beginning of these experiments.

Anomalies — Quite early in the beginning of the experiments it was observed that the night-time records at both 27 and 100 kc/s.

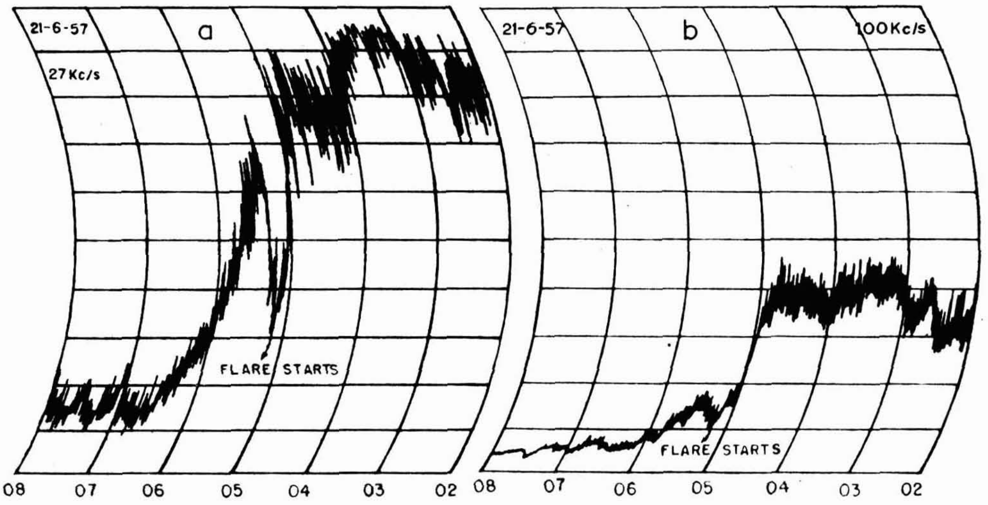

FIG. 9 — SIMULTANEOUS S.E.A. AT (a) 27 KC/S. AND (b) 100 KC/S. ON 21-6-57

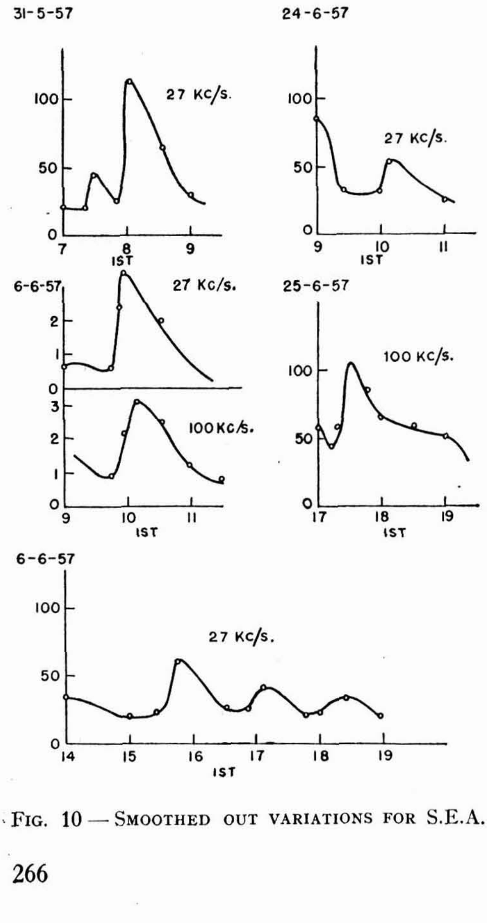

FIG. 10 — SMOOTHED OUT VARIATIONS FOR S.E.A.

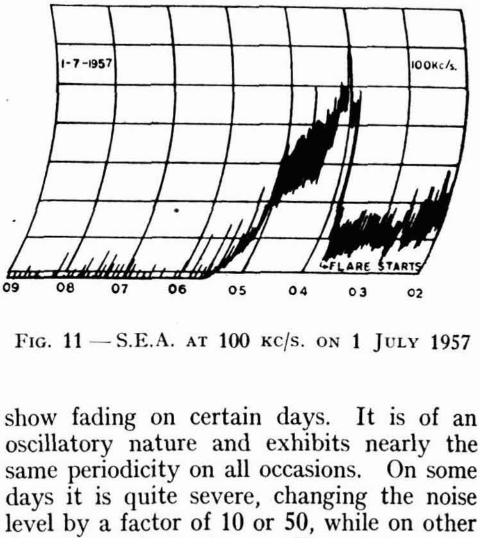

FIG. 11 — S.E.A. AT 100 KC/S. ON 1 JULY 1957

show fading on certain days. It is of an oscillatory nature and exhibits nearly the same periodicity on all occasions. On some days it is quite severe, changing the noise level by a factor of 10 or 50, while on other days it is entirely absent. Fig. 12 shows the records on three typical nights, provisionally denoted as quiet, mildly disturbed, and strongly disturbed. Fig. 11 also shows the same feature preceding the intense flare of 1 July. The morning fall is almost completely absent.

Discussion

The results reported here are preliminary and are much too limited in extent to allow any definite deductions. Nevertheless, some

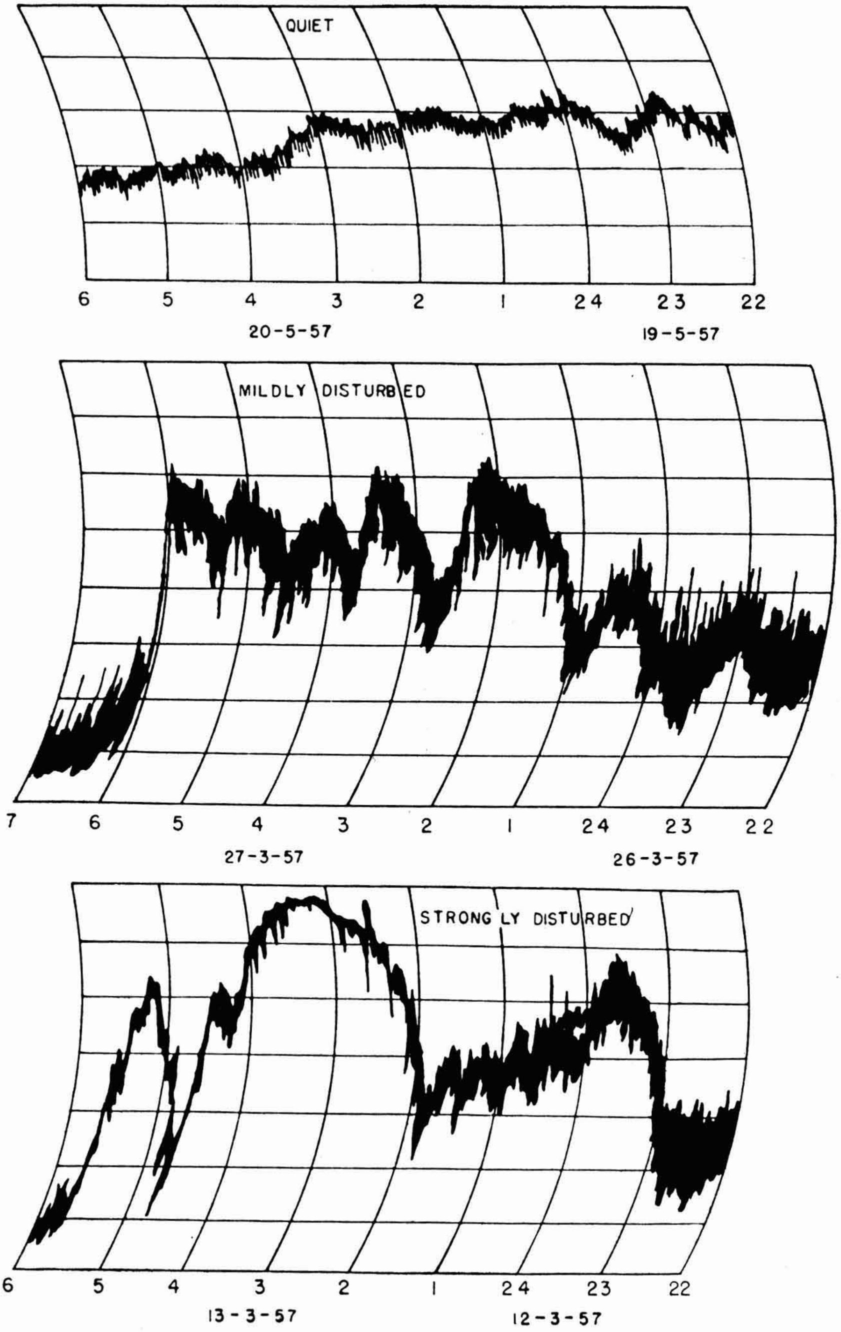

FIG. 12 — NIGHT-TIME ANOMALY AT 100 KC/S.

features appear to be established. These include the sunrise effect, the afternoon increase in noise level in summer, the SID effects and the severe oscillations observed on certain nights.

Sunrise effect — The sunrise decrease is well pronounced. It is, however, sharper at 100 kc/s. than at 27 kc/s. In winter the decrease is often followed by an increase in the early morning hours. In summer this is eclipsed by local thunderstorms. It seems likely that the time at which the effect starts depends on the longitude of the place over which reflection occurs. The large day to day variation in sunrise time would then imply a similar variation in the originating sources. It is possible to make a rough estimate of the location of the source, if one uses simultaneously a DF equipment, and assumes an appropriate height for the reflecting layer. As mentioned earlier, the scatter diagram relating the differences between the ground sunrise and the effect appears to indicate a random distribution of the thunderstorm sources around Delhi.

Sudden ionospheric disturbances — At both 100 and 27 kc/s., the SID effect is an increase in the noise level, that at 100 kc/s. being generally less pronounced. Bureau[4] was the first to notice this enhancement between 22·2 and 30 kc/s. with a maximum at 27 kc/s. The transition region (i.e. where the flares seemed to produce no effect) was between 17·6 and 22·2 kc/s. He did not observe any enhancement below 17·6 kc/s. He concluded that the waves are reflected at a level below that of maximum absorption. He further pointed out that under normal daylight conditions, these waves already have the advantage of a metal-like surface which is not affected by an increase in ionization gradient, the reverse of which occurs on the higher frequencies. He observed that S.E.A.'s did not always occur and that the association depended on the magnitude of the flare. Gardner[1] has, on the other hand, found a marked attenuation below 10 kc/s. for distances of the order of 1000 km. or more. However, he also found maximum enhancement above 20 and below 30 kc/s. Very different conclusions have, however, been advanced recently by Sullivan[8] and by King and Sullivan[6]. From observations of the average atmospheric noise at 50, 100, 189 and 500 kc/s. at Florida, Sullivan found that the most pronounced effect takes place at 189 kc/s. King and Sullivan reported maximum enhancement at 100 kc/s. It has been suggested that this difference is due to the difference in the geographical location of the receivers relative to the noise sources. Our own results agree with the concept of maximum enhancement around 27 kc/s. While conceivably the geographical position of the noise source will affect the frequency distribution of effect, difference as large as this from only a difference in distance does not seem likely. In King and Sullivan's work, the region around 27 kc/s. has been left out. Observations in this region may make a difference.

An important question in regard to the observed characteristics of the S.E.A. is: to what extent can the S.E.A. be used as a flare patrol technique?

The answer to this at present seems rather involved. It appears that while S.E.A. is a very sensitive tool for detecting flares at frequencies around 27 kc/s., it does not seem possible to infer from the size of an individual S.E.A. any very useful estimate of the intensity of the flare which caused it[5]. The results at Delhi, though limited in extent, appear to confirm this. During the period of observation altogether 45 definite cases have been observed for 100 kc/s.; of these 12 agree (some approximately) with the visual data supplied by different observatories. The corresponding figures for 27 kc/s. are 22 and 8. The data presented in the Appendix refer to only definite cases. Uncertain cases, many of which may be real S.E.A.'s, have been left out. The possible effect of distance from sub-solar point on the variation of S.E.A. occurrence and magnitude, has not been examined because of paucity of data.

When the experiments were originally undertaken, it was one of our objectives to locate the frequency at which transition occurs from enhancement to fadeout (Fig. 1, dotted portion). For this reason, 100 kc/s., which we felt will not be far from this point, was chosen. As we have mentioned before, observations indicate appreciable enhancement, although smaller than at 27 kc/s., still occurring at this frequency. However, on one occasion a fadeout was, in fact, observed at this frequency. We, therefore, feel that the transition cannot be very far from 100 kc/s.

Night-time anomaly — As mentioned above, the night-time records show severe fading on certain days. The cause of this is not yet known. Correlation with other phenomena has to be undertaken for this anomaly.

Conclusion

A preliminary survey of VLF atmospheric noise observations extending over a year points to the following conclusions:

(1) The average noise level below 100 kc/s. is appreciable during night hours but during daytime, fairly good communications can be maintained in this range especially when frequency congestion in the medium frequency range necessitates a search for an alternative band.

(2) The study of the sunrise effect shows that sources of thunderstorms are present more or less uniformly around Delhi; this should, however, be confirmed by D.F. equipment.

(3) The S.E.A. produced by solar flares is more prominent at 27 kc/s. than at 100 kc/s. especially during the hours immediately after sunrise. Only a few cases are there when no effect was observed at 27 kc/s. when 100 kc/s. shows enhancement. No case has yet been observed for which the maximum effect at 100 kc/s. occurs earlier.

Acknowledgement

This project was started at Dr. A. P. Mitra's suggestion. I am deeply grateful to him for his constant help and guidance throughout the survey.

References

1. Gardner, *Phil. Mag.*, **41** (1950), 1259.
2. Bowe, *Phil. Mag.*, **42** (1951), 121; **44** (1953), 833.
3. Pierce, *Symposium on Sferics* (W.M.O., Zürich), 1953.
4. Bureau, *Nature, Lond.*, **139** (1937), 110.
5. Ellison, *J. atmos. terr. Phys.*, **4** (1953), 236.
6. King & Sullivan, *Trans. Inst. Radio Engrs*, AP-**5** (1957), 78.
7. Sachdev, *RRC-S7, Sci. Rep., Radio Res. Comm., Nat. Phys. Lab., New Delhi, India*, 1947.
8. Sullivan, *Trans. Amer. geophys. Un.*, **33** (1952), 650.

APPENDIX — OBSERVED S.E.A.'S

Date	Visual data				S.E.A.			
	Start *U.T.*	Max. *U.T.*	End *U.T.*	Class	Frequency *kc/s.*	Start *U.T.*	Max. *U.T.*	End *U.T.*
1-2-57	—	—	—	—	100 100 100	1425 1500 1440	1438 1515 1500	1500 1617 1630
4-2-57	b1335	—	—	1	100	1340	1410	1600
13-2-57	0044	—	0110	2	100	0050	0100	0130
23-2-57	1512	—	a1530	1	100	1514	1545	1625
28-2-57	0005	—	0057	3	100	0015	0030	0220
22-3-57	—	—	—	—	100	2315	2345	0045
7-4-57	—	—	—	—	100	0810	0835	1030
10-4-57	—	—	—	—	100	0020	0027	0045
12-4-57	—	—	—	—	100	2330	0010	0130
14-4-57	0230	—	0245	1	100	0220	0228	0250
12-5-57	—	—	—	—	100	2250	2302	2315
13-5-57	—	—	—	—	100	0340	0410	0530
23-5-57	b1355	—	1408	1	100	1330	1337	1410
31-5-57	—	—	—	—	100	0930	0945	1013
31-5-57	—	—	—	—	100	1014	1018	1030
1-6-57	0354	0357	0405	2	100	0357	0405	0520
1-6-57	—	—	—	—	27	2137	2155	2330
2-6-57	—	—	—	—	100	0345	0355	0415
2-6-57	—	—	—	—	27	1245	1252	1307
3-6-57	0535	0537	0542	1	100	0535	0600	0623
3-6-57	—	—	—	—	27	1420	—	1535
6-6-57	—	—	—	—	27	0415	0425	0555
6-6-57	—	—	—	—	100	0415	0435	0505
6-6-57	b1053	—	1058	—	100	b1102	1122	1215
7-6-57	0326	0336	0354	1+	100	0330	0340	0405
9-6-57	—	—	—	—	27	0008	0022	0107
9-6-57	—	—	—	—	100	0010	0030	0115
20-6-57	—	—	—	—	27 100	2340 2340	2355 0000	0120 0023
24-6-57	—	—	—	—	100	0430	0448	0530
24-6-57	—	—	—	—	27	0431	0445	0526
25-6-57	—	—	—	—	100	0205	0223	0312
25-6-57	b0557	—	0603	2	100	0550	0605	0635
28-6-57	—	—	—	—	100	1137	1200	1340
30-6-57	—	—	—	—	100	2215	2230	0002
8-7-57	0929	—	1008	1+	27	0925	0940	1025
8-7-57	—	—	—	—	27	1330	1342	1419
11-7-57	—	—	—	—	100	1255	1310	1430
14-7-57	—	—	—	—	100	0540	0600	0630
14-7-57	—	—	—	—	100	0645	0705	0740

APPENDIX — OBSERVED S.E.A.'S — *contd.*

DATE	VISUAL DATA				S.E.A.			
	Start *U.T.*	Max. *U.T.*	End *U.T.*	Class	Frequency *kc/s,*	Start *U.T.*	Max. *U.T.*	End *U.T.*
14-7-57	—	—	—	—	27	2255	2307	2350
18-7-57	b2328	—	2412a	2	27	0000	0010	0130
20-7-57	0830	—	0845	—	27	0840	0852	0915
26-7-57	—	—	—	—	100	1137	1143	1230
28-7-57	—	—	—	—	27	0958	1007	1030
28-7-57	—	—	—	—	100	0954	1010	1030
28-7-57	1033	—	1105a	—	27	1033	1055	1145
29-7-57	—	—	—	—	27	1010	1030	1110
					100	1005	1030	1115
30-7-57	2317	—	2327	—	100	2315	2325	2355
2-8-57	—	—	—	—	27	0823	0830	0850
2-8-57	b0905	—	0915	1	27	0857	0905	0930
8-8-57	—	—	—	—	100	0504	0515	0532
9-8-57	—	—	—	—	27	0130	0145	0320
9-8-57	L0813	—	0819	—	27	0805	0814	0902
9-8-57	—	—	—	—	100	1255	1320	1545
9-8-57	—	—	—	—	100	2320	2340	0015
10-8-57	—	—	—	—	100	1000	1030	1110
14-8-57	—	—	—	—	27	1150	1200	1223
15-8-57	0914	—	0932	1	27	0915	0930	1018
16-8-57	2325	—	2330	1	27	2320	2325	2345
17-8-57	—	—	—	—	27	0415	0435	0510
20-8-57	—	—	—	—	100	0200	0215	0315
26-8-57	—	—	—	—	100	0005	0025	0105
18-9-57	—	—	—	—	100	0640	0645	0730
18-9-57	—	—	—	—	100	1237	1245	1255

Industrial Synthesis of Papaverine

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Methods for the synthesis of papaverine have been reviewed with special reference to their commercial possibilities.

Since the commercial synthesis of papaverine depends on the economic production of homoveratric acid and homoveratrylamine, an improved method for the synthesis of these compounds, starting from vanillin, is described.

The next important step in the synthesis of papaverine is the preparation of homoveratrylhomoveratramide, which is obtained in quantitative yield by heating homoveratric acid and homoveratrylamine in tetralin medium. The amide, on treatment with phosphorus oxychloride, cyclizes to 3 : 4-dihydropapaverine, which, on dehydrogenation in the presence of palladized charcoal, gives papaverine in quantitative yield (95 per cent of the theoretical).

PAPAVERINE[1,2] occurs in opium usually to the extent of 0·5-1·0 per cent and was first isolated from the mother liquors of the morphine extraction in 1848 by Merck[3]. The species of poppy plant cultivated at present in Gazipore yields opium containing *c.* 0·5 per cent papaverine.

To cope with the increasing demand of papaverine as a spasmolytic drug which has been included in the *British Pharmacopoeia*, the *United States Pharmacopoeia*, etc., processes for the industrial synthesis of the drug have been developed and are in use, although certain processes which have been successfully worked out in the laboratory, have not been exploited commercially[4]. In spite of renewed efforts to simplify the isolation and purification of papaverine from opium[5-8], the cost of recovery of natural papaverine at the Government Factory at Gazipore is high. The present communication describes the industrial synthesis of papaverine.

Pictet and Gams[9,10] carried out the first complete synthesis of the alkaloid in 1909.

In this synthesis veratrole was acetylated (Friedel-Crafts reaction) and the acetoveratrone was treated with nitrous acid and the corresponding *iso*nitroso derivative reduced with tin and hydrochloric acid to aminoacetoveratrone. The aminoketone, upon condensation with homoveratroyl chloride, gave homoveratramidoacetoveratrone which was reduced to the corresponding secondary alcohol. The latter yielded papaverine on treatment with phosphorus pentoxide.

Prior to this synthesis, Pictet and Finkelstein[11] had attempted to synthesize papaverine via dihydropapaverine but were unable to remove the two extra hydrogen atoms of the pyridine ring. This step was accomplished by Späth and Burger[12] who carried out dehydrogenation of 3, 4-dihydropapaverine in the presence of palladized asbestos at 200°. Homoveratrylamine and homoveratroyl chloride were condensed, and the amide converted to 3, 4-dihydropapaverine by Bischler-Napieralski reaction.

Kindler and Peschke[13] made certain improvements in this synthesis. They carried out dehydrogenation of dihydropapaverine in tetralin solution in the presence of palladium black. They developed two methods of preparation of homoveratric acid and formation of homoveratrylhomoveratramide by heating the amine and acid in tetralin solution. Milliken *et al.*[14] have made some improvements in the procedure of Kindler and Peschke. Purification of dihydropapaverine was effected by a single treatment of the original acid mixture with anhydrous caustic soda followed by washing with water. The intermediate was then dried by azeotropic distillation.

Another synthesis of papaverine which is claimed to be of commercial importance was performed by Wahl[15] in which 3, 4-dimethoxyphenylpyruvic acid prepared from veratraldehyde and hippuric acid, was treated with ammonia and the resultant diamide was hydrolysed to the corresponding β-(3, 4-dimethoxyphenyl)-α-(3, 4-dimethoxyphenylacetamido) propionic acid. The methyl ester of this compound, upon cyclization, saponification, decarboxylation and dehydrogenation, produced papaverine. The overall yield claimed in this synthesis is four parts of vanillin leading to one part of the alkaloid. An almost identical method has been described by Galat[16].

Wahl's synthesis suffers from the drawback that it depends on hippuric acid, a rather costly material to be used in a commercial synthesis.

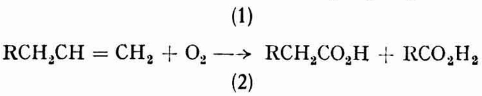
Based on the observation that the addition of a methoxy group in β-nitrostyrenes occurs when it is treated with sodium methoxide, Rosenmund[17] and also Mannich[18] prepared 1-methoxy-1-(3, 4-dimethoxyphenyl)-2-nitroethane, reduced the nitro group, and cyclized the homoveratroyl derivative with phosphorus oxychloride to papaverine. The yield of papaverine was rather poor in this process.

3, 4-Dihydropapaverine has also been obtained through the following steps[19-21]. Homoveratramidoacetoveratrone, when treated with phosphoryl chloride, gave 5-(3′, 4′-dimethoxyphenyl)-2-veratryl oxazole which was catalytically reduced to homoveratrylhomoveratramide. This on cyclization gave 3: 4-dihydropapaverine.

Späth and Burger[22] accomplished the synthesi of tetrahydropapaverine under mild conditions. Homoveratrylamine was condensed with homoveratraldehyde in ether solution at room temperature and the resulting product yielded 8 per cent of tetrahydropapaverine on treatment with hydrochloric acid apparently by hydration of the double bond and subsequent elimination of water. These last two syntheses are of academic interest only.

Comparing all these syntheses, it appears that the procedure of Kindler and Peschke may be extended into an industrial process if the two key materials, homoveratric acid and homoveratrylamine, can be prepared by cheap methods.

The general methods by which homoveratric acid may be obtained are indicated below:

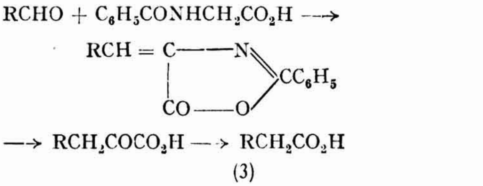


$$\mathrm{RH + HCl + HCHO \longrightarrow RCH_2Cl \longrightarrow RCH_2CN \longrightarrow RCH_2CO_2H}$$
$$\mathrm{RCH_2CN \longrightarrow RCH_2CH_2NH_2}$$

(1)

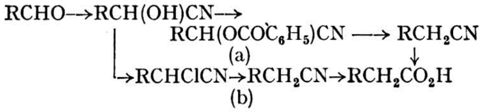
$$\mathrm{RCH_2CH = CH_2 + O_2 \longrightarrow RCH_2CO_2H + RCO_2H_2}$$

(2)



$$\mathrm{RCHO + C_6H_5CONHCH_2CO_2H \longrightarrow RCH{=}C\text{—}N{=}CC_6H_5\text{—}O\text{—}CO \longrightarrow RCH_2COCO_2H \longrightarrow RCH_2CO_2H}$$

(3)

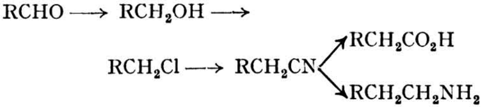


$$RCHO \rightarrow RCH(OH)CN \rightarrow RCH(OCOC_6H_5)CN \xrightarrow{(a)} RCH_2CN$$
$$\rightarrow RCHClCN \rightarrow RCH_2CN \rightarrow RCH_2CO_2H \quad (b)$$

(4)

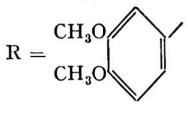
$$RH \longrightarrow RCOCH_3 \longrightarrow RCH_2CSNMe_2 \longrightarrow RCH_2CO_2H$$

(5)



$$RCHO \longrightarrow RCH_2OH \longrightarrow RCH_2Cl \longrightarrow RCH_2CN \rightarrow RCH_2CO_2H \text{ or } RCH_2CH_2NH_2$$

(6)



Gawron[23] is reported to have prepared veratryl chloride by chloromethylation of veratrole (1) in 54 per cent yield. The method is cumbersome and the yield is also poor. The yield of homoveratric acid by the azalactone method (3) is fair, but the method is rather expensive as it uses costly hippuric acid. Kindler developed the methods (4a) and (5) for the preparation of this acid. The former depends on hydrogenolysis and the latter uses dimethylamine in a Willgerodt reaction. These methods are too lengthy and costly to be of industrial interest.

Method (4b) appeared promising and was, therefore, tried for the preparation of homoveratronitrile. The bisulphite compound of veratraldehyde on treatment with potassium cyanide produced the cyanhydrin in fair yield. Chlorination of cyanhydrin with thionyl chloride in the presence of pyridine was attempted but the attempts to reduce the chloronitrile with zinc and acetic acid were unsuccessful.

Though method (6) has been reported to be unworkable by others[15], the method was investigated as it appears to be a cheaper method for the synthesis of homoveratric acid. The first step is rather easy. Veratryl alcohol is prepared from veratraldehyde (1) by crossed Cannizzaro reaction in *c.* 76 per cent yield and (2) by high pressure hydrogenation over Raney nickel catalyst at *c.* 100° in almost quantitative yield. It has been reported earlier[24] that veratraldehyde on hydrogenation with platinum activated by iron yields considerable amount of diveratryl ether besides veratryl alcohol. No such side reaction occurs during high pressure hydrogenation over Raney nickel catalyst. The second step involves certain difficulties. Veratryl chloride is rather unstable and treatment of the veratryl alcohol with thionyl chloride in the presence of pyridine leads to the formation of an anthracene derivative probably by intermolecular condensation as reported by Carré and Liebermann[25]. Wahl[15] also made an unsuccessful attempt to prepare the chloride by passing hydrochloric acid gas through a cooled solution of the alcohol in toluene. In this connection it is of interest to note that during the single-phase chloromethylation of veratrole[23], similar anthracene derivative was formed. Veratryl alcohol, on treatment with thionyl chloride in the presence of dimethylaniline in chloroform medium, was found to yield the chloride in satisfactory yield. Treatment of the chloride with potassium cyanide in alcohol or acetone medium in the presence of small amounts of potassium iodide as activating agent gave an unsatisfactory yield of the desired homoveratronitrile. However, the nitrile is obtained in excellent yield by carrying out the above reaction in two phases. The chloride obtained by the above method was directly converted into the nitrile without any further purification. The chloride was dissolved in benzene and heated under reflux and stirred with an aqueous solution of potassium cyanide. The addition of alkali iodides as activating agents is not necessary as the chloride itself is sufficiently reactive. Homoveratronitrile is next converted into homoveratric acid by alcoholic potassium hydroxide according to the method of Kindler and Peschke[13]. The overall yield of homoveratric acid by this method is *c.* 65 per cent of the theoretical amount (calculation based on veratraldehyde). Homoveratronitrile was hydrogenated over Raney nickel catalyst at *c.* 500 lb./sq. in. pressure and 100° in the presence of ammonia to homoveratrylamine following the method of Icke and Redemann[26].

Other methods of preparation of homoveratrylamine which are likely to be of industrial importance are indicated below. Method (7) has been utilized by Kindler[27] for the preparation of homoveratrylamine.

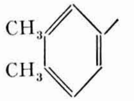
$$RCHO \longrightarrow RCH = CHCO_2CH_3 \longrightarrow RCH_2CH_2CO_2CH_3 \longrightarrow RCH_2CH_2CONH_2 \longrightarrow RCH_2CH_2NH_2$$

(7)

$$RCHO + CH_3NO_2 \longrightarrow RCH = CHNO_2 \longrightarrow RCH_2CH_2NH_2 \qquad (8)$$

$$RCHO \longrightarrow RCH(OH)CN \longrightarrow RCH_2CH_2NH_2 \qquad (9)$$

R =



Wahl[15] is reported to have made some improvements in the last step of the process. Although the yield is fair, the process is rather long and involves operations which are difficult to realize in industrial practice. Method (8) utilizes nitromethane which is a costly material.

Thus the successful solution of the problem of cheap production of homoveratric acid and homoveratrylamine paves the way to the commercial synthesis of papaverine. The homoveratrylhomoveratramide has been prepared by the method of Kindler and Peschke[13] in almost quantitative yield by heating homoveratric acid and homoveratrylamine in tetralin medium and distilling the water formed in the reaction. The amide on treatment with phosphorus oxychloride cyclized to dihydropapaverine which on dehydrogenation over 10 per cent palladized charcoal in boiling tetralin medium furnished papaverine. The overall yield of papaverine by this method is about four parts of the alkaloid for seven parts of vanillin.

Dehydrogenation of ethyl dihydropapaverine-3-carboxylate by means of sulphur at a temperature not exceeding 180° has also been described in a patent[28]. The study of dehydrogenation of dihydropapaverine with sulphur is in progress in this laboratory.

Experimental procedure*

3, 4-*Dimethoxybenzyl alcohol* (*veratryl alcohol*) — This has been prepared by two methods:

(a) The procedure[29] for the preparation of *p*-tolyl carbinol from *p*-tolualdehyde was followed.

The apparatus consists of a 1 litre three-necked flask fitted with a mercury-sealed mechanical stirrer, a reflux condenser, a dropping funnel, and a thermometer which reaches almost to the bottom of the flask. Potassium hydroxide pellets (85 per cent; 100 g.) and 150 ml. of absolute methanol (free from acetone) were placed in the flask and stirred. The bulk of the alkali dissolved in a few minutes with the evolution of heat. The flask was then transferred to a cold water bath, and when the temperature of contents dropped to 60°, a mixture of veratraldehyde[30] (99·6 g.), formalin (40 per cent; 60 ml.) and absolute methanol (60 ml.) was added at such a rate that the temperature of the contents remained at 60°-70°. The time taken for this was *c.* 10 min. The temperature of the contents was maintained at 60°-70° for 3 hr., after which the reflux condenser was replaced by a downward condenser and the methyl alcohol distilled with the aid of a brine bath until the temperature of the contents reached 101°. Cold water (180 ml.) was then added to the warm residue, and the mixture cooled. The resulting two layers were separated and the aqueous layer extracted with three 40 ml. portions of benzene. The combined oil and benzene extracts were washed with five or six 20 ml. portions of water, and the combined washings extracted with 25 ml. of benzene, the benzene layer being added to the washed extract. The benzene solution was dried (anhydrous sodium sulphate) and distilled under diminished pressure. After the removal of benzene, 76·4 g. (76 per cent of the theoretical amount) of 3, 4-dimethoxybenzyl alcohol (b.p. 150°-52°/5 mm.) was obtained.

(b) A solution of veratraldehyde (purified by distillation; 93 g.) in ethanol (186 ml.) was placed in a high-pressure hydrogenation bomb with 1·2 litre void, 10 ml. of settled Raney nickel catalyst[31] (*c.* 6 g.) added, the bomb closed and hydrogen introduced until the pressure was *c.* 1450 lb./sq. in. The bomb was shaken and heated to *c.*100° until the absorption of hydrogen ceased (*c.* 3-4 hr.). The bomb was cooled and opened and the contents removed. The bomb was rinsed with ethanol (100 ml. × 3) and the combined liquids poured through a fluted filter to remove the catalyst. The solvent was removed by distillation and the residue distilled under vacuum; yield quantitative, b.p. 150°-52°/5 mm. (Table 1).

3, 4-*Dimethoxyphenylacetonitrile* (*homoveratronitrile*) — 3, 4-Dimethoxybenzyl alcohol (98 g.) and dimethylaniline (105 g.) were dissolved in 128 ml. of dry chloroform in a

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*All melting points of substances are corrected; boiling points are uncorrected.

TABLE 1—HYDROGENATION OF VERATRALDEHYDE

TIME *hr.*	TEMP. °C.	PRESURE *lb./sq. in.*
0	15	1450 (initial cold pressure)
1	64	1600
1½	100	1800
2	100	1700
2½	100	1650
3	100	1650
4	100	1650
—	18	1250 (final cold pressure)

1 litre three-necked flask fitted with a mercury-sealed mechanical stirrer and a dropping funnel. The flask was cooled in ice and a solution of thionyl chloride (105 g.) in 128 ml. of dry chloroform added dropwise in *c.* 2 hr. Stirring was continued for a further period of 15 min. and the flask heated on steam bath for *c.* 1 hr. till the evolution of hydrogen chloride ceased. After cooling, the red solution was poured in 1400 ml. of cold 1N hydrochloric acid solution and the aqueous phase thoroughly extracted with chloroform. The combined chloroform extract was washed with 1N hydrochloric acid, water, 2N sodium bicarbonate solution and water, dried over calcium chloride and chloroform removed by heating on the water bath under vacuum.

The crude chloride obtained above was dissolved in 140 ml. of benzene and heated under reflux with stirring for *c.* 4 hr. after adding a solution of 93 g. of potassium cyanide in 350 ml. of water. The organic phase was separated, thoroughly washed with water, dried and evaporated. The residue was distilled; yield 69-70 g. (67·8 per cent of theory); b.p. 162° (5 mm.).

Thionyl chloride (E. Merck) was distilled and the fraction distilling at 76°-78° was used in this preparation. Dimethylaniline E. Merck) was dried over sodium hydroxide pellets and then distilled. The fraction distilling at 194°-95° was used.

Dimethylaniline can be recovered in almost quantitative yield from the acidic wash liquor. The combined aqueous extract was made alkaline with sodium hydroxide to liberate dimethylaniline and extracted with benzene. The benzene extract was washed with water and distilled. The fraction distilling at 193°-96° was collected.

3, 4-*Dimethoxyphenylacetic acid* (*homoveratric acid*)[13] — Homoveratric acid was prepared from homoveratronitrile by hydrolysis according to the method of Kindler and Peschke[13].

A solution of 3, 4-dimethoxyphenylacetonitrile (177 g.) in ethanol (550 ml.) was treated with aqueous solution of potassium hydroxide (50 per cent; 300 g.) and the mixture heated on the water bath under reflux for *c.* 4 hr. When the hydrolysis was complete (judged by placing a drop of the reaction mixture in water; the liquid remains clear when the reaction is complete), alcohol was removed and the residue was cooled and dissolved in *c.* 630 ml. of ice-cold water. The mixture was extracted twice with ether and the ether extract washed with water each time. The combined aqueous solution was poured drop by drop with stirring into a beaker containing a mixture of 540 ml. of concentrated hydrochloric acid and 1260 g. of cracked ice. Homoveratric acid hydrate crystallized at once; the whole mass was kept in ice for 30 min., filtered and washed with ice-cold water to remove sodium chloride. It was finally dried in a vacuum desiccator; m.p. 97°. The yield is almost quantitative. The acid could be further purified by recrystallization from a mixture of benzene and petroleum ether (b.p. range 60°-80°) with slight loss; the recrystallized product melts at 98°

3, 4-*Dimethoxyphenylethylamine* (*homoveratrylamine*) — Homoveratrylamine has been prepared from homoveratronitrile in excellent yield following the procedure of Icke and Redemann[26]. Commercial anhydrous methanol was saturated with ammonia gas at 0°; this solution was approximately 10N. A solution of 36 g. (0·2 mole) of homoveratronitrile in 120 ml. of 10N methanolic ammonia (the ratio of ammonia to homoveratronitrile should be at least 5: 1 in order to minimize the formation of secondary amine) was placed in a high pressure hydrogenation bomb of 1·2 litre void, 1-2 ml. of settled Raney nickel catalyst[31] added, the bomb closed and hydrogen introduced until the pressure was 500-1000 lb./sq. in. The bomb was shaken and heated to 100°-25° until absorption of hydrogen ceased (*c.* 2 hr.). The bomb was cooled, opened and the contents removed. The bomb was rinsed with methanol (50 ml. × 3) and the combined liquids were filtered through a fluted filter to remove the catalyst. The solvent and the ammonia were removed by distillation and the residue fractionated through a short column. The yield

TABLE 2 — HYDROGENATION OF HOMOVERATRONITRILE

TIME *min.*	TEMP. °*C.*	PRESSURE *lb./sq. in.*
0	20	825 (initial cold pressure)
40	70	925
50	90	800
53	100	700
68	100	700
—	15	425 (final cold pressure)

of homoveratrylamine, b.p. 119°-119·5°/1 mm., was 29 g. (*c.* 80 per cent of the theoretical) (Table 2).

Homoveratrylhomoveratramide—Homoveratric acid (52·7 g.) and homoveratrylamine (48·7 g.) were dissolved in 260 ml. of freshly distilled tetralin (b.p. 203°-4°) in an atmosphere of nitrogen. After this, *c.* 230 ml. of tetralin were distilled within about an hour; a slow current of nitrogen was passed through the solution during the distillation of tetralin. Water formed in the reaction was also carried over. When the separation of water was complete, the rest of tetralin was distilled under reduced pressure. The residue was crystallized from toluene and the crystals filtered under suction and washed with ether. The amide melted at 124°. The yield was *c.* 96 per cent of the theoretical.

3 : 4-*Dihydropapaverine*[13]— Homoveratrylhomoveratramide (100 g.) was dissolved in 400 ml. of thiophene-free dry benzene and the solution treated with 60 ml. of phosphorus oxychloride. A slow current of carbon dioxide was passed through the solution during the reaction. The mixture was heated under reflux on steam bath for *c.* 30 min. Benzene was distilled off under reduced pressure. The residue was dissolved in 200 ml. of water and the solution washed once with benzene to remove non-basic impurities. Dihydropapaverine was then separated by adding potassium hydroxide solution and collecting in benzene. The benzene solution was dried over potassium hydroxide pellets, decolourized (charcoal) and evaporated under reduced pressure. Crystalline dihydropapaverine separated; m.p. 105°. The yield was almost quantitative.

Papaverine[16] — 3, 4-Dihydropapaverine (10 g.), palladium-charcoal[32] (10 per cent Pd; 1 g.) and 15 ml. of tetralin were heated in an oil bath at 240°-50° in a current of nitrogen for 3 hr. The catalyst was removed by filtration, the filtrate cooled and seeded with papaverine. After *c.* 1 hr., the crystals of papaverine formed were filtered, washed with a small amount of cold tetralin and dried *in vacuo*; yield 9·0 g., m.p. 140°. The filtrate was used once to treat another 10 g. of dihydropapaverine and yielded 9·6 g. of crude papaverine (m.p. 140°). The yield was *c.* 95 per cent of the theoretical. The crude papaverine was recrystallized from *iso*-propyl alcohol and a product melting at 146°-47° (lit. 145°, 147°)[13,33] was obtained. The hydrochloride melted at 231° (lit. 231°)[13].

Acknowledgement

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References

1. DRAGENDORFF, G., *Die Heilpflangen* (Enke, Stuttgart), 1898, 249.
2. MACHIGUCHI, E., *Chem. Abstr.*, **20** (1926), 2725.
3. MERCK, G., *Liebig's Ann.*, **66** (1848), 125; **73** (1850), 50.
4. ALFRED BURGER, *The Alkaloids*, Vol. IV (Academic Press Inc., New York), 1954, 42.
5. LEVI, J. R. & CASTELLI, F., *Chem. Abstr.*, **34** (1940), 7532.
6. WILLSTAEDT, H., *Chem. Abstr.*, **40** (1946), 990.
7. ALYAVDINA, L. A., *Chem. Abstr.*, **30** (1936), 3591.
8. BUSSE, S. & BUSSE, V., *Chem. Abstr.*, **28** (1934), 478.
9. PICTET, A. & GAMS, A., *Ber. dtsch. chem. Ges.*, **44** (1911), 2480.
10. PICTET, A. & GAMS, A., *C.R. Acad. Sci., Paris*, **149** (1909), 210.
11. PICTET, A. & FINKELSTEIN, M., *Ber. dtsch. chem. Ges.*, **42** (1909), 1779.
12. SPÄTH, E. & BURGER, A., *Ber. dtsch. chem. Ges.*, **60** (1927), 704.
13. KINDLER, K. & PESCHKE, W., *Arch. Pharm.*, **272** (1934), 236; **271** (1933), 434.
14. MILLIKEN, W. *et al.*, *Div. of Medicinal Chem., Abstracts*, 111*th Meeting A.C.S., Atlanta City, N.J.*, 14 April 1947.
15. WAHL, H., *Bull. Soc. chim. Fr.*, **18** (1951), D1.
16. GALAT, A., *J. Amer. chem. Soc.*, **72** (1950), 4436; **73** (1951), 3654.
17. ROSENMUND, K. W. *et al.*, *Ber. dtsch. chem. Ges.*, **60** (1927), 392.
18. MANNICH, C., *Arch. Pharm.*, **265** (1927), 1.
19. BUCK, J. S., *J. Amer. chem. Soc.*, **52** (1930), 3610.
20. BUCK, J. S., *J. chem. Soc.*, (1933), 740.
21. YOUNG, P. C. & ROBINSON, R., *J. chem. Soc.*, (1933), 275.
22. SPÄTH, E. & BURGER, F., *Ber. dtsch. chem. Ges.*, **63** (1930), 2098.

23. OSCAR GAWRON, *J. Amer. chem. Soc.*, **71** (1949), 744.
24. BENGT O. LINDGREN, *Chem. Abstr.*, **45** (1951), 6602.
25. CARRÉ & LIEBERMANN, *Bull. Soc. chim. Fr.*, **2** (1935), 292.
26. ICKE, R. N. & REDEMANN, C. E., *Organic Syntheses*, Coll. Vol. 3 (John Wiley & Sons Inc., New York), 1955, 721.
27. KINDLER, K., *Arch. Pharm.*, **269** (1931), 77.
28. BOEDECKER, F. & HEYMANS, A., *Germ. Pat.* 674,400 (1937).
29. DAVID DAVIDSON & MARVIN WEISS, *Organic Syntheses*, Coll. Vol. 2 (John Wiley & Sons, Inc., New York), 1950, 590.
30. BARGER & SILBERSCHMIDT, *J. chem. Soc.*, **133** (1928), 2924.
31. RALPH MOZINGO, *Organic Syntheses*, Coll. Vol. 3 (John Wiley & Sons Inc., New York), 1955, 181.
32. RALPH MOZINGO, *Organic Syntheses*, Coll. Vol. 3 (John Wiley & Sons Inc., New York), 1955, 687.
33. WAHL, H., *Bull. Soc. chim. Fr.*, **17** (1950), 680.

High Pressure Ammonolysis of Ethylene Dichloride

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The influence of various factors such as ratio of reactants, concentration of ammonia, and duration and temperature of reaction on the efficiency of conversion of ethylene dichloride to ethylene diamine by heating it in the presence of ammonia in a closed tube has been studied. Maximum yield (*c.* 90 per cent) is obtained by carrying out ammonolysis under the following conditions: ammonia-ethylene dichloride ratio, 19 : 1; temp., 110°C.; reaction period, 1 hr.; the yield is independent of the concentration of ammonia so long as the total amount remains constant. It is advantageous to use liquor ammonia than anhydrous ammonia as the former gives better yields and the reaction can be carried out under simpler operating conditions.

Though the reaction is exothermic, the yield increases with rise in reaction temperature which indicates that the rate of reaction is very slow at low temperatures.

CURME[1], Lauter[2] and Bersworth[3] have patented methods for the production of ethylene diamine by the ammonolysis of ethylene dichloride using liquor ammonia or anhydrous ammonia as the aminating agent at elevated pressures and at temperatures of 100°-180°C. Some workers[4] have used catalysts like cuprous chloride, zinc chloride and zinc oxide to speed up the reaction. However, the optimum conditions for obtaining maximum yield of the amine are not available in literature.

A systematic study of the ammonolysis of ethylene dichloride has been carried out in order to determine the optimum conditions for obtaining maximum yield of the amine and the results are presented in this paper.

Experimental procedure

The apparatus used in the present investigation was similar to the one used by Bhrany and Rao[5]. The reactor had a volume of 290 ml. and the stainless steel tube a volume of 180 ml.

Known quantities of ethylene dichloride (B.D.H. reagent, distilled at 80°C./683 mm.) and liquor ammonia (12N) were mixed in the stainless steel tube and placed inside the reactor fitted with a calibrated gauge. The reactor was then closed and heated electrically to the desired temperature and held at it for definite periods. After the reaction was complete, the reactor was allowed to cool and the products discharged into two spiral condensers cooled in freezing mixture. The uncondensed gases were collected in a carboy over brine and analysed.

The products from the condensers and the stainless steel tube were diluted with water to a known volume from which an aliquot was refluxed with excess of sodium hydroxide for 2-3 hr. to expel the free ammonia as well as the ammonia from ammonium chloride. The resulting solution was distilled and the amine content determined by titration with decinormal acid using methyl orange as indicator.

It has been pointed out by Groggins and Stirton[6] that in addition to ethylene diamine higher amines of the type diethylene triamine and triethylene tetramine are also produced during ammonolysis of ethylene dichloride. Some experiments carried out in this laboratory using higher ammonia-ethylene dichloride ratios indicated ethylene diamine to be the main product.

Results

Effect of varying ammonia ratio — For investigating the effect of varying ammonia-ethylene dichloride ratio, two procedures were adopted: In the first (experiments 1-7, Table 1), the quantity of liquor ammonia was varied keeping the amount of ethylene dichloride constant. In the second case (experiments 8-10, Table 1) the quantity of liquor ammonia was kept constant and the amount of ethylene dichloride varied. The experimental conditions and the results obtained are given in Table 1.

It is clear from Table 1 that the yield of the amine increases with increase in ammonia-ethylene dichloride ratio and a maximum yield of *c.* 90 per cent of the diamine is obtained with an ammonia-ethylene dichloride ratio of 19.

Effect of varying ammonia concentration — Although an ammonia-ethylene dichloride ratio of 19 was found to be optimum for the reaction, the effect of varying ammonia concentration was investigated at an ammonia-ethylene dichloride ratio of 7·4. A fixed quantity (40 ml.) of liquor ammonia (12N) was diluted with water to various concentrations and used for the experiments. The results obtained are given in Table 2.

The results given in Table 2 indicate that the yield of the amine obtained is independent of the concentration of ammonia as long as the total amount of ammonia in the reaction mixture is constant.

Effect of varying reaction period — Experiments were carried out to determine the effect of varying the reaction period on the yield of the diamine. The results showed that a maximum yield of 90 per cent of the diamine is obtained by heating the reactants for 20 min. at 110°C.

TABLE 1 — EFFECT OF VARYING AMMONIA-ETHYLENE DICHLORIDE RATIO

(*Temp.*, 120°*C.*; *reaction period*, 5 *hr.*; *pressure*, 180-200 *lb./sq. in.*; *strength of liquor ammonia*, 12*N*)

EXPT. No.	VOL. OF REACTANTS *ml.*		$\frac{NH_3}{(CH_2Cl)_2}$ (molar ratio)	YIELD OF ETHYLENE DIAMINE %
	Liquor ammonia	Ethylene dichloride		
1	10	5	1·85	28·30
2	20	5	3·69	50·79
3	40	5	7·38	61·81
4	65	5	12·00	74·01
5	80	5	14·76	77·84
6	100	5	18·45	89·17
7	120	5	22·14	91·58
8	100	10	9·23	72·28
9	100	15	6·15	59·82
10	100	20	4·61	36·97

TABLE 2 — EFFECT OF VARYING AMMONIA CONCENTRATION

[*Reactants*: 5 *ml. of* $(CH_2Cl)_2$+40 *ml. of liquor ammonia* (12*N*) *diluted to different volumes*; *temp.*, 110°*C.*; *reaction period*, 5 *hr.*; $\frac{NH_3}{(CH_2Cl)_2}$ (*molar*), 7·4]

EXPT. No.	LIQUOR AMMONIA		PRESSURE *lb./sq. in.*	YILED OF DIAMINE %
	Concn. *N*	Vol. *ml.*		
1	12·0	40	180	62·05
2	8·0	60	150	59·60
3	6·0	80	100	61·38
4	4·8	100	70	62·19
5	4·0	120	60	62·88

TABLE 3 — EFFECT OF VARYING TEMPERATURE

[*Reactants*: 5 *ml. of* $(CH_2Cl)_2$+100 *ml. of liquor ammonia* (12*N*); *reaction period*, 1 *hr.*]

EXPT. No.	TEMP. °C.	PRESSURE *lb./sq. in.*	YIELD OF DIAMINE %
1	27	15	39·66*
2	60	90	57·69
3	110	180	89·17
4	130	210	90·03
5	150	360	90·03
6	200	700	81·02†

*Time required, 19 days.
†Gaseous products were obtained.

Effect of varying temperature — The results given in Table 3 show that a reaction period of 1 hr. is adequate for satisfactory progress of the reaction, particularly for temperatures of 110°-50°C. and above; the best yield (89·17 per cent) is obtained at 110°C.

Use of anhydrous ammonia as aminating agent — Experiments were also carried out employing anhydrous ammonia as the aminating agent. To transfer a known quantity of ammonia, an intermediate high pressure condenser was employed[7]. The results are given in Table 4.

TABLE 4 — EFFECT OF USING ANHYDROUS AMMONIA AS AMINATING AGENT

[*Reactants*: $(CH_2Cl)_2$, 5 *ml.*+*anhydrous ammonia*, 20 *g.*; *reaction period*, 3 *hr.*]

Expt. No.	Temp. °C.	Pressure *lb./sq. in.*	Yield of diamine %
1	40	285	34·07
2	40	290	36·51
3	60	450	40·57
4	90	650	34·89*
5	110	700	17·84*

*Resinous products obtained.

TABLE 5 — THERMODYNAMIC DATA

Compound	State	Heat of formation at 250°C. *cal./mole*	Free energy of formation at 25°C. *cal./mole*
Ethylene dichloride	(L)	−39700	−19200
Ammonia	(g)	−11040	−3976
	(Aq)	−19000	−5789
Ethylene diamine	(L)	−6360	+1300
	(Aq)	−13860	−1286
Ammonium chloride	(c)	−75380	−48730
	(Aq)	−71600	−50000

TABLE 6 — EQUILIBRIUM CONSTANTS

Reaction carried out with	Heat of reaction at 25°C. *cal.*	Free energy change at 25°C. *cal.*	Equilibrium constant at	
			25°C.	110°C.
Anhydrous ammonia	−73260	−61056	$5 \cdot 9 \times 10^{44}$	$7 \cdot 1 \times 10^{32}$
Liquor ammonia	−41360	−58930	$1 \cdot 3 \times 10^{43}$	$2 \cdot 4 \times 10^{36}$

It is seen from Table 4 that maximum yield of the diamine is obtained at 60°C.

A comparison of the results given in Tables 3 and 4 shows that it is more advantageous to use liquor ammonia than anhydrous ammonia since the yield of the amine is greater with liquor ammonia and the reaction is carried out under simpler operating conditions.

Discussion

An increase in the temperature of ammonolysis in the liquid phase increases the pressure of the system, which in turn increases the activity of ammonia in the solution. It can, therefore, be expected that an increase in reaction temperature should result in higher yields of the amine. The results obtained in the present study support this view. It is seen from Table 3 that the yield has increased from 40 to 90 per cent when the temperature is increased from 27° to 110°C. The diminished yield after 150°C. is due to the side reactions taking place in the system giving rise to gaseous products.

The thermodynamic data[8] for the reaction $ClCH_2CH_2Cl + 4NH_3 \longrightarrow NH_2CH_2CH_2NH_2 + 2NH_4Cl$ are given in Table 5.

The free energy of formation of ethylene diamine was calculated by the structural modification method (taking a value of −10,700 cal./mole for the free energy of formation of ethane) as given by Parks and Huffman[9]. The free energies of formation of aqueous solutions of ammonia, ethylene diamine and ammonium chloride were estimated from their partial vapour pressures in aqueous solution.

From the data given in Table 5, the heat of reaction and the free energy change were calculated both for gaseous reaction and for the reaction in aqueous solution. The equilibrium constants at 25° were then calculated by using the equation $\Delta F = -RTlnk$. Assuming the heat of reaction to be constant with increase in temperature, equilibrium constants at 110°C. were calculated by Vant Hoff equation and the results are given in Table 6.

It can be inferred from the values given in Table 6 that the reaction is highly exothermic and the yields should, therefore, diminish with increase in temperature both in the gaseous phase as well as in solution phase. But the results presented in Table 3 show that with increase in temperature the yield of the amine increases. It is probable that the rate of the reaction, especially at lower temperatures, is slow and the products obtained by the experiment do not represent the equilibrium values.

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References

1. Curme, G. O. & Lowmmen, F. W., *Chem. Abstr.*, **26** (1932), 999.
2. Lauter, W. M., *Chem. Abstr.*, **30** (1936), 486.
3. Bersworth, F. C., *Chem. Abstr.*, **30** (1936), 1395.
4. Goodyear Tyre & Rubber Co., *Fr. Pat.* 739,317 (1932).

5. Bhrany, U. N. & Rao, M. R. A., *J. Indian Inst. Sci.*, **37** (1955), 242.
6. Groggins, P. H. & Stirton, A. J., *Industr. Engng Chem.*, **29** (1937), 1355.
7. Krishna Murthy, V. A. & Rao, M. R. A., *J. Indian Inst. Sci.*, **39** (1957), 138.
8. Rossini, F. D. *et al.*, *Selected Values of Chemical Thermodynamics, Circular of the National Bureau of Standards, No.* 500, 1952.
9. Parks & Huffman, *The Free Energies of Some Organic Compounds* (Chem. Catalog Co., New York), 1932, 209.

Coating Compositions from Modified Kamala Seed Oil

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The suitability of kamala seed oil, modified by alcoholysis with monohydric alcohols, for the preparation of coating compositions has been investigated. The modified oil gave varnishes which were slightly inferior in hardness, drying and other characteristics to those prepared from tung oil.

THE oil obtained from kamala seeds by solvent extraction gels rapidly and is, therefore, not suitable for the preparation of varnishes and other coating compositions[1]. However, on alcoholysis with monohydric alcohols like butyl or amyl alcohol, in the presence of minute quantities of alcoholic hydrochloric acid, the gelation properties of the oil are reported to be similar to those of tung oil[2]. The present paper reports the characteristics of oleoresinous varnishes prepared from modified kamala seed oil using different resins.

Materials and methods

A mixture of benzene-extracted kamala seed oil (1 kg.), moisture-free butyl or amyl alcohol (1·5 litres) and 10N alcoholic hydrochloric acid (10 ml.) was heated on boiling water bath for 90 min. and then alcohol distilled off under reduced pressure. The butylated and amylated oils obtained had Brown heat test values of 9·5 and 9·3 respectively and colour intensities on Lovibond scale (through 1 cm. cell) 9R-27·6Y and 8·6R-27·8Y respectively.

The following resins were used for preparing varnishes:

Phenolic resins — (i) Bedesol 99 (I.C.I.); and (ii) Bedesol 66 (I.C.I.).

Maleic resins — (i) EPOK M4000 (British Resin Products; m.p. 113°-17°C., acid val. 9-12); and (ii) 8 per cent maleic glycerol esterified resin prepared in the laboratory by condensing rosin with 8 per cent of its weight of maleic anhydride at 160°-80°C. and esterifying the product.

Miscellaneous resins — (i) Congo Copal ester (R. H. Cole & Co.); (ii) Coumarone Indene Resin (R. H. Cole & Co.); (iii) penta ester gum; (iv) ester gum; (v) rosin; and (vi) CNSL modified rosin.

The formulations tried (Tables 1 and 2) and the methods employed for cooking the varnishes were the same as reported by some earlier workers[3-5]. After allowing to age for one week, the films of the filtered varnishes were applied on glass and iron plates by sheen applicator of 0·002 in. film thickness and allowed to dry. The times taken by the films for surface and hard drying were noted. The films, after drying for 2 days, were tested for different characteristics according to the method recommended in the Indian Standard Specification No. 101, 1950. The dissolving action of different solvents on the films for varying periods of contact was also determined[6,7].

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Results and discussion

All the varnish compositions prepared from either tung oil or modified kamala seed oil were found to keep well on storage for 6 months in corked glass bottles.

Most of the formulations based on modified kamala seed oil were found to be inferior to those based on tung oil. The characteristics of those formulations whose films gave performances comparable to those of films obtained using tung oil are given in Table 3.

Varnishes based on modified kamala seed oil were, in general, darker than those based on tung oil. In the case of formulation 2, the varnishes prepared by using tung oil and butylated kamala seed oil had colour intensities 5·4R-27Y and 6·0R-68Y respectively. On replacing the phenolic resin (Bedesol 99) in this formulation by maleic resin (EPOK M4000), the same trend was evident, the colour intensities of varnishes based on tung oil and amylated kamala seed oil being 2·4R-14Y and 4·0R-28Y respectively.

The time taken by the films to dry was slightly more in the case of modified kamala seed based varnishes than those prepared from tung oil based varnishes. In the case of all the formulations included in Table 3, the scratch hardness of the films was independent of the conjugating oil used. But in other formulations, varnishes made with alkylated kamala seed oil were somewhat

TABLE 1 — FORMULATIONS BASED ON PHENOL RESIN

COMPONENT g.	FORMULATION											
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	No. 11	No. 12
Bedesol 99	94·50	121·00	116·00	100·0	100·00	23·30	16·8	—	—	91·75	—	—
Bedesol 66	—	—	—	—	—	—	—	125·0	121·00	186·00	156·00	179·00
Run Congo	—	—	20·00	—	—	—	—	—	—	—	—	—
Conjugated oil	227·00	119·00	94·00	250·0	300·00	46·60	16·8	150·0	48·50	444·00	164·00	282·00
Linseed stand oil (100 p.)	—	—	—	—	—	—	—	50·0	—	—	—	—
Linseed stand oil (50 p.)	37·75	—	—	45·0	—	—	33·6	50·0	194·00	111·00	46·75	—
Refined linseed oil	—	119·00	48·00	—	—	—	—	—	—	—	—	—
Dehydrated castor oil	—	—	94·00	—	—	—	—	—	—	—	—	—
White spirit	359·00	297·00	372·00	340·0	250·00	30·10	32·8	375·0	363·25	932·00	374·00	—
Turpentine	—	—	—	30·0	50·00	—	—	—	—	—	—	—
Xylol	—	73·00	—	—	—	—	—	—	—	—	—	311·00
Cobalt naphthenate	1·50	1·94	2·10	1·0	0·75	0·23	0·5	2·0	2·00	4·52	1·25	—
Manganese naphthenate	0·75	0·85	—	—	—	—	—	—	1·50	3·42	—	—
Lead naphthenate	2·75	—	2·55	3·0	3·50	0·90	2·0	5·0	2·50	5·72	3·10	7·25
Zinc naphthenate	—	—	—	—	0·75	—	—	—	—	—	—	—
Calcium naphthenate	—	2·43	—	—	—	—	—	—	—	—	—	—

TABLE 2 — FORMULATIONS BASED ON MALEIC AND OTHER RESINS

COMPONENT g.	FORMULATION								
	No. 13	No. 14	No. 15	No. 16	No. 17	No. 18	No. 19	No. 20	No. 21
Penta ester gum	106·25	—	—	—	—	—	—	—	—
Coumarone indene resin	—	150·0	100·00	—	—	—	—	—	—
Rosin	—	—	—	100·0	100·0	—	—	—	—
CNSL modified rosin[7]	—	—	—	—	—	100·0	100·0	—	—
Ester gum	—	50·0	—	—	—	—	—	100·0	—
Congo Copal ester	—	—	—	—	—	—	—	—	60·0
Dehydrated castor oil	170·00	—	—	—	—	—	—	—	—
Conjugated oil	85·00	450·0	110·00	320·0	240·0	80·0	200·0	250·0	120·0
Linseed stand oil (14 p.)	—	—	—	—	—	40·0	40·0	—	—
Linseed stand oil (100 p.)	—	—	—	—	—	—	—	—	40·0
D.B. linseed oil	—	90·0	—	—	80·0	—	—	—	—
White spirit	361·00	700·0	140·00	480·0	620·0	200·0	340·0	350·0	200·0
Turpentine	—	—	60·00	—	—	—	—	—	—
Cobalt naphthenate	1·75	—	—	—	—	—	—	—	—
Manganese naphthenate	0·75	—	—	—	—	—	—	—	—
Lead naphthenate	2·50	—	—	—	—	—	—	—	—
Cobalt linoleate	—	4·5	0·25	—	—	1·0	1·5	0·5	—
Lead linoleate	—	5·5	1·00	4·0	6·0	2·5	3·5	—	—
Manganese linoleate	—	—	—	3·0	4·0	—	—	—	—
Calcium hydrate	—	—	—	6·0	6·0	—	—	—	—
Lead resinate	—	—	—	—	—	—	—	16·0	8·0
Manganese resinate	—	—	—	—	—	—	—	—	4·0

TABLE 3 — PERFORMANCE OF SOME PROMISING FORMULATIONS

FORMULATION No.*	CONJUGATED OIL	COLOUR INTENSITY (LOVIBOND UNITS)		DRYING TIME hr.		SCRATCH HARDNESS g.	EFFECT OF SOLVENTS ON FILMS						
		Red	Yellow	Surface dry	Hand dry		Distilled water (48 hr.)	White spirit (15 min.)	Alcohol (15 min.)	Turpentine (15 min.)	Artificial sea water (48 hr.)	NaOH (1%) (15 min.)	H_2SO_4 (3%) (15 min.)
1	Tung oil	2·0	18·0	8·0	ON	1200	NE	NE	—	W, D	—	—	—
	Amylated kamala seed oil	3·0	11·3	8·0	30	1200	MB	NE	—	W, D	—	—	—
2	Tung oil	5·4	27·0	8·0	ON	1800	NE	NE	—	W, D	—	—	—
	Butylated kamala seed oil	6·0	68·0	8·0	ON	1800	NE	NE	—	W, D	—	—	—
2a†	Tung oil	2·4	14·0	2·0	ON	2000	FB	—	NE	—	—	NE	—
	Amylated kamala seed oil	4·0	28·0	2·5	ON	2000	FB	—	NE	—	—	FB	—
9	Tung oil	4·4	38·0	2·0	ON	1500	FB	—	—	NE	FB	HB	FB
	Amylated kamala seed oil	9·1	41·0	2·0	ON	1500	MB	—	—	NE	FB	HB	FB
11	Tung oil	7·0	35·0	2·0	ON	2000	FB	—	—	SW	NE	NE	NE
	Amylated kamala seed oil	8·0	38·0	3·5	ON	2000	FB	—	—	W, D	NE	NE	NE
11a‡	Tung oil	2·0	24·0	2·0	ON	2200	NE	—	NE	—	—	NE	—
	Butylated kamala seed oil	2·1	20·0	2·0	ON	2200	MB	—	MB	—	—	FB	—
13	Tung oil	2·1	18·0	2·0	ON	1500	MB	—	FB	—	—	Dark partly dissolved	NE
	Butylated kamala seed oil	2·4	16·0	3·0	ON	1500	MB	—	FB	—	—	do	NE
18	Tung oil	28·0	68·0	1·5	ON	1800	FB	—	SD	—	—	FB	NE
	Amylated kamala seed oil	30·0	78·0	1·5	ON	1800	FB	—	NE	—	—	MB	NE

*The serial numbers refer to formulations given in Tables 1 and 2.

†The composition of the formulation was same as of 2 except that Bedesol 99 was replaced by maleic resin EPOK M4000.

‡The composition of the formulation was same as of 11 except that Bedesol 66 was replaced by maleic glycerol esterified rosin (8%).

ON, overnight; NE, no effect; FB, faint blush; MB, moderate blush; W, wrinkled; D, detached; SW, slightly wrinkled; SD, slightly detached.

inferior in this respect to those made from tung oil. The varnishes based on maleic resins had higher scratch hardness than the corresponding varnishes based on phenolic resins. Thus by replacing Bedesol 99 by EPOK M4000 in formulation 2, the scratch hardness of the film increased from 1800 to 2000 g. Similarly by replacing Bedesol 66 by glycerol esterified resin in formulation 11, the scratch hardness increased from 2000 to 2200 g.

Films obtained from most of the formulations blushed in contact with water and other solutions, but became clear again on drying. Almost all the films wrinkled and detached when immersed in benzene for 15 min.

The comparatively poor performance of varnishes based on modified kamala seed oil may be due to the fact that it contains lower amounts of conjugated acids than tung oil.

References

1. OJHA, V. N. & AGGARWAL, J. S., *J. sci. industr. Res.*, **15B** (1956), 656.
2. OJHA, V. N. *et al.*, *J. sci. industr. Res.*, **16A** (1957), 213.
3. HAFELI, J. M., *J. Amer. Oil Chem. Soc.*, **27** (1950), 523.
4. REMINGTON, J. S., *Drying Oils, Thinners and Varnishes* (Abacus Ltd., London), 1950, 115.
5. Imperial Chemical Industries Ltd., *Synthetic Resins for Surface Coatings*, Pamphlet No. 3, Second Edition, 10, 11, 24, 25.
6. McCUTCHEON, J. W., *Synthetic Detergents* (MacNair Dorland Co., New York), 1950, 95.
7. MATHUR, H. H. & AGGARWAL, J. S., *J. sci. industr. Res.*, **12B** (1953), 411.

Mineral Matter Separation in Coal by Froth Flotation

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The separation of finely disseminated mineral matter from a few low grade Indian coals (ash, 28-34 per cent) has been investigated by the froth flotation technique. The nature of the mineral matter, its distribution in different size fractions, and the influence of factors such as particle size, pre-treatment, combined action of flotation reagents on the flotation of coal have been studied.

Considerable variation is observed in the ash content of different size fractions; the ash gradually decreases from coarser to finer fractions up to 64 microns. The extent of variation between the maximum and minimum ash values is 10-12 per cent. Marked separation of mineral matter from coal with high recovery is effected by reducing coal particles to sizes in the range 64-200 microns, milling and conditioning prior to flotation, and by using Dow-Froth reagents in combination with kerosene and cresylic acid.

THE difficulties in cleaning most Indian coals arise because of the large proportion of intergrown mineral matter in them. In order to effect a clear separation of mineral matter from the associated coal, a knowledge of the nature of the mineral species present, particularly their grain size, friability and mode of distribution, is essential. While size reduction of coal promotes the liberation of small grains of minerals from it, froth flotation is the best means of effecting their separation.

The present investigation was undertaken to study the nature of the mineral matter present in some low grade Indian coals, their distribution in different size fractions obtained on crushing and sieving, and the extent of separation that can be effected by the froth flotation. The influence of factors such as particle size, milling prior to flotation, use of frother collectors, etc., in the separation of the inorganic matter in the coals has also been investigated.

Materials and methods

Two coals from Bokaro and Digwadih, and a middling from Jamadoba washing plant were employed in this study. The ultimate and proximate analyses of the samples are given in Table 1.

For the determination of the distribution of mineral matter in different size fractions obtained by size degradation, the original coal samples were crushed in a jaw crusher of the Blake type with an opening of $\frac{1}{4}$ in. and then fractionated into various size fractions using a standard B.S.S. Ash in each fraction was determined following the standard B.S.I. procedure.

Float and sink tests were made to determine the washability characteristics of the coals. The coal samples were reduced to below $\frac{1}{2}$ in. in size and the tests performed in a tall jar using mixtures of benzene-carbon tetrachloride and bromoform-carbon tetrachloride as the media. The float and sink fractions were dried, weighed and their ash content determined in accordance with the standard procedure.

TABLE 1 — PROXIMATE AND ULTIMATE ANALYSES OF COALS

COAL	ASH %	VOL. MATTER %	MOISTURE %	F.C. %	C %	H %
Bokaro*	33·8	23·4	1·1	41·7	57·0	3·1
Digwadih grade IIIB†	30·8	23·8	0·6	45·2	61·0	3·9
Jamadoba middlings†	28·5	24·6	2·2	44·7	59·1	3·1

*From Bokaro Thermal Power Station, Bihar.
†Supplied by Messrs Tata Iron & Steel Co. Ltd., Jamshedpur.

Froth flotation experiments were conducted in a Denver Sub A type flotation cell. To a properly ground coal charge of 200 g., water was added to obtain a pulp of 10 per cent solids by weight. Prior to flotation, reagents were added by means of a graduated pipette and slowly stirred for 5 min. for conditioning. In experiments in which the effect of milling of coal in water was studied, the charge (coal : water, 2 : 1) was first placed in a pebble mill, milled for periods varying from 30 to 60 min. and flotation tests were carried out following the usual procedure. The froth which flowed over the lip of the cell was collected till no more was obtained and the tailings recovered from the bottom of the cell. The froth and tailings fractions were filtered under vacuum, dried and their ash contents determined. Of the several flotation reagents tried, kerosene, cresylic acid and some reagents (DF 200 and DF 250) obtained from Dow-Froth Co., U.S.A., were found to be effective, either singly or in combinations.

Results and discussion

Washability characteristics — The washability characteristics of the three coal samples are shown in Fig. 1. The shape of the characteristic curve of Bokaro coal shows that it could be divided into three fractions: (1) a light fraction comprising 20 per cent of the raw coal with 17 per cent ash; in this fraction there is a steady increase in ash with yield; (2) a middling fraction, the ash value of which increases at a comparatively lower rate for yields between 20 and 50 per cent; and (3) a heavy portion in which there is rapid increase in ash content with yield.

The washability curve of the middlings from the Jamadoba washery does not indicate any clear-cut separation of coal from mineral matter. This is evidently due to the presence of intergrown mineral matter. However, the washability curve indicates that it may be possible to obtain a cut of 60 per cent clean coal with *c.* 22 per cent ash.

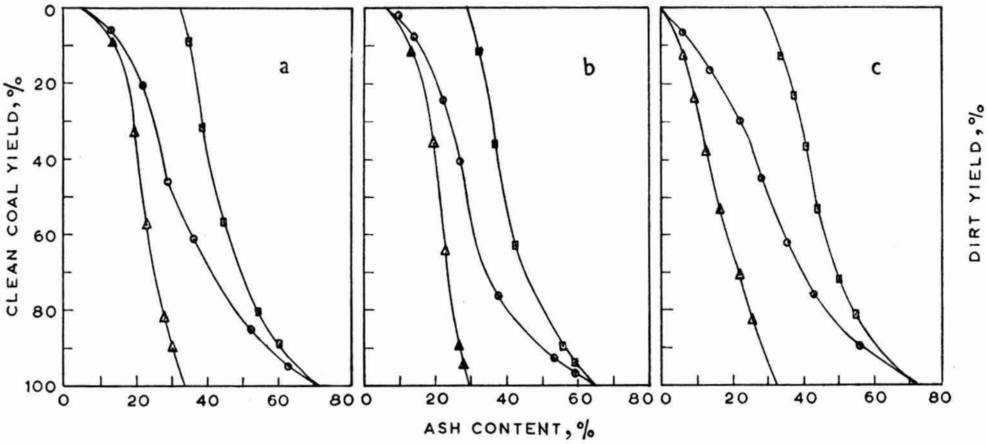


FIG. 1 — WASHABILITY CURVES FOR (a) BOKARO COAL, (b) JAMADOBA WASHERY MIDDLINGS, AND (c) DIGWADIH GRADE IIIB COAL [△—△, clean coal curve; ⊡—⊡, dirt curve; ⊙—⊙, characteristic curve]

TABLE 2 — ASH CONTENT OF DIFFERENT SIZE FRACTIONS

FRACTION *B.S. mesh*	BOKARO		DIGWADIH GRADE IIIB		JAMADOBA MIDDLINGS	
	Wt. %	Ash %	Wt. %	Ash %	Wt. %	Ash %
Original sample	—	33·8	—	30·8	—	28·5
+5	48·0	33·8	16·3	36·7	50·1	30·1
−5+10	28·4	33·9	30·4	34·5	21·1	29·6
−10+25	10·0	29·3	26·2	27·4	14·7	27·6
−25+52	5·7	25·5	10·9	25·3	6·0	24·1
−52+150	4·6	23·2	10·3	26·6	5·1	22·8
−150+200	3·0	22·1	2·0	25·3	1·1	23·5
−200	1·0	22·1	3·5	26·0	1·6	27·0

TABLE 3 — EFFECT OF MILLING

(*Digwadih grade IIIB coal; pulp density,* 15% *solids by weight; milling time,* 30 *min. at* 28°-30°*C.*; *reagent,* 2 *lb. kerosene per ton*)

COAL	ASH %	TREATMENT	YIELD OF CLEAN COAL %	ASH %	YIELD OF TAILINGS %	ASH %
Fraction −52+150 mesh	26·6	nil	50·0	17·0	50·0	37·0
do	26·6	Milled	50·0	14·0	50·0	38·0
Total coal	30·8	nil	50·0	20·0	50·0	41·0
do	30·8	Milled	53·0	16·5	47·0	45·0

TABLE 4 — FLOTATION OF THE DIFFERENT SIZE FRACTIONS

(*Reagent*: *cresylic acid-kerosene* (1:1), 1·5 *lb./ton*; *milling time,* 1 *hr.*; *pulp concentration,* 15% *solids by weight*)

FEED SIZE *B.S. mesh*	ASH %	YIELD OF CLEAN COAL %	ASH %	YIELD OF TAILINGS %	ASH %	RECOVERY OF COMBUSTIBLE MATTER %
			Bokaro coal (*ash*, 33·8%)			
+5	33·8	7·8	12·7	92·2	36·0	10·3
−5+10	33·9	14·4	11·3	85·4	38·5	19·7
−10+25	28·3	21·2	11·6	78·8	33·0	26·0
−25+52	25·5	23·5	9·6	76·5	30·0	28·2
−52+150	23·2	55·0	10·0	45·0	33·3	64·5
−150	22·1	60·0	9·6	40·0	40·0	70·0
			Digwadih grade IIIB coal (*ash*, 30·5%)			
+5	36·7	20·0	14·1	80·0	42·7	27·2*
−5+10	34·5	31·0	14·2	69·0	43·8	40·5*
−10+25	27·4	37·3	14·5	62·7	34·8	43·7
−25+52	25·3	42·4	12·6	57·6	34·7	49·5
−52+150	26·6	49·0	11·2	51·0	41·4	59·5
−150	25·7	60·0	9·6	40·0	48·2	72·3
			Middlings from Jamadoba washery (*ash*, 28·5%)			
+5	30·1	28·2	13·0	71·8	36·0	35·0*
−5+10	29·6	28·2	11·8	71·8	36·4	35·4*
−10+25	27·6	37·0	11·0	63·0	36·0	45·5
−25+52	24·1	52·0	11·7	48·0	37·5	60·5
−52+150	22·8	52·0	8·6	48·0	36·0	61·5
−150	26·0	54·4	8·5	45·6	45·0	67·0

*Quantity of reagent was doubled.

TABLE 5 — PARTICLE SIZE ANALYSIS OF COAL SAMPLES

(*Yield expressed as per cent of total charge*)

FEED SIZE *B.S. mesh*	FLOTATION	YIELD OF CLEAN COAL OF PARTICLE SIZE (*B.S. mesh*)							
		+5	+10	+25	+52	+150	+200	+300	−300
				Bokaro coal (*ash*, 33·8%)					
−5+10	Froth	—	—	—	—	0·7	0·7	11·5	2·1
	Tailings	—	44·0	15·3	2·3	7·0	2·3	10·2	4·2
−10+25	Froth	—	—	—	1·5	4·3	1·5	11·7	2·1
	Tailings	—	—	28·2	17·5	16·3	5·5	9·4	1·7
−25+52	Froth	—	—	—	—	6·5	2·8	12·9	1·2
	Tailings	—	—	—	0·8	32·0	21·8	17·6	3·1
−52+150	Froth	—	—	—	—	3·1	12·7	37·8	1·6
	Tailings	—	—	—	—	1·4	2·9	32·0	8·7
−150	Froth	—	—	—	—	—	22·8	39·6	1·4
	Tailings	—	—	—	—	—	8·0	30·0	1·8
				Jamadoba washery coal (*ash*, 28·5%)					
+5	Froth	—	—	—	0·3	2·5	8·8	15·0	1·7
	Tailings	28·3	14·7	4·4	2·9	4·6	5·9	8·7	2·3
−5+10	Froth	—	—	0·3	0·3	6·0	7·8	12·5	2·2
	Tailings	—	48·0	14·4	1·2	1·2	0·7	3·7	3·7
−10+25	Froth	—	—	—	0·5	2·1	4·3	26·8	3·2
	Tailings	—	—	40·0	10·3	4·1	0·6	4·1	4·1
−25+52	Froth	—	—	—	—	11·2	11·2	26·5	3·3
	Tailings	—	—	—	0·7	6·8	15·2	21·4	3·9
−52+150	Froth	—	—	—	—	0·6	13·4	33·8	3·9
	Tailings	—	—	—	—	4·7	1·6	34·0	7·7
−150	Froth	—	—	—	—	—	3·9	46·5	4·1
	Tailings	—	—	—	—	—	5·9	30·6	9·0

The washability curve of Digwadih grade IIIB coal shows that the ash content of the fractions steadily rises with increase in the yield of clean coal, indicating thereby that it is difficult to wash this coal.

Distribution of ash with size degradation — Since size reduction of lump coal liberates the finely disseminated mineral matter from the matrix of coal, the distribution of ash in different size fractions obtained by crushing and screening of the coal samples was studied. The results obtained for the coal samples are given in Table 2.

Table 2 shows that the trend in the variation of ash content from coarser to finer size fractions is similar for all the coal samples. In general, the ash content gradually decreases in the finer sizes up to 200 mesh. In fines below this size there is again a slight increase in ash. The extent of variation between the maximum and minimum ash values is quite marked. In Bokaro coal it is about 12 per cent; in Digwadih grade IIIB, 11·7 per cent; and in Jamadoba middlings, 7·0 per cent.

Effect of milling — To determine the effect of milling pre-treatment prior to froth flotation, experiments were performed under controlled conditions. Kerosene, which acts as a mild frother as well as a collector, was used as the reagent. A few of the typical results obtained with Digwadih grade IIIB coal and one of its fractions (−52+150 mesh) are given in Table 3.

The beneficial effect of milling on flotation will be evident from the improved yield of 53 per cent clean coal with 16·5 per cent ash as against 50 per cent clean coal with 20 per cent ash without milling. This may be due to the fact that in the case of these coals milling, which involves both impact and shearing forces, leads to further particle size reduction as well as liberation of the hydrophobic coal particles from the finely disseminated hydrophilic mineral matter.

Influence of particle size — In view of the considerable variation in the ash content of different particle size fractions, a detailed study of the flotation characteristics of the fractions was carried out. In Table 4 are given the results obtained for the three coals.

It can be seen from Table 4 that fractions having feed sizes < 52 mesh can be cleaned satisfactorily. Flotation recoveries were best on sizes < 150 mesh.

Since each fraction was milled in water prior to flotation, further size reduction of

TABLE 6 — PARTICLE SIZE ANALYSIS OF FLOTATION FRACTIONS

(*Digwadih grade IIIB*; *ash*, 30·8%)

FEED SIZE *B.S. mesh*	ASH %	FLOTATION FRACTION	ANALYSIS OF FRACTIONS															
		Particle size range *B.S. mesh*	+5		−5+10		−10+25		−25+52		−52+150		−150+200		−200+300		−300	
		Av. size of particles*	—		94·5		42·7		17·0		7·6		3·4		2·4		1·0	
			Yield %	Ash %	Yield %	Ash %	Yield %	Ash %	Yield %	Ash %	Yield %	Ash %	Yield %	Ash %	Yield %	Ash %	Yield %	Ash %
+5	36·7	Froth	—	—	0·1	25·2	0·3	17·4	2·3	14·6	7·7	14·6	4·9	14·8	3·7	12·3	1·1	13·9
		Tailings	31·0	45·5	26·6	38·6	3·8	40·0	1·7	43·0	3·2	47·7	1·4	47·7	8·2	44·0	3·8	52·0
−5+10	34·5	Froth	—	—	—	—	0·4	19·1	0·6	18·5	3·6	12·1	1·5	15·7	15·5	14·5	9·4	14·4
		Tailings	—	—	36·0	40·5	19·0	40·5	2·8	40·5	4·7	39·0	1·3	41·0	3·5	53·7	2·5	55·7
−10+25	27·4	Froth	—	—	—	—	1·7	16·4	4·0	14·7	5·1	12·0	1·7	12·0	21·0	14·7	3·7	15·0
		Tailings	—	—	—	—	30·0	41·0	14·9	36·0	8·3	36·8	1·4	42·0	5·8	53·7	2·6	71·0
−25+52	25·3	Froth	—	—	—	—	—	—	0·5	9·5	5·0	10·7	3·4	10·8	32·6	12·8	1·0	14·1
		Tailings	—	—	—	—	—	—	1·8	32·8	22·8	30·8	7·2	27·2	23·8	41·5	1·8	50·5
−52+150	26·6	Froth	—	—	—	—	—	—	—	—	0·5	10·9	11·0	11·4	36·0	11·3	1·9	14·0
		Tailings	—	—	—	—	—	—	—	—	0·5	32·8	1·2	39·8	46·0	40·8	3·9	47·2

*The values are obtained by multiplying the average size of the particles (expressed in microns) by the factor 0·03774 which is the reciprocal of the average particle size, .e. 26·5 microns of the −300 mesh fraction.

particles of the original feed occurs. A closer analysis of the results was, therefore, sought through the particle size analysis of the froth and tailings. The size analysis was carried out manually by dry sieving[1]. The sieves were frequently tapped and a light brush was used to prevent clogging of the holes. The results are presented in Tables 5 and 6.

It will be seen from Tables 5 and 6 that it is difficult to float +5 mesh size particles and the flotability of –10 to +52 mesh sizes is poor under the operating conditions employed. An examination of the screen analyses of the products of flotation shows that particles of sizes ranging from 64 to 201 microns may be considered as the optimum size range for maximum recovery by flotation of the coals.

Influence of reagents — The flotation efficiency of kerosene, cresylic acid and of reagents obtained from the Dow-Froth Co., U.S.A., was studied with Digwadih grade IIIB coal. Some typical results are given in Table 7. It will be observed that with 1·37 lb./ton of 1: 1 cresylic acid-kerosene mixture optimum results were obtained. The yield of clean coal was 63·3 per cent (77·0 per cent combustibles recovery) with 17·1 per cent ash.

Two Dow-Froth reagents were tested and of these Dow-Froth 200 gave best performance with a dosage of $\frac{2}{3}$ lb./ton. The yield of clean coal was 75 per cent (88·0 per cent combustibles recovery) with 19·1 per cent ash. The combined action of cresylic acid, kerosene and Dow-Froth 200 was next studied. It will be seen that high yields of clean coal can be obtained by using as little as 1 lb./ton of a mixture of these reagents in equal proportions.

The results of flotation tests on Bokaro and Digwadih coals and Jamadoba washery middlings using 1: 1 cresylic acid-kerosene mixture are presented in Table 8. Digwadih grade IIIB coal yields 68·5 per cent clean coal with 18·2 per cent ash amounting to about 81·0 per cent combustibles recovery. From Jamadoba washery middlings also a similar high yield of 68·5 per cent clean coal with 15·0 per cent ash was obtained. With

TABLE 7 — FLOTATION OF DIGWADIH GRADE IIIB COAL

[*Digwadih grade IIIB coal; reagents: cresylic acid (C), kerosene (K), Dow-Froth (DF); milling time, 30 min.; original size, <30 mesh; pulp concentration, 10% solids by weight*]

YIELD OF CLEAN COAL %	ASH %	YIELD OF TAILINGS %	ASH %	COMBUSTIBLES RECOVERY %	REAGENT *lb./ton*
30·0	14·0	70·0	37·0	37·0	1·0 K
53·0	16·5	47·0	45·0	64·0	2·0 K
45·0	15·7	55·0	46·0	55·0	1·0 C
59·0	17·0	41·0	49·0	70·5	1·5 C
34·0	13·0	62·0	41·0	42·8	1·0 (K+C)
57·0	17·0	43·0	49·0	69·0	1·25 (K+C)
62·0	17·6	38·0	52·0	74·0	1·37 (K+C)
63·3	17·1	36·7	54·5	77·0	1·37 (K+C)
68·0	18·8	32·0	56·0	80·0	1·5 (K+C)
68·5	18·2	31·5	59·1	81·5	1·5 (K+C)
48·0	12·7	52·0	46·3	60·6	0·33 DF 200
75·0	19·1	25·0	61·0	88·0	0·66 DF 200
78·0	20·6	22·0	63·5	90·0	1·00 DF 200
44·0	12·3	56·0	44·6	57·7	0·33 DF 250
74·0	19·5	26·0	55·0	86·5	0·66 DF 250
77·0	22·0	23·0	63·0	88·0	1·00 DF 250
57·0	14·0	43·0	51·0	69·5	0·33 DF 200+0·66 K
54·0	12·9	46·0	50·0	63·4	0·33 DF 200+0·66 K
61·0	16·4	39·0	52·5	74·0	0·33 DF 200+0·33 C +0·33 K

TABLE 8 — FLOTATION OF DIFFERENT COAL SAMPLES

[*Size, <30 mesh; conditioning time, 5 min.; milling time, 30 min.; temp., 28°C.; reagent ratio, cresylic acid-kerosene* (1: 1), 1·5 *lb./ton*]

COAL	ASH %	CLEANS		TAILINGS		COMBUSTIBLES RECOVERY %
		Yield %	Ash %	Yield %	Ash %	
Bokaro coalfield	33·8	47·4	10·9	52·6	51·0	64·0
		65·0	16·5	35·0	66·0	82·0
Digwadih grade IIIB	30·8	68·5	18·2	31·5	59·1	81·5
		63·3	17·1	36·7	54·5	77·0
Jamadoba washery middlings	28·5	68·5	15·0	31·5	59·1	81·0
		60·0	14·3	40·0	47·8	71·5

Bokaro coal, for an yield of 47·4 per cent clean coal with 11 per cent ash, the recovery of combustibles is 64·0. For a higher yield (65 per cent), the ash content of the clean coal is 16·5 per cent and the recovery of combustibles is 82·0 per cent.

Summary

The results of investigations on the separation of mineral matter present in two low grade coals from Bokaro and Digwadih and middlings from Jamadoba washery with an ash content ranging from 28·5 to 33·8 per cent are presented.

The different size fractions of coals obtained by size reduction and screening show considerable variation in their ash contents, the trend in variation being similar with all the coals.

A gradual decrease in ash content is observed from coarser to finer sizes up to 64 microns; the variation between maximum and minimum ash values is 10-12 per cent.

Froth flotation tests show that size reduction of coal particles should be in the range 64-200 microns for high flotation recovery. Pre-treatment and conditioning including milling prior to flotation promote better mineral matter separation. Of the various flotation reagents tested, a mixture of cresylic acid and kerosene, has been found to be effective. For example, with Digwadih grade III coal containing 30·8 per cent ash, the recovery of combustibles is 77·0 per cent with an ash content of 17·0 per cent. Dow-Froth reagents, alone or in combination with the above reagent, effect better separation. The maximum recovery of combustibles that can be effected in the case of Digwadih coal is 90 per cent with an ash content of 20·6 per cent; the maximum reduction in ash content that could be effected is 8-9 per cent with a recovery of 60-65 per cent of combustibles.

Reference

1. *Standard Method of Test for Screen Analysis of Coals, A.S.T.M. Designation:* D410-38, *A.S.T.M. Standards on Coal and Coke,* 1947.

Nature of Coal—A Symposium

A THREE-DAY SYMPOSIUM ON THE 'NATURE of Coal', organized under the joint auspices of the Central Fuel Research Institute, Jealgora, the Institution of Chemists, India and the Institute of Fuel, U.K. (India Branch), is proposed to be held from 9 to 11 February 1959 at the Central Fuel Research Institute, Jealgora. Delegates from Australia, Canada, Czechoslovakia, France, Germany, Japan, the Netherlands, Poland, U.K., U.S.A. and U.S.S.R. are being invited to participate in the symposium and contribute papers to it.

Six technical sessions covering the following subjects have been planned: I. *Origin and Systematics of Coal*; II. *Petrographic and X-ray Studies of Coal*; III. *Ultra-fine Structure of Coal* (Sorption of Polar and Nonpolar Liquids; Heat of Wetting; Surface Area of Coal; and Solvent Extraction of Coal); IV. *Coal Constitution*; *Physical Methods* (Density; Reflectance; Infrared and Ultraviolet Spectroscopy, etc., and Ultrasonic and Other Methods); V. *Coal Constitution*; *Chemical Methods* (Oxidation; Hydrogenation; Hydrogenolysis; Halogenation; Sulphonation; Functional Groups and Pyrolysis); VI. *Physico-chemical Properties of Coal* (Electrical and Magnetic Properties; Mechanical Properties; Thermal Properties; Behaviour of Coal on Heating; and Reactivity of Coal).

The papers (not exceeding 3000 words) together with abstracts are requested to be sent to the Director, Central Fuel Research Institute, P.O. Fuel Research Institute, Dhanbad District, Bihar, India so as to reach him not later than 31 October 1958.

REVIEWS

CORROSION AND WEAR HAND BOOK FOR WATER COOLED REACTORS. Edited by D. J. DePaul (McGraw-Hill Book Co. Inc., New York-Toronto-London), 1957. Pp. ix + 293. Price $ 6.00

This handbook is the sixth in a series on basic reactor technology sponsored by the Naval Reactors Branch of the U.S. Atomic Energy Commission and is devoted to the use of high purity water as a heat-transfer medium in nuclear reactors. Many research organizations had collaborated in the project and this volume brings together and correlates their pertinent findings into a single source and in a readily available form to the engineering profession.

The handbook is divided into three parts. Part A, which is introductory, comprises of four chapters. It provides general background and principles of corrosion and wear. Emphasis is laid on the factors which distinguish the behaviour of water in reactor systems from that in conventional steam plants. The role of nuclear irradiation in producing induced radioactivity receives special attention.

Part B of the handbook consists of four chapters. In it are presented tabulated data and detailed information for reference use in actual design work. Basic information is provided on methods of testing, and on the inherent corrosion and wear resistance of various materials and combinations of materials under different environmental conditions. The design of autoclaves, loops and multi-channel circuits is described.

Part C of the handbook consists of six chapters and deals with special corrosion and wear problems such as crevice corrosion, stress corrosion and intergranular corrosion; build-up of corrosion products in recirculating systems; wear resistance of materials; and impact of manufacturing procedures on corrosion and wear resistance.

Much of the information on the behaviour of water in nuclear reactors is widely dispersed in technical literatures, and the editor has done a great service in bringing out this volume which will be of immense value to the engineer and the scientist concerned with the design of nuclear plants. Exhaustive bibliographies at the end of each chapter greatly add to the value of the book. An excellent feature of the publication is the stress on the many unsolved problems in corrosion and wear, with special reference to the use of water in a nuclear power plant.

BRAHM PRAKASH

A SHORT COURSE IN QUANTITATIVE ANALYSIS by H. H. Willard, N. H. Furman & E. K. Bacon (D. Van Nostrand Co. Inc., Princeton, New Jersey), Second Edition, 1957. Pp. vi + 243. Price $ 4.75 or 35*s*.

The aim of this book is to provide clear and detailed instructions for laboratory work and to present commonly accepted analytical procedures in a simple and coherent form to the undergraduate students.

Of the 15 chapters in this book, the first five deal with elementary but fundamental principles of laboratory practice, methods of measurements and evaluation of errors. The remaining chapters describe different classical methods of volumetric and gravimetric analysis with special emphasis on the theoretical principles involved in the particular procedure under review. Every chapter is appended with a variety of problems having a direct bearing on the topic discussed; by solving these the student is sure to grasp the theoretical aspect of the analytical procedure. The technical applications of quantitative analysis have also been described with reference to the analysis of brass and limestone. The chapter on analysis by physical measurements, however, includes only the colorimetric and potentiometric methods. Thus, the attempt to familiarize the student with recent concepts of quantitative analysis falls short by several years.

The judicious combination of theoretical principles and practical applications of various analytical procedures is indeed an excellent idea as it brings home to the student the necessity of gaining good familiarity with fundamental theories for a proper appreciation and co-ordination of the factual aspects of analytical chemistry. The book is remarkably free from errors and will prove of invaluable assistance to students and teachers interested in inorganic analytical chemistry.

G. S. DESHMUKH

WELDING HANDBOOK: Part I. Edited by Arthur L. Phillips (American Welding Society, New York; *Distributors*: Cleaver-Hume Press Ltd., London), 1957. Pp. 555. Price 72*s*.

The fourth edition of this handbook is a thorough revision and substantial enlargement of the third edition published in 1950 and is a reference volume for all those interested in welding as a manufacturing process. The aim of the editors in enlarging and recasting the book appears to have been to make available all the information pertaining to welding in one book. In this attempt they have succeeded in a large measure.

The new volume has been recast into five independent volumes, each on a single broad aspect of welding. The present volume entitled " Basic Principles and Data " is the first in the series. The remaining four volumes to be published deal with welding processes, miscellaneous processes and cutting, metals and their weldability, and applied welding.

The handbook is intended to serve as a comprehensive source of information for engineers in the design, fabrication and inspection of welded construction. It is evident from the contents of this volume that the subject matter covered in the five volumes will be extensive and comprehensive. The publication is profusely illustrated which makes for easy understanding of the underlying principles and techniques of welding.

The first volume contains 500 pages, 220 illustrations and 90 data tables. It starts with illustrated definitions and proceeds to discuss such aspects as welding metallurgy; properties and design of welded joints; thermal and mechanical treatment of weldments; testing and inspection of welds; statistical quality control in welding; cost estimating; and safe practices in welding. The chapter on " Thermal and Mechanical Treatments of Weldments " is particularly well written.

The volume contains exhaustive bibliography and one can always find a reference to the literature for extensive and intensive study of any aspect of welding. Tables containing data on metals, gases, etc., provide valuable aid to welding engineers.

One of the aims of the editors in publishing this book in five separate volumes and adopting modern method of numbering has been that this arrangement affords an opportunity to effect changes without delay and permit easy incorporation of fresh matter. This is a happy idea since it is realized that welding is not an exact science as yet and much remains to be investigated and established. More up-to-date information on aspects such as corrosion resistance, adherance of organic and inorganic coating on weldments, plant and equipment, brazing, and soldering will considerably enhance the value of the handbook.

K. G. CHANDIRAMANI

INTRODUCTION TO HEAT TRANSFER by Aubrey I. Brown & Salvatore M. Marco (McGraw-Hill Book Co. Inc., New York-Toronto-London), 1958. Pp. vii + 332. Price $ 6.75

This book is primarily meant for engineering students of the third and fourth years in a university and is particularly useful to the beginning student.

The earlier part of the book deals with the principles and theories governing the different mode of heat transfer, namely conduction, convection and radiation. To facilitate the understanding of heat transfer problems, the theory of each process has been treated separately and then its application to the solution of actual problems has been considered together with the experimental work of various investigators.

There are useful chapters on fundamental units and dimensional analysis, and fluid flow in the convection process. These chapters will be of considerable help to students in understanding the principles governing heat transfer. An important chapter in the book is the one dealing with the application of the principles of heat transfer to design problems. Calculations of heat transfer in feed-water heaters and surface condensers, through fin-tube fan coils, and in electrical transformers, have been dealt with in this chapter. The purpose of the author in presenting this chapter is twofold: firstly to indicate the manner and extent in which the principles of heat transfer may be applied in the design of certain forms of heat exchangers, and secondly to give students further practice in applying these principles to design problems. Graphical and numerical methods for solving heat conduction problems have been treated in the last chapter.

The book conforms to the high standards one is accustomed to with all McGraw-Hill publications. The printing and get-up are excellent.

M.D.P.

DECHEMA-MONOGRAPHIEN NR. 392-410, BAND 29 — Scientific Fundamentals Concerning Chemical Reactions on an Industrial Scale (Verlag Chemie GMBH Weinheim'Bergstrasse), 1957. Pp. 380. Price about Rs. 50.00

This publication is a collection of nineteen papers presented at the Thirty-first Annual Session of the Dechema (German Society for Chemical Apparatus) held at Frankfurt am Main in 1956.

The first of these is a discussion of the " Problems of Chemical Technology in Research, Education and Industry " by Prof. K. Winnacker, the present Chairman of this Society. Devoting himself mainly to the education of industrial chemists, the learned professor states that the time has now come for German universities and engineering colleges (Technische Hochschule) to adapt their syllabuses more closely to the present requirements. He holds the view that the method of co-operation between the chemist and the engineer, which has proved to be so successful in German industry, may be allowed to continue, but that it is necessary to orientate the training of chemists more towards technology. The way to do this is to teach them not only the historical development of chemical technology but also to emphasize the guiding principles that governed this development. This would impart to them an insight into industrial operations, and by enabling them to speak and understand the language of their engineering colleagues, help them to play their full part in the future development of the industry, particularly in the direction of rationalization and automation.

It must be noted that Prof. Winnacker does not consider it advisable for Germany, under the present conditions, to adopt the Anglo-Saxon method of training chemical engineers, that is to teach the trainees the basic principles of both chemistry and engineering. He thinks that special institutes should be set up by industry which should devote themselves to research and not concern themselves with teaching. Teaching can best be done in the wider framework of a university or engineering college where fruitful contacts with other branches of science are possible. Teaching, if it is not to become dead and sterile, must go hand in hand with research, a principle that has been the great, living tradition of all higher education in Germany.

Three papers (Nos. 393-395) deal respectively with the reactions of hydrogen, carbon monoxide, and oxygen at high temperatures and under high pressure, while three more (Nos. 396-398) deal with some aspects of gaseous reactions and their kinetics. Dr. Hoffmann describes in his paper (No. 399) a simple, graphical method that might prove useful for the design of a cylindrical, solid bed reactor, while Dr. Patat discusses the possibilities of kinetic measurements on enzyme reactions, using dextrane sucrase as an example (No. 400). Electrochemistry is represented by a very practical paper, " The Splitting of Hydrocarbons by Small Electric Arcs " (No. 401), and physical chemistry by a theoretical paper on " The Importance of the Fermi Potential of Catalysts for Reaction Control ". The next three papers (Nos. 403-405) are concerned with process technology, or unit operations; the first of these is a study of the causes of self-ignition of carbon in adsorption installations for solvent separation, the second is " A Systematic Classification of Chemical Process and Apparatus ", and the third discusses a typical flowsheet in nuclear engineering. Four of the remaining papers (Nos. 406-409) treat certain selected topics connected with gas bubbles and liquid drops, and the problems of bubble columns and uniform spraying. In the last paper (No. 410) the authors refer to a new ' adsorption theory ' of O. Fuchs as applied to the separating effect of rectifying columns and observe that " in the case of mixtures of methanol and ethanol, where nickelled fillers are most effective, one may think of binding forces between the hydroxyl groups and the nickel surface ".

Two general conclusions can be drawn from these papers: First, that the scientific method of enquiry yields tangible profits even in complex fields such as reaction kinetics, and secondly, that the theoretical study of a problem can furnish guiding principles for design that reduce experimentation to a minimum, and eliminate the haphazard and wasteful attempts that had hitherto provided empirical solutions.

K. M. DASTUR
Y. K. RAGHUNATHA RAO

DIGITAL COMPUTER, COMPONENTS AND CIRCUITS by R. K. Richards (D. Van Nostrand Co. Inc., Princeton-New Jersey-New York), 1958. Pp. vii + 511. Price 64*s*.

In this book, the author sets himself a specific task, i.e. that of supplying engineers with "information needed to reduce the ideas about arithmetic and logic to a working machine". Stress is laid on the various electronic devices that make up a computer while the actual treatment of the basic arithmetic and logic involved in computing operations is left to a companion volume entitled *Arithmetic Operations in Digital Computers* by the same author. The electronics information is given in a simple and straightforward manner with surprisingly small use of involved mathematics or analysis. The treatment is, nevertheless, very thorough, comprehensive and of a high calibre. An attempt is made not only to present the basic and time-proven components and circuits but also to treat the more recent advances as well as discuss improvements which might still be considered to be in the experimental or laboratory stage.

After giving general concepts about the field of computers in a chapter on "History and Introduction", the author begins with diode switching circuits, first with the basic units leading on into diode arrays and diode matrices. The subsequent chapters III, IV and V are devoted to the three major 'revolutions' in computer circuitry. Chapter III presents circuit logic using vacuum tubes which essentially led to the field of electronic computers ... the 'flip-flop', the 'ring-counter' and systems and combinations of basic circuits. In the same way, Chapter IV treats the presently developing art of transistor circuit logic; the author very succinctly points out the basic differences and similarities between transistors and tubes and then goes on to treat transistor circuits for computers. Chapter V deals with the use of magnetic cores in computers. The 'shifting circuits', which form the central idea of the magnetic core system, are thoroughly explained and developed into combinations for logical functions.

The next three chapters deal with descriptions of the important systems of information storage. Chapter VI treats storage in various kinds of non-magnetic devices. Chapter VII is on magnetic surface storage and Chapter VIII on magnetic core storage. The author brings out a clear though elementary picture of the subject, without recourse to any of the advanced physical concepts required for a deeper understanding.

A chapter is devoted to the important problem of decimal counting in order to bring about correspondence between the binary and switching operations of the computer and the common decimal usage. The subsequent chapter entitled "Miscellaneous" comprises short and terse treatments of several of the latest developments which might still be said to be in the experimental stage; the basic idea of each case is outlined and developed to indicate its contribution to the computer art and its future importance. The final chapter treats various methods of converting data from analogue-to-digital and vice versa.

The book should be especially useful to workers who wish to set about building a computing system. The stress is almost completely on describing the job that each component or circuit is to perform; the very minimum of space is devoted to the analysis of the circuit or a discussion of its basic principles of design which, in most cases, are treated in books on circuit theory. The fact that the information is up to date can be judged from the references at the end of each chapter; the majority of the articles referred to were published within the past five years. It is no small achievement to combine the latest advances into a cohesive and useful volume, especially when obsolescence sets in on some techniques almost overnight.

B. H. WADIA

FLAME PHOTOMETRY by F. Burriel-Marti & J. Ramirez-Munoz (Elsevier Publishing Co., Amsterdam-London-New York-Princeton; *Distributors*: Cleaver-Hume Press Ltd., London), 1957. Pp. xii + 531. Price 65*s*.

On account of the simplicity of technique, flame photometry is increasingly used in the detection of metals, particularly alkali metals and alkaline earths. Thus, for ascertaining the presence of sodium and potassium in biological fluids or in soils and plant materials, flame photometry is extensively utilized. A book devoted to such subject is, therefore, welcome, since there are not many books exclusively devoted to flame photometry, though there are a large number of papers dispersed in scientific journals. The authors are staff members of the Analytical Chemistry Department at the University of Madrid. They have taken great pains in collecting the material as evidenced by the fact that the bibliography covers fifty pages incorporating nearly 900 references.

The principal part of the flame photometer is the burner, in which is introduced a sample, usually in solution, through various forms of spray-producing devices. The light emitted is selected at different wavelengths, either by monochromators or interference filters. The emission is to be detected either by a photographic plate or by photocells. In the first hundred pages are discussed the design of the burner, different forms of atomizers, selection of wavelengths and the detecting systems, both photographic and photoelectric.

In the following 35 pages are described various commercial photometers. In most cases photographs of the outward housing are given which do not give any idea of the details about the burner, atomizer or the detecting system. Also, nothing is mentioned about the quantity of samples required for analysis.

Though the flame photometer is a convenient source for excitation of spectra, the number of elements which can be excited is restricted, owing to the limited energy of the flame. Moreover, the presence of other elements produces interference which reduces the sensitivity. All such factors on which sensitivity depends are discussed in detail in Part IV extending over 60 pages. The following 75 pages are devoted to the practical details of preparing the sample for flame photometry and methods of measurement.

Part VI (125 pages) is the most useful section of the book, giving details of applications to various fields of analysis, such as the analysis of (i) natural and industrial waters, (ii) liquid mineral products, (iii) foodstuffs, and (iv) liquids of biological origin such as blood.

The book is comprehensive and contains much information useful to those who wish to utilize flame photometry as an analytical tool.

W. M. VAIDYA

EPOXY RESINS AND THEIR APPLICATIONS AND TECHNOLOGY by Henry Lee & Kris Neville (McGraw-Hill Book Co. Inc., New York-Toronto-London), 1957. Pp. 305. Price $ 8.00

Epoxy resins developed during the last few years offer a new class of resins, having excellent adhesion, good flexibility, improved chemical resistance and superior electrical properties. By virtue of this unique combination of properties, they are increasingly used in several industries and in spite of their high cost they have replaced some of the conventional materials.

Several research laboratories are engaged in the development of these products and many original papers on the subject have appeared during the last few years. The book under reference provides both theoretical and practical information on these resins and can be classified as a comprehensive guide on this subject. All the relevant information scattered in numerous publications during the last ten years or so has been well arranged and presented in a very lucid and readable manner for the general use of chemists, technologists and electrical engineers.

The book is divided into 12 chapters, covering syntheses, raw materials, curing agents and mechanism of curing, modifiers and such varied industrial applications as casting, potting, sealing, adhesion, lamination, coatings and safety precautions in their handling. The treatment of the mechanism of curing and the physical and chemical properties of the end products obtained with different curing agents is quite comprehensive. There is a good account of the general structures and related properties of cured resins. Methods for characterizing both cured and uncured resins are also presented. Four chapters are exclusively devoted to the applications of these resins and the exact role of each constituent of particular formulation is clearly brought out. There is, however, no mention of certain special uses of these resins, e.g. use as stabilizers for polyvinyl chloride and other chlorinated compounds. The inclusion of practical formulations for various applications and tabulated data are important features of the book. It would have been helpful if, while emphasizing special properties, a few typical examples of practical applications had also been included.

Some of the information has been collected from sources not readily accessible and it serves to answer some of the questions on the practical use of these resins. The book is recommended as a useful guide to all those engaged in research and practical utilization of these versatile products. The paper and quality of printing are excellent.

S. L. KAPUR

QUANTITATIVE PHARMACEUTICAL CHEMISTRY by Glenn L. Jenkins, John E. Christian & George P. Hager (Blackiston Division, McGraw-Hill Book Co. Inc., New York-Toronto-London), Fifth Edition, 1957. Pp. xviii + 552. Price $ 8.50

Prof. Jenkins, Dean of the School of Pharmacy at Purdue University, has long experience as a professor of pharmaceutical chemistry and has been intimately associated for several years with all the salient phases of pharmaceutical activity in the United States. Dr. Christian is a professor of pharmaceutical chemistry at the Purdue University and Dr. Hager is a senior scientist with the Smith, Kline and French Laboratories, a reputed pharmaceutical manufacturing house in the United States. A book written by such specialists is bound not only to give an authoritative account of the subject it is meant to cover, but also to take special note of those difficulties which a young student has to be helped out of, and which only an experienced teacher can visualize. Judged from the pharmaceutical student's point of view, the book is a useful one.

The book is divided into three parts. Part I (230 pages) deals with general methods, Part II (152 pages) with special methods and Part III (143 pages) with physico-chemical methods used in the official pharmaceutical analyses of the *United States Pharmacopoeia* (*U.S.P.*). In the part dealing with general methods there are chapters dealing with principles of gravimetric analysis and of volumetric analysis, besides chapters on gravimetric methods, alkalimetry, acidimetry, precipitation methods, oxidation-reduction methods, iodimetric and iodometric methods and gasometric methods. The chapters under special methods deal with topics like determination of moisture, ash, extractives, crude fibre, analytical constants of fats, waxes, resins and balsams, assay of volatile oils, alkaloidal assays and assay of enzyme-containing preparations. It is this part which gives this book its distinctive pharmaceutical character, since matter of this type is usually not included in books on general analytical chemistry. The part dealing with physico-chemical methods includes chapters on specific gravity, melting and boiling temperatures, solubility, viscosity, refractivity, optical rotation, *p*H, colorimetry, nephelometry and turbidimetry, spectrophotometry and fluorophotometry, chromatography and radioactivity. There is also a 3-page chapter on electro-analysis (under the name electrolytic methods). A section on potentiometric titrations dealing mainly with acid-base titrations and the Karl-Fischer method of moisture determination is found at the end of the chapter on hydrogen ion concentration.

The following mode of treatment has been generally adopted in most chapters and sections. First the principles underlying the use of the method are explained, then follow one or two typical experiments with supplementary explanations wherever required, and then comes a table listing all the *U.S.P.* substances or preparations which require to be examined by the particular method, together with the *U.S.P.* standards for them. As the authors mention in the preface, the book is intended to cover the quantitative chemical and physical methods employed in the *U.S.P.* and the *National Formulary*, but it can largely serve the student for understanding the quantitative methods of other pharmacopoeias as well. At the end of most sections and chapters, there are appended some questions and problems of relevance to the matter discussed in the sections. The printing and get-up are good.

As far as the reviewer is aware, there are few books in the English language available to the pharmaceutical student and teacher to help them in the field of quantitative analysis. The book under review, which, according to the preface, reflects the changes that have taken place in the content of the *U.S.P.*, will fulfil an important need in the field of pharmaceutical education. It is the reviewer's opinion that every student of pharmacy and teacher of pharmaceutical analysis in the English-speaking parts of the world should own a copy of this book and that it should find a place in the libraries of all institutions imparting instruction in pharmacy.

S.R.

PUBLICATIONS RECEIVED

THE NEW INDIA — PROGRESS THROUGH DEMOCRACY. Published by the Planning Commission, Government of India (Macmillan Company, New York), 1958. Pp. x + 412. Price $ 2.50

THE SPECTROSCOPY OF FLAMES by A. G. Gaydon (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), 1957. Pp. ix + 279. Price 50*s*. net

GUIDED WEAPONS by Eric Burgess (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), 1957. Pp. 225. Price 25*s*. net

ROCKETS, MISSILES AND SPACE TRAVEL by Willy Ley (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), Revised Edition, 1957. Pp. xv + 528 + 31 Plates. Price 50*s*. net

SATELLITES AND SPACE FLIGHT by Eric Burgess (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), 1957. Pp. vii + 159. Price 21*s*. net

DESIGN OF CONCRETE STRUCTURES, McGraw-Hill Civil Engineering Series by Leonard Church Urquhart, Charles Edward O'Rourke & George Winter (McGraw-Hill Book Co. Inc., New York-Toronto-London), Sixth Edition, 1958. Pp. ix + 546. Price $ 8.00

LANDING GEAR DESIGN by H. G. Conway (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), 1958. Pp. viii + 342. Price 56*s*. net

AIRCRAFT HYDRAULICS, VOL. 2 — COMPONENT DESIGN by H. G. Conway (Chapman & Hall Ltd., London; *Distrubutors in India*: Asia Publishing House, Bombay), 1957. Pp. ix + 198. Price 45*s*. net

SYNCHRONOUS MOTORS AND CONDENSERS by D. D. Stephen (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), 1958. Pp. ix + 500. Price 60*s*. net

IRRIGATION AND HYDRAULIC DESIGN, Vol. 2, by Serge Leliavsky (Chapman & Hall Ltd., London; *Distrubutors in India*: Asia Publishing House, Bombay), 1957. Pp. xvi+864. Price 14 Guineas net

ORGANIC ELECTRODE PROCESSES by Milton J. Allen (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), 1958. Pp. xiv + 174. Price 32*s*. net

ELECTRONIC MEASURING INSTRUMENTS by E. H. W. Banner (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), Second Revised Edition, 1958. Pp. xvi + 496. Price 56*s*. net

DISINFECTANTS — THEIR VALUES AND USES by W. E. Finch (Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), 1958. Pp. 188. Price 30*s*. net

LIGHT SCATTERING BY SMALL PARTICLES by H. C. Van De Hulst (John Wiley & Sons Inc., New York and Chapman & Hall Ltd., London; *Distributors in India*: Asia Publishing House, Bombay), 1957. Pp. xiii + 470. Price $ 12.00

ERRATA

This *Journal*, **17A**, March 1958, pages 124 and 128: The data given for the books '*Principles of Engineering Inspection*' and '*Strength of Materials*' respectively on these pages are *incorrect*. They should *read* as follows:

PRINCIPLES OF ENGINEERING INSPECTION by Geoffrey K. King & C. T. Butler (Cleaver-Hume Press Ltd., London), 1957. Pp. viii + 264. Price 25*s*.

STRENGTH OF MATERIALS by G. H. Ryder (Cleaver-Hume Press Ltd., London), Second Edition, 1957. Pp. x + 337. Price 22*s*. 6*d*.

NOTES & NEWS

Origin of Life

AN INTERNATIONAL SYMPOSIUM ON the Origin of Life on Earth, organized by the Academy of Sciences, U.S.S.R., under the auspices of the International Union of Biochemistry, was held in Moscow during 19-24 August 1957. The seven sessions of the symposium were devoted to discussions on (1) various ideas concerning the primitive atmosphere, lithosphere and hydrosphere, (2) the formation of amino acids from assumedly primitive gases, (3) the origin of proteins, nucleoproteins and enzymes, (4) relationships between viruses and genes as represented especially by transduction phenomenon, (5) the origin of structure and metabolism, (6) evolution of metabolism, (7) production of amino acids and the evolution of phototrophy.

The origin of proteins, nucleoproteins and enzymes was discussed with special reference to the production of polyglycine. Experiments with polyglycine on kaolinite, to yield serine, threonine and leucine or *iso*leucine residues were discussed. Stress was laid on the similarities of biological reactions and reactions in organic chemistry. Thermal experiments producing amino acids and peptides from the critical dicarboxylic amino acids and yielding the nucleic acid intermediate, ureidosuccinic acid, were reported. Deoxyribonucleic acid preparations have been found to consist of tracts of polypyrimidines and polypurines in groups of three, and the arrangement is regular.

The nature of viruses, genes and life, with special emphasis on relationships between viruses and genes as represented by transduction phenomenon, was discussed. Stress was laid on the central place of the viruses in providing a transition from molecules to organisms. Recent studies have shown that small nucleotides can be good substitutes for nucleic acids and serve as cofactors for the synthesis of adenosine triphosphate.

In the session on the origin of structure and metabolism, it was observed that a single precursor may serve in the production of separate enzymes, which finding is supported by recent work. On the evolution of metabolism, it was reported that aspartic acid is more primitive than glutamic acid. Based on evidence from nutritional data it was proposed that life began in thermal waters. It was pointed out that early short-chain proteins might have been more thermostable than contemporary proteins [*Science*, **127** (1958), 346].

Soviet nuclear research

NEW AND INTERESTING INFORMATION on the interaction of high energy protons with nuclei of emulsions has been obtained as a result of work of the Soviet physicists on the world's most powerful 10,000 MeV. proton synchrotron at the Joint Nuclear Research Institute, Dubna, Moscow Region.

A beam of protons with energies *c.* 9000 MeV. is directed on thick-layer photographic emulsions and several groups of plates are exposed to radiation in this way. Silver bromide, dissolved in gelatine, is capable of continuously tracking the movement of charged particles which tear through the emulsion layers with very high velocity close to that of light.

By tracing the path of protons of 9000 MeV. energy, the Soviet physicists have established the ' interaction run ' of these particles, i.e. the distance a proton travels on the average before clashing with the atomic nuclei of the emulsion. So far no such data have been available as particles could not be accelerated to such enormous energies.

About 500 ' stars ' formed during the interaction of swift protons with atomic nuclei of the emulsion have been studied. A number of physical phenomena characterizing these interactions have been ascertained, in particular, the average number of swift particles formed during the interaction. This number has been found to be much higher than the one established by previous experiments. Some of the ' stars ' are of great scientific interest. They are most probably the result of the full fission of the nuclei of silver during which all nucleons comprising the nucleus evaporate as a result of heating to hundreds of milliards of degrees during the absorption of this enormous amount of energy transmitted by the proton to the nucleus. A number of cases of violent interaction of primary protons with hydrogen nuclei have been observed. In these cases, as distinct from all ordinary ' stars ', a narrow beam of rays arises which consists of relativistic particles, most probably the short-lived mesons. Stars containing up to eight such particles have been found [*Communication from the Information Department, U.S.S.R. Embassy in India, dated* 28 *April* 1958].

Molecular spectra by shock waves

NEW INFORMATION ON THE SPECTROSCOPIC characteristics of the light emitted from air, nitrogen, oxygen, carbon monoxide and carbon dioxide or mixtures of these has been gathered from studies carried out with the shock tube at the Imperial College of Science and Technology, London. The shock tube is particularly useful for the study of spectra of astrophysical interest, because of the attainment of very high temperatures. Knowledge about the spectrum of air is important in any consideration of its high-temperature emissivity which may be required in analysing problems of supersonic flight at high altitude.

Strong reflected shock waves through pure argon gave a spectrum showing argon lines superposed on a continuum with a number of impurity lines, including the Balmer lines of hydrogen, lines of Hg, Na, Ca, Ca^+ and K, and strong CN-bands. Shock excitation of oxygen showed the Schumann-Runge bands of O_2 and with these clearer spectra, the structure of the bands was more evident and extended to 4300A. Nitrogen/argon mixtures showed the Second Positive system of N_2 and the First Negative system of N_2^+. The First Positive system, however, was not observed. A continuum towards the red, and the red and violet system of CN were observed. Relatively mild shocks through argon/air showed the Schumann-Runge bands of O_2 strongly and as the temperature was increased, the N_2 Second Positive, N_2^+, CN-violet and CN-red, all superposed on the Schumann-Runge, were observed between 3000 and 7000A. along with certain other bands. Bands of the β-system of NO were, however, not observed. The yellow-green continuum, due to association between NO and atomic oxygen (air afterglow) was not observed. In laboratory experiments, the CN-red bands in the near infrared probably contribute to the emissivity of air, although they might be absent from clean air. Both CO and CO_2 gave very strong Swan bands of C_2; CH from CO containing H_2 or H_2O was also observed. In very hot shocks, through CO, the Mulliken band of C_2 near 2325A. was observed; the C line at 2478A. was strong and some bands of the Fourth Positive system of CO were visible. Neither the strong Angstrom nor the Third Positive systems of CO were, however, observed. The C_2 high-pressure bands were absent.

The results show that the spectra excited by shock waves are very different from those obtained by electrical discharges through similar mixtures. The Schumann-Runge bands occur in shock waves, but not in ordinary discharges, while the N_2 First Positive and the CO Third Positive and Angstrom bands, which are such strong features of Geissler tube spectra, do not occur. In discharges, excitation is due mainly to electron impact, whereas molecular collisions are probably more important in the thermal excitation in shock waves. Shock tube spectra thus tend to resemble those excited in King furnaces. However, while temperatures in King furnaces are limited to below 3000°C., shock tubes enable attainment of temperatures nearer those of stellar sources [*Nature, Lond.*, **180** (1957), 1342].

Intensification of photographic images

EXPERIMENTAL WORK CONDUCTED at the Polymer Research Institute, Polytechnic Institute of Brooklyn, New York, has shown that the overall production of developable silver in the silver halide grains of a fast photographic emulsion can be increased a millionfold by utilizing the amplification obtainable from chain polymerization of a vinyl compound developer, namely *p*-aminophenol. An intensification of the photographic image can thus be obtained.

Polymerization of vinyl compounds is initiated when silver halide crystals placed in contact with *p*-aminophenol at *p*H 8·0 are irradiated by light. The exposed crystals without developer do not initiate polymerization. A suspension of silver bromide crystals in water, exposed to blue light, was divided into two parts, one of which was developed with a 1/10M solution of *p*-aminophenol hydrochloride buffered at *p*H 8·0 and the other with the same solution plus 20 per cent by weight of calcium acrylate. After 15 min. it was found that the sample with monomer turns intensely black and a large precipitate results; the sample without monomer turns into a murky brown stable suspension.

A photographic plate containing silver bromide crystals in gelatin gel was exposed to feeble light just sufficient to produce an image which was barely perceptible when developed by conventional means. When developed with *p*-aminophenol and monomer, and warming for a short period at 35°C. to allow for mobility of the grains, an intense black image was produced; however, the intensification occurrs at the sacrifice of some resolution [*Nature, Lond.*, **180** (1957), 1275].

New theory of superconductivity

A NEW THEORY WHICH ACCOUNTS for the most important facts of the still incompletely understood phenomenon of superconductivity has been proposed by scientists at the University of California. The theory is based on interactions between electrons and vibrational motions of the atoms of a metal.

The new theory proposed can be explained briefly as follows: Interplay between the motion of electrons and atomic vibrations causes many electrons in a superconductor to be associated in pairs such that the net momentum of each pair is the same. Explanation of this pairing is based on the quantum theory. When there is no current, net momentum of the pairs is zero. Causing current to flow sets these pairs of electrons into motion in such a way that each pair has the same net momentum as every other pair. Electron scattering occurring locally is not enough to change the common momentum of the electron pairs or the total current. The current can change only by the action of some force (like an electric field) that acts on all the electrons at the same time. Without such a force, a current once set up persists. According to the originators of this theory the most important qualitative and quantitative properties of superconductivity are accounted for by it. The theory can be checked quantitatively when the theoretical parameters are determined experimentally. Of these, only one parameter, the critical temperature, involves the superconducting state itself [*Chem. Engng News*, **35** (51) (1957), 30].

Granulation — theory and practice

IN A PAPER ENTITLED *A contribution to the theory and practice of granulation*, presented at a recent meeting of the Institution of Chemical Engineers held in London on 4 March 1958, it was shown that the properties of a granule differ from those of a pendular-bound aggregate in two important respects. Firstly, the maximum cohesive stress within a saturated granular mass, which depends upon the entry-suction, is considerably greater than the cohesion present in the pendular condition; secondly, the full suction in the pore-liquid is not generated until the capillary meniscii are withdrawn into the surface-waists of the granule. Normally, granules appear to have a moist surface and it is only when a load is applied and the granule is strained that the moisture is withdrawn into the surface-waists. When the granule is not under stress, there is little suction in the pores and the resulting reduction in internal friction allows the granule a considerable degree of surface-plasticity. A granule is, therefore, easily formed into a spherical shape by the rolling action in the granulator, and yet offers a considerable resistance to large stresses which tend to disrupt it.

The techniques devised to measure the interfacial areas and bubble sizes were discussed in a paper on *The interfacial area in gas-liquid contacting with mechanical agitation.* Correlations were given for interfacial areas and bubble sizes in terms of power dissipated by the agitator, the gas flow rate to the equipment and the physical properties of the system including the effect of solutes in aerated aqueous solution.

A satisfactory interface control in a simple mixer settler has been obtained without pumps or tilted boxes in a simplified contactor. It is possible to fix the positions of the interfaces in such a box, but complete design is not possible because of the lack of information on the properties of immiscible liquid mixtures [*Chem. & Ind.*, (1958), 358].

A new radio telescope

A NEW HIGH PRECISION RADIO telescope has been built at a site 30 miles from Sydney, Australia. Work on the instrument designed by Dr. Wilbur Christiansen started in 1954 and was completed in July 1957. The instrument is now being used for collecting data for the International Geophysical Year. The telescope is designed specifically to pinpoint and study small areas of the sun and is capable of accurately focussing on an area that represents less than 1 per cent of the sun's surface.

The construction of this new telescope was undertaken because of the inadequacy of the multiple interferometer for dealing with the large number of simultaneous active regions occurring in the sun since 1954 due to the unusual solar activity. The multiple interferometer has established the origin of a few radio and visible phenomena resulting from solar activity; but this type of instrument which has a 'knife-edged' beam performance is ideal only for a period of minimum sunspot activity when there is seldom more than one active region at any time; but, in a period of maximum solar activity, there is no certainty that a recorded peak with this type of interferometer came from that point to which it is focussed.

The telescope is in the form of a cross, intersecting at right angles, each arm being nearly a quarter of a mile long and containing 32 parabolic dishes each 19 ft. in diameter and made of aluminium tubing and lined with chicken wire. Each dish is erected on an equatorial mount; a long projecting pole acts as an aerial. Radio signals from the sun are received by the chicken wire and reflected to the aerials which, in turn, feed the information into a system of wires connected to the recording apparatus.

The instrument can pinpoint a spot instead of scanning along a line. It produces two series of 'knife-edged' beams, each beam only 1/30 degree wide. One series intersects the other at right angles forming an invisible celestial radio lattice. The design of the instrument is such that the connecting lines of the lattice are cut out leaving only the point of intersection; only one point of intersection can, at a time, fall on the sun. As soon as one point scans across the sun, another starts, and so on, each moving a little higher than the preceding one [*Research*, **11** (1958), 117].

Rare earth-iron garnets

A NEW CLASS OF MAGNETIC OXIDES, structurally distinct from the ferrites (sintered mixtures of powdered oxides of magnesium, manganese or iron) has recently been developed. These materials, known as rare earth-iron garnets, are transparent and hence permit the internal magnetic domain structure to be observed through a polarization microscope. The usual magnetic materials and ferrites being opaque to visible light, their internal magnetic structure could only be inferred from reflection of polarized light by their surfaces or the domain boundaries delineated with the help of colloidal magnetic oxides. The phenomenon of ferrimagnetism exhibited by these garnets was first discovered at the Institut Fourier, Grenoble, France, and the Bell Telephone Laboratories, U.S.A. (where the optical and magnetic resonance behaviour is being studied).

While ferrites cannot be made with absolute uniformity, the garnets (single or polycrystals), crystallized from liquid melts at high temperatures, are extremely reproducible in their electrical properties. Electronic-grade garnets are chiefly used in missile systems and serve in such components as waveguides, transmitters, couplers, polarizers and isolators for separating specific energy bands.

Yttrium-iron garnet (YIG), $Y_3Fe_2(FeO_4)_3$, is the most completely studied member of the garnet materials. YIG has three chief advantages over commercial ferrites: (1) low loss in transmitting short-wave energy, (2) low activation power requirements and (3) high reproducibility in resolution of transmitted energy.

The most convenient way to grow YIG's is in a 'solvent melt' of lead oxide. Proper amounts of iron, yttrium and lead oxides are heated to *c.* 1325°C. With slow cooling, crystals begin to form near 1250°C. The matrix is then removed by heating the solidified melt in dilute nitric or acetic acid. Little garnet growth occurs below 980°C. For obtaining magnetic YIG, a typical composition contains 52·5 mol per cent lead oxide, 44 mol per cent ferric oxide and 3·5 mol per cent yttrium oxide. Gallium can be substituted for part of the iron in this formulation. Rare earth oxides can also replace Y_2O_3 in the original composition to vary some of its properties.

YIG has a Curie temperature of 545°K. and a spontaneous magnetization at zero temperature and infinite field of 4·96 Bohr magnetons per molecule (close to the theoretical value of 5·0). This magnetization results from superexchange interactions through the O^{2-} ions between Fe^{3+} ions in crystallographically different positions in the cubic lattice. The number of such interactions per Fe^{3+} ion in YIG is three-fifths of that in a ferrite. Correspondingly the observed Curie temperature is 0·64 of that for magnetite, i.e. $0{\cdot}64 \times 848 = 545$°K.

An interesting and important feature of yttrium-iron garnet is that it contains magnetic ions with but a single valence. X-ray and neutron diffraction studies have shown that, unlike in the ferrites, the interactions between identical magnetic ions wholly occupying two different crystallographic sites are responsible for ferrimagnetism in the garnet structure. In yttrium-iron garnet the width of the ferrimagnetic resonance absorption line at 9300 and 24,000 Mc/s. depends on crystallographic direction. Line widths of only 0·8 oersted were observed along the [100] direction in thin disks at 10,000 Mc/s. when the temperature was held at 540°K. These line widths increased as the temperature decreased and passed through peak values of several hundred oersteds between 65°K. and 4·2°K.

A polarized light beam passing through the transparent magnetic domains in the garnets has its plane of polarization rotated in one direction in one domain but in the opposite direction in an adjacent and oppositely magnetized domain. This Faraday rotation is dependent on wavelength and amounts to several degrees per mol of thickness; it makes the domains within the crystals clearly visible. Thus, for the first time, it is now possible to correlate Faraday rotation in a magnetic material with spectroscopic data over a broad temperature range [*J. Franklin Inst.*, **265** (1958), 144 and *Chem. Engng News*, **35**(49) (1957), 62].

Thermionic converter

A UNIQUE ELECTRONIC DEVICE, developed at the General Electric Research Laboratory, directly converts heat energy into electrical energy with a conversion efficiency of 8 per cent. The efficiency of conventional thermocouples to produce electricity is below 1 per cent. The thermionic converter which eliminates the use of rotating parts takes advantage of the fact that electrons can be 'boiled out' of a hot metal surface and used to produce electric current. The development should be considered a definite advance in research, though commercial exploitation of the converter as a major new source of power production is not yet achieved.

The thermionic converter combines several known scientific principles in a unique manner. Two electrodes within the tube-like device are maintained at high, but different, temperatures. Improvements in the design of the electrodes, materials used and the gas environment within the envelope have resulted in a more efficient flow of electrons than ever achieved before.

An obvious difference between the thermionic converter and the thermocouple is that in the thermionic converter the metals are separated by a gas at very low pressure. There is an electrical flow between the electrodes, but there is less flow of heat than through a metal. Thus the electrodes can be at different temperatures and the efficiency is greatly raised. It is expected that thermionic converters ultimately may be able to change more than 30 per cent of heat energy directly into electricity. The possibilities of developing solid state semiconductor devices for conversion of heat into electricity are also being explored at the General Electric Research Laboratory [*J. Franklin Inst.*, **265** (1958), 279].

Tecnetron

A NEW ELECTRONIC AMPLIFIER (OF the semiconducting type), the 'tecnetron', developed at the National Centre of Telecommunications Study, Paris, is expected to lead to great advances in the fields of television, guided missiles and telecommunications. The device whose manufacture is simpler than that of transistors can operate as an amplifier up to 500 Mc/s.

The device consists of a cylindrical rod of n-type germanium, *c.* 0·5 mm. in diameter and 2 mm. long, in which there is a central hole filled with indium. Of the three electrodes, two are soldered to the two ends and the third to the indium core. The arrangement, in effect, is equivalent to an evacuated pentode. The effective amplification varies with frequency, but optimum performance is obtained in the region 200-500 Mc/s. For industrial applications of the tecnetron, the following figures are quoted: a gain of 22 db. at 110 Mc/s. for a band-pass of 1·7 Mc/s.; 16 db. at 200 Mc/s. for a band-pass of 6 Mc/s. and 9 db. at 430 Mc/s. for a band-pass of 30 Mc/s. [*Research*, **11** (1958), 118].

Germanium resistance thermometer

A GERMANIUM RESISTANCE THERMOMETER of the size of a common pin, having high sensitivity and exceptional stability in the temperature range near absolute zero, has been developed at the Bell Telephone Laboratories, New York. When mounted in a suitable space vehicle, it may be useful for measuring accurately temperatures in the outer space. It does not require recalibration despite repeated cycling from 300°K. to 1°K.

The thermometer consists of a very small bridge ($0{\cdot}025 \times 0{\cdot}020 \times 0{\cdot}210$ in.) cut from a single crystal of arsenic-doped germanium which produces a high and fairly constant temperature coefficient at temperatures near the boiling point of helium. Current and potential leads are attached to the bridge, which is supported, free from strain, in a platinum-glass enclosure containing a small amount of helium gas to facilitate thermal conduction. The resistance is determined by measuring the potential drop when a small (approximately 10 ma.) known current is passed through the bridge. To avoid excessive heating when measurements are being made, the resistance of the thermometer should be kept as large as possible. However, for simplicity in measurements, a low resistance is desirable. For a specific application, a compromise can be reached by controlling the doping of the germanium crystal [*News from Bell Telephone Laboratories*].

Gravity measurements in open sea

THE FIRST SUCCESSFUL SURFACE measurements of gravity in the open sea were made in November 1957 at the Lamont Geological Observatory by the U.S. National Committee as part of the International Geophysical Year programme to chart gravity variations throughout the world. The measurements were begun 200 miles south-east of New York and were continued on a track moving from the deep ocean basin across the continental slope and onto the continental shelf ending off Long Island.

Previously, gravity values for ocean areas had to be measured using pendulums carried in submarines submerged to quiet depths. Till now the difficulty of fitting submarines for this purpose had resulted in the acute paucity of gravity data for 80 per cent of the earth's surface that is covered with water. To date there have been only 4000 measurements of gravity at sea made throughout the world, about half of which have been made by the Lamont Geological Observatory in the past 10 years. In the above unprecedented surface measurements a new sea gravimeter developed by Anton Graf of Munich, Germany, was used. Mounted on a gyro-stabilized platform installed aboard the USS *Compass Island*, the new gravimeter makes it possible to take gravity measurements simply and expeditiously at any place on the ocean surface which was difficult — or even impossible sometimes — till now.

Measurements with this gravimeter could be completed in 9 hr. compared with 2 days for the earlier measurements with submarines. Reduction of the data for analysis took just half a day compared with two weeks needed to compute data from submarine observations.

Gavity measurements, together with seismic measurements, can lead to a determination of the thickness of the earth's crust. Once sufficient values of gravity over a wide enough area of the ocean are available, maps and charts can be greatly improved [*J. Franklin Inst.*, **265** (1958), 166].

Analysis of long-lived radioactive isotopes

A RADIOCHEMICAL METHOD FOR the determination of low concentrations of long-lived radioactive isotopes of Ru, Sr, Cs and Ce formed as byproducts in a nuclear reactor is described. The method is applicable to a wide variety of natural materials, namely soil, water, or biological material, in the presence of impurities commonly occurring in nature. The method is useful in monitoring the drainage basin of the area where radioactive wastes are disposed of. The movement of activity in the disposal area is followed by drilling wells and taking soil samples, by sampling the drainage water, and by taking specimens of vegetation and animals from the surrounding area.

In actual practice, water samples are evaporated to a small volume; organic materials are wet ashed with nitric acid and hydrogen peroxide and soil samples are ground to a fine powder. Suitable quantities of materials, determined by rough counting, are weighed into nickel crucibles, and standardized carrier solution amounting to 20 mg. Sr, Ce and Cs and 10 mg. of ruthenium added. The mix is neutralized with potassium hydroxide and dried under an infrared lamp. A fusion mixture of potassium hydroxide, nitrate and carbonate (2 : 1 : 1 by weight) is added to the crucible to *c.* 5-10 times the weight of the sample. The material is fused in a muffle at 550°C. for 2 hr. with regular shaking. The melt is allowed to cool and is leached twice with 10 ml. of water. Ruthenium and cesium go into solution while strontium and cerium remain in the melt as insoluble carbonate and hydroxide. Ruthenium is extracted into carbon tetrachloride as ruthenium tetroxide and cerium is absorbed in Duotite-3 cation-exchange resin from alkaline solution. Strontium and cerium are extracted from the melt with 6N hydrochloric and nitric acids. Cerium is precipitated as hydroxide and strontium as the carbonate. The precipitates are weighed and counted on 1 in. aluminium dishes, using a β scintillation counter [*Canad. J. Chem.*, **36** (1958), 425)].

Perchlorylation

PERCHLORYLATION, A NEW PROcess for introducing, by electrophilic substitution, a new functional group, $-ClO_3$, in the aromatic ring, employs the reagent perchloryl fluoride. This reaction opens the way to many new organic chemicals — perchloryl aromatic products and their derivatives. Perchlorylation proceeds smoothly, contrary to the powerful oxidizing action of the reagent, and is analogous to a Friedel-Crafts acylation; yields of *c.* 90 per cent are obtained in most cases. A number of perchloryl compounds find possible use as explosives and dyes or as drug intermediates. Perchloryl fluoride is also a useful reagent in one-step fluorination. In the synthesis of fluoro drugs like fluorobarbiturates, fluorosteroids and some anti-cancer drugs such as 5-fluorouracils, it can replace a very toxic fluorinating agent monofluoroacetic acid [*Chem. Engng News*, **36**(8) (1958), 52].

Assay of tocopherol

A SIMPLE CHROMATOGRAPHIC METHOD is reported for the routine assay of tocopherol in pasture samples. Chlorophyll, carotenoids, vitamin A and unsaturated acids do not interfere in the estimation.

The sample is extracted with acetone in the cold. The separation procedure involves double chromatography, first on magnesia to remove chlorophyll and xanthophyll, and then on alumina to remove carotene. A solution of the pigments in 1 per cent ethanol (vol./vol.) in light petroleum ether is poured on to a magnesia column which adsorbs chlorophyll and xanthophyll in a band at the top and allows carotene and tocopherol to move through. The latter are quantitatively recovered in the eluate. The eluate is next dried and taken up in 2·3 ml. of light petroleum for the second chromatography. The solution is poured on to an alumina column which holds tocopherol and allows carotene to pass through, its passage being complete after development with 1 per cent acetone (vol./vol.) in light petroleum. Tocopherol is recovered quantitatively after further development with 3·8 per cent ethanol in light petroleum and is then dried and taken up in ethanol for estimation. Tocopherol is estimated colorimetrically, making use of Emmerie-Engel reaction [*J. Sci. Fd Agric.*, **9** (1958), 122].

Determination of cyclethrin

A COLORIMETRIC METHOD FOR THE determination of the purity of cyclethrin [3 - (2 - cyclopentenyl)-2-methyl-4-oxo-2-cyclopentenyl-chrysanthemummonocarboxylate] and its concentration in insecticide formulations is reported. The method enables the determination of as little as 10 γ of cyclethrin. Cyclethrin, when heated with 85 per cent of *ortho*phosphoric acid for 40 min. at 100°C., produces a red colour which has an absorption maxima from 545 to 550 mμ and the colour intensity obeys Beer's law within the concentration range of 10-90 γ.

Piperonyl butoxide, sulphoxide, etc., which are used as synergists, suppress the colour development by cyclethrin; no colour is produced if piperonyl butoxide is present in the ratio of 10 parts of piperonyl butoxide to 1 part of cyclethrin. Before the estimation of cyclethrin is carried out, the synergists should be separated on a chromatographic column consisting of 2 parts by weight of silicic acid and 1 part of Celite 545. The absorbent materials, before use in the chromatographic column, are mixed thoroughly and activated by heating for 12 hr. at 150°C. The eluting solution consists of a mixture of 10 per cent by volume of ethyl ether in petroleum ether. The synergists remain in the absorbent while cyclethrin is carried in the eluate. The eluate is evaporated to dryness and the colour developed in the usual manner [*J. agric. Fd Chem.*, **5** (1957), 671].

Ascaridole from chenopodium oil

MESSRS GURMIT SINGH, S. S. Chaudhary, K. L. Handa and I. C. Chopra of the Regional Research Laboratory, Jammu, describe in the following communication, dated 25 March 1958, a simple method for the preparation of pure ascaridole from Indian chenopodium oil by low temperature crystallization technique.

Chenopodium ambrosoides var. *anthelminticum*, an exotic from North America, is being successfully cultivated in Jammu. The plant yields an oil (1 per cent by weight) containing 66-70 per cent ascaridole.

A sample of the oil (ascaridole content, *c.* 60 per cent), when fractionated at 4 mm. of mercury, yields a major fraction containing *c.* 88 per cent ascaridole. Repeated fractionation did not enhance the purity of the product. Attempts were, therefore, made to separate ascaridole from the oil in a crystalline form by low temperature crystallization.

The oil was dissolved in twice its volume of light petroleum ether (b.p. range 40°-60°C.) and chilled to −35°C. with solid carbon dioxide, with occasional shaking of the mixture. The mixture was maintained at −35°C. for 15 min. and filtered. Ascaridole which separated out in a crystalline form was washed with cold petroleum ether. The filtrate and washings were collected, concentrated and the concentrate chilled to −45°C. when a second crop of crystalline ascaridole was obtained. The total yield of ascaridole was *c.* 70 per cent of that present in the oil. From the mother liquor, petroleum ether and hydrocarbons were removed by distillation under reduced pressure when a residual oil containing *c.* 70 per cent ascaridole was obtained which can be used as such in the treatment of anthelmintic infections.

Anaesthetics — structure and activity

THE INFLUENCE OF VARIOUS CHEmical groups on the activity of anaesthetics has been reviewed.

Methane acts as an anaesthetic at concentrations a little below the toxic levels, but increased molecular weight enhances both narcotic power and toxicity, the first factor outstepping the second and becoming maximal with heptane. The corresponding unsaturated hydrocarbons, probably on account of their greater powers of penetration into nervous tissue, are more potent.

Olefine chains exceeding three carbon atoms tend to stimulate before they depress. Cycloalkanes increase in narcotic and toxic potency with higher molecular weight. Alkyl side-chains confer high toxicity, ethyl substituents proving more toxic than methyl substituents. Aromatic hydrocarbons are far too toxic to have any anaesthetic interest.

Halogenation of an aliphatic hydrocarbon markedly increases its anaesthetic potency and the greater the number of halogen atoms the more potent the drug — except in respect of fluorine compounds. Chloro compounds are much more active than bromo, iodo or fluoro compounds. Narcotic properties are retained even after the total substitution of hydrogen by chlorine, as in hexachloroethane.

The pattern of toxicity, however, is more intricate than these wide rules would suggest. Ethyl chloride and methyl chloride have been used in anaesthesia, but are irritant and too volatile. Ethyl bromide readily depresses respiration at anaesthetic levels, while methyl bromide and iodide are neurotoxic. Monochloro-monobromo-methane, however, is quite potent as an anaesthetic and comparatively mildly toxic. Among the unsaturated compounds, vinyl chloride is not very potent, and allyl chloride is a violent irritant of mucous membranes.

Dichloro-hydrocarbons are narcotic but very toxic. Trichloro compounds offer a promising range of anaesthetic agents, for they are comparatively less toxic while their narcotic power is marked. Chloroform is still the most powerful inhalation-anaesthetic available although it has a tendency to cause cardiac irregularities. Trichloroethylene, which has a rapid onset of action, is less potent and less toxic than chloroform.

Introducing fluorine atoms into the chloroform structure progressively weakens both the potency and the toxicity of the drug. The corresponding trichloroethane derivatives include one notable though expensive drug, halothane (2-bromo-2-chloro-1, 1, 1-trifluoroethane), twice as potent as chloroform. The replacement of all but one hydrogen atom in this drug renders it non-flammable, while the high fluorine content effectively reduces its toxicity. Tetra-halogenated compounds are powerful narcotics, but have marked toxic effects upon the liver; tetrachloroethylene is not useful as an anaesthetic drug. All aromatic halogenated hydrocarbons are highly toxic and almost devoid of narcotic activity.

Alcohols are generally nervous depressants but are not useful as anaesthetics. Halogenation increases the potency of alcohols. Tribromoethanol (bromethol) has been used only as a basal anaesthetic in high dilution; its effect is not easily controllable, and full doses cause serious respiratory and circulatory depression. Aromatic alcohols when injected into tissue exert local anaesthesia. Phenylethanol is more effective than benzyl alcohol, though both compounds are approximately equitoxic.

Ethers, with the general structure R—O—R′, provide many excellent, though highly flammable and explosive, inhalation anaesthetics. With increase of molecular weight, narcosis and toxicity increase, but volatility is soon lost. As the series ascend, the safety margin between effective and toxic doses narrows. Ethers derived from dihydric and trihydric alcohols are not volatile enough to be useful.

Unsaturated aliphatic ethers are more powerful than the saturated ethers but only divinyl and ethyl vinyl ethers possess low toxicity. *Iso*propyl vinyl ether is relatively safe; the introduction of an alkyl group renders these ethers very irritant. As with the chlorinated compounds, toxicity and flammability of ethers may be lowered by the introduction of fluorine atoms. One such compound is 1,1, 1-trifluoroethylvinyl ether, which is as potent as diethyl ether and has low toxicity. Some of the aromatic ethers, particularly hydroxylated ones like eugenol, are mild surface anaesthetics.

Some barbiturates have been used effectively as anaesthetics by the intravenous route. The general requirements of barbiturate formulation are that the groups at position 5 should total 4-8 carbons, and that each of these groups should have more than 2 carbon atoms; there are, however, some exceptions.

One new intravenous anaesthetic, hydroxydione (21-hydroxypregnane-3, 20-dione), is a solid steroid, without any of the undesirable side-effects associated with steroids in general.

Cocaine, yohimbine, papaverine, quinidine and other alkaloids are powerful local anaesthetics but are toxic. The most successful synthetics are the *p*-aminobenzoates. Esters, like ethyl and butyl *p*-aminobenzoates, used as anaesthetics are undesirable because of their sensitizing activity. Halogenation of the aromatic nucleus lengthens the duration of anaesthesia. Some of these derivatives, such as chloroprocaine (diethylaminoethyl-2-chloro-4-aminobenzoate), are less toxic than their parent. Hydroxylation of the nucleus produces more active variants, but alkylation of this hydroxylic group increases the toxicity; one such compound, ravocaine (2-diethylaminoethyl-4-aminopropoxylate), has been used clinically. In this series of drugs the greatest anaesthetic potency (and incidentally sensitizing power) accompanies an amino group in the *para* position of the nucleus. Alkylation of this group increases both the potency and the toxicity, in the order methyl to butyl; higher alkyls reduce them again. Lignocaine (α-diethylamino-2, 6-acetoxylidide) is an exception by the presence of an amide linkage within the nucleus and no *para* substituent. Alkylation (dimethyl in this case) is necessary for clinical potency. Hexylcaine (1-cyclohexyl-amino-2-propylbenzoate) dispenses with the aromatic amine altogether. Both compounds, though more toxic than procaine, are more potent. They are effective on mucous surfaces, which gives them a clinical advantage over procaine [*Chem. Age*, **79** (1958), 505].

Some new drugs

THE PROPERTIES OF SOME RECENT analgesics, anti-epileptics, antihistamines, diuretics, sex steroids, anti-tubercular and anti-leukaemic drugs, etc., have been reviewed [*Amer. J. Pharm.*, **130**(1) (1958), 4].

New analgesics reported are α-d-4 dimethylamino-1, 2-diphenyl-3-methyl-2-propionoxybutane hydrochloride (Darvon), 1-methyl-4-carbethoxy-4-phenyl hexamethylenimine citrate (Ethoheptazine) and 4-aminophenmethyl derivative of meperidine (Anileridine phosphate). Darvon has the same analgesic potency as codeine, is non-narcotic and does not produce euphoria. Ethoheptazine is structurally related to meperidine except that the piperidine ring in meperidine is replaced by a seven-membered nitrogen containing saturated ring. Its action is superior to that of codeine and it is non-addicting. Anileridine phosphate is more potent than meperidine and is useful as a pre-anaesthetic drug.

Most of the ataractic drugs are phenothiazine derivatives. A new ataractic belonging to the phenothiazine group is 10-(3-dimethylamino-propyl)-2-trifluoromethylphenothiazine hydrochloride (Vesprin). It is twice as potent as chlorpromazine. A tranquillizer of somewhat broader application is 2-chloro-10-[3-methyl-4-piperazinylpropyl]-phenothiazine (Compazine). The drug is useful in controlling nausea and vomiting from such causes as gastroenteritis, radiation therapy, cancer and pregnancy. Mepazine, 10-(N-methyl-3-piperidyl methyl) phenothiazine, is useful not only in severe mental diseases but also as a general tranquillizer to potentiate analgesics, as pre-anaesthetic medication in nausea, etc. It has a selective action on the central nervous system without inhibiting the cortex and causing hypnosis.

The latest diuretic, ideally suited for use in the control of edema, is 6-chloro-7-sulphamyl-1, 2, 4-benzothiadiazine-1, 1-dioxide (Diuril). It is a carbonic anhydrase inhibitor and enhances the secretion of both sodium and chloride by the kidney tubules.

Water-soluble adrenal steroid derivatives (prednisolone 21-phosphate) for use in ophthalmic and dermatologic products are described. The solubility of the prednisolone compound is about 2000 times that of the parent compound which should provide better penetration into inflamed tissue. The improvement in the clinical usefulness of cortisone has been obtained by introducing a double bond in the Δ' position (prednisone). A new steroid recently introduced is 6-methyl-Δ'-hydrocortisone (Medrol). It is more effective than prednisolone, almost completely eliminating the hazard of edema. The potency of adrenal steroids is enhanced by the introduction of a methyl group at position 2. 2-Methyl-hydrocortisone is much more potent in both its glucocorticoid and minerocorticoid activity than hydrocortisone. A number of related steroids having an anabolic but with minimal androgenic activity have been developed. The steroid is 17-α-ethyl-17-hydroxynorandrostenone. It is used to create positive nitrogen balance in recovery from prolonged illness, in wasting diseases, in premature infants, and malnutrition. It should not be given in prostatic carcinoma and should be administered carefully where there is evidence of liver damage.

A new antibiotic substance, ristocetin, composed of ristocetin A and B, has been isolated from a new species of Actinomycetes, *Nocardia lurida*. Both ristocetin A and B are large molecules with a molecular weight of about 4000. Clinically, it has proved life-saving in many cases where patients were dying with an infection resistant to all other therapies. Ristocetin unlike many antibiotics is bactericidal rather than bacteriostatic. It cannot be used orally or by other parental routes since, in contact with tissue, it is irritating.

A new potent tuberculostatic drug reported is pyrazinoic acid amide. It is intended for the short-term protection of patients who are seriously ill and when other drugs are ineffective. The drug, along with isoniazid, is considered to be a potent tuberculostatic drug combination.

A new potent anti-leukaemia drug related to nitrogen mustards is *p*-(di-2-chlorethyl) aminophenylbutyric acid (Leukeran). It is indicated in the treatment of chronic lymphocytic leukaemia, malignant lymphomas, and Hodgkin's disease.

A new and interesting approach to the therapy of fungal infections of the skin is afforded by the drug Enzactic Cream containing 25 per cent glyceryl triacetate. Its mechanism of action depends on its hydrolysis under the influence of esterases found on the skin and elaborated by fungi and other organisms. On hydrolysis, free acetic acid is formed which acts both to restore the *p*H of the skin to normal and also retard the growth of the fungi. This mechanism is self-regulating since the liberated acid also retards esterase activity until the *p*H again rises when more glyceryl triacetate is hydrolysed and more acid formed.

A number of unsaturated fats in the treatment of hypercholesterolemia and, thus atherosclerosis, have been reported. Safflower oil, a rich source of unsaturated fatty acids and pyridoxine, is now being marketed as an emulsion under the name Linodoxine.

High-purity thorium

A NEW PROCESS HAS BEEN DEVELOPED for the manufacture of high-purity thorium by a U.S. firm under contract with the U.S. Atomic Energy Commission. Metallic thorium made by the Ames process, which was employed hitherto, is not of very high purity; the production costs are $ 15-20 per lb. The new method developed is an electrolytic process which has enabled the production of high-purity thorium required for nuclear research and other purposes, the production costs being only $ 2 per lb.

The process consists in precipitating thorium oxycarbonate from a solution of the nitrate by the addition of sodium carbonate. Certain impurities are eliminated during this precipitation by an electrolytic method. The oxycarbonate is ignited to the oxide which is then mixed with finely divided carbon black and treated with chlorine at a temperature lying between 1350° and 1400°F. The tetrachloride (70 parts) is mixed with sodium chloride (30 parts) and this mixture is used as the electrolyte. The molten salt mixture is placed in a cell lined with graphite [*Chem. Tr. J.*, **142** (1958), 17].

Treatment of gas effluents

THE EFFICIENT TREATMENT OF GAS effluents and methods to reduce atmospheric pollution were discussed in a paper *On the high efficiency fibre filters for the treatment of fine mists*, presented at a meeting of the Institution of Chemical Engineers, held in London on 4 March 1958. The treatment of plant tail gases to remove completely sulphuric acid mist with particles of less than 2 μ has been studied. It has been found that if a filter of fine glass-wool is treated with a silicone to provide a water-repellent surface, the acidity of the gases leaving the filter is reduced to 0·2 per cent of the inlet acidity as compared with 2·5 per cent for the untreated wool under similar conditions of operation. This twelve-fold improvement of scrubbing efficiency was important since those gases scrubbed with the silicone-treated fibre were invisible when vented to atmosphere, whereas a visible plume persisted after scrubbing with the untreated fibre. A similar though not quite so outstanding result was obtained with garnetted terylene polyester fibre (which is water repellent) when a scrubbing efficiency of 99·6 per cent was obtained [*Chem. & Ind.*, (1958), 358].

Journal of the All-India Institute of Mental Health

THE ALL-INDIA INSTITUTE OF Mental Health has commenced the publication of a new bi-annual periodical entitled *Journal of the All-India Institute of Mental Health* (Sanskrit title: *PRATIBHA*).

The need for a medium for the dissemination of the results of the researches carried out in the Institute and elsewhere was being felt ever since the establishment of the Institute in 1954 for postgraduate training of psychiatrists, psychologists, psychiatric nurses and social workers. A journal specially devoted to publicize the work carried out in these fields was the logical solution. The work of the Institute has been rapidly expanding and the appearance of *Pratibha* is most opportune. The frequency of the journal will have to be increased not only for accommodating the increasing research output but also to see that research papers are expeditiously published.

The first issue of the journal (Vol. 1, No. 1 — January 1958, pp. 1-92), in addition to a brief article on the Mysore Government Mental Hospital and the Institute at Bangalore, contains ten technical articles. A section is devoted to Clinical and Research Notes and two others to Book Reviews and Abstracts respectively.

The journal is neatly printed. The annual subscription is Rs. 12 or £ 1.

Announcements

- *Bombay Natural History Society* — The following have been nominated office-bearers of the Society for the year 1958-59: Shri Sri Prakasa (*President*); Shri Sahib Singh Sokhey, Rev. Fr. H. Santapau, and Shri Salim Ali (*Vice-Presidents*); and Shri Humayun Abdulali (*Hon. Secretary*).

- *Award of Doctorate Degrees* — The following have been awarded the Ph.D. degree of the Poona University for the theses noted in brackets against their names: Shri Ajit Kumar Das Gupta (*Diffusion and evaporation of trace impurities from the matrix of a host lattice*); Shri Kuku Daulatram Karamchandani (*Chemistry of preparation of peptones*); Shri Vishwaniyant Gopal Naik (*Synthesis of nitroflavonols*); and Shri Kashinath Ganesh Gore (*Experiments in benzopyrones*).

INSTRUMENTS AND APPLIANCES

MICROFLUOROSCOPE

An X-ray microfluoroscope for observing and measuring directly the growth in living cells has been developed at Stanford University's X-ray Research Laboratory. Artificial staining is not necessary with the microfluoroscope and a specimen can be mounted for viewing in ordinary air. Analysis of specimens weighing as little as one-trillionth of a gramme is possible.

The microfluoroscope is similar to the fluoroscope used in medicine and promises to make possible quantitative X-ray absorption measurements of mass, thickness and content of tiny structures [*Sci. Newslett., Wash.*, **73** (1958), 119].

MICROSPECTROPHOTOMETER WITH ULTRAVIOLET MICROSCOPE

Messrs Neutronics Research Company, Waltham, Mass., U.S.A., have announced the first commercial microspectrophotometer with ultraviolet microscope.

A single UV-visible source supplies a double-beam monochromator adjustable from 2300A. to 6000A. Via time-sharing the specimen in the microscope is irradiated in such a way that reliable pilot-channel automatic calibration can be maintained. The transmission through the photomultiplier and amplifying system controls a recorder, linked to the monochromator, which records transmission or absorption for areas as small as one square micron at the flip of a switch. Thus, the recorder automatically plots transmission or absorption as a function of wavelength [*J. Franklin Inst.*, **265** (1958), 278].

INVERTED-MAGNETRON GAUGE

A cold-cathode ionization gauge with axial magnetic field and radial electric field has been designed, which has proved useful in the range of pressures from 10^{-3} to about 10^{-12} mm. Hg; it has a sensitivity of about 1 amp./mm. Hg. The gauge has the structure of an inverted magnetron with an auxiliary cathode which provides the initial field emission and allows the positive-ion current to be measured independently of the field-emission current.

The gauge has been designed to overcome a number of defects which made the earlier types unsuitable for the measurement of extremely low pressure, e.g. most designs of the Penning gauge fail to strike at pressures below *c.* 10^{-5} mm. Hg; the ion-collecting electrode is exposed to high electric fields and the resultant field emission from this electrode is indistinguishable in the measuring circuit from the positive-ion current.

The ion collector in the instrument is a close-ended cylinder with holes in the centre of the end plates through which the anode and the shielding tubes pass. The auxiliary cathode is a box-like structure enclosing the ion collector and acting as an electrostatic shield. Welded through the sides of the box are two short tubular shields which project into the ion collector and through which the anode passes. The shield cylinders project 2 mm. inside the ion collector and protect the end plates of the ion collector from exposure to high electric fields; they also provide the field emission current which initiates the discharge [*Canad. J. Phys.*, **36** (1958), 271].

General Purpose Counters (Type 1339A)

General purpose counting equipment type 1339A has been designed and developed by the Airmec Ltd., High Wycombe, Bucks, England, in conjunction with the U.K. Atomic Energy Research Establishment to provide comprehensive scaling facilities for Geiger and scintillation counters used for the accurate assay of alpha, beta and gamma active samples. The equipment is of interest to those engaged in atomic research and development and to those who apply the end products of nuclear physics in hospitals and industry.

Subminiature Electrolytic Capacitors

The new Cornell-Dubilier type EC aluminium-foil electrolytic capacitors are specifically designed for transistor and 'low-B' tube applications where compactness is an important design factor. The smallest case size is 0·187 in. in diameter and ½ in. long, and the largest size, only 0·375 × 1½ in.

Type EC capacitors are available in ratings from 3 to 75 V. d.c. working, and in capacitance from 1·0 to 250 mfd, depending on voltage ratings. The range of operating temperature is −20°C. to 65°C. The capacitors are housed in special tubular ceramic cases. The capacitor sections are sealed with a special cast resin, making them moisture-tight and heat-resistant. Tinned copper leads are welded to the anode and cathode tabs to ensure low-resistance contact. Leads will not pull loose. Stable capacitance, low dissipation factor and low d.c. leakage current assure dependable performance under all normal operating conditions [*J. Franklin Inst.*, **265** (1958), 278].

Automatic Sulphur Dioxide Recorder

An automatic instrument which continuously records sulphur dioxide concentrations in the range 0-0·5 p.p.m. with a sensitivity of 0·01 p.p.m. has been developed. The instrument has been designed especially for measuring atmospheric pollution and is suitable for long periods of continuous operation on a fixed site.

The sulphur dioxide in air is absorbed by a hydrogen peroxide reagent in a continuous countercurrent absorption column. This results in the formation of sulphuric acid in the reagent solution and the consequent increase in its conductivity is measured. The important component of the recorder is the conductivity cell which should have high sensitivity. The platinum electrodes of the cell are made in the form of two concentric cylinders in a glass tube so that the liquid flows through the annular space between the cylinders. The shape of the cell is such that the liquid in it is swept away without dilution by the incoming liquid. To eliminate temperature variations, the absorption column, reagent feed lines and conductivity cells are enclosed in a thermostatically controlled heated compartment.

The instrument was calibrated with mixtures of sulphur dioxide and air over the range 0-0·5 p.p.m.

Portable sulphur dioxide meter — A portable meter for determining the sulphur dioxide content of air has also been developed; the instrument can be used in a small car for carrying rapid surveys in a locality.

The principle employed in the instrument is based on the reaction between sulphur dioxide in the air and starch iodine reagent in a countercurrent absorption column. The amount of light absorbed by the unchanged and the partially decolourized reagent are compared by photoelectric cells connected to a galvanometer.

The degree of decolourization of the reagent depends on the ratio of air and reagent flow rates. For comparative measurements this ratio must be constant and the flow rates are adjusted by means of rotameters [*J. Inst. Fuel*, **30** (1957), 628].

Chloride Ion Analyser

An instrument whose chief component is an improved silver-silver chloride electrode has been developed for continuous measurement and detection of chloride ions in aqueous solutions. The instrument is expected to be useful in chemical process industries for measuring the salt contents of processed foods, for detecting leaks in heat exchangers where sea water or salt-containing water is used for cooling, for monitoring industrial wastes and in water treatment where chloride ion control is important.

The silver-silver chloride electrode is made of a mixture of the metal and its corresponding chloride salt, formed under high pressure. The billet is structurally sound and provides a large volume and area of metal-metallic salt which gives long service under stream conditions.

In operation, the electrode and a reference electrode are immersed on the process stream. The silver-silver chloride electrode measures the potential developed between the silver metal and its ions. The concentration of silver ions, in turn, depends on the concentration of chloride ions in the stream. The electrodes develop a millivolt potential which is fed to the analyser. The analyser meter then gives a reading direct in p.p.m.

The instrument is not suitable for measuring chloride ion concentrations in solutions containing strong oxidizing or reducing agents or substances which increase the solubility of silver chloride by forming complex ions [*Chem. Engng News*, **36**(2) (1958), 50].

IMPACT LEATHER TESTER

A machine for evaluating the strength, stretch or crackiness of leathers, developed at the National Bureau of Standards, Washington, U.S.A., is described. The physical properties of the material can be determined from the data on grain cracking and elongation obtained during the breaking-down process. The equipment is valuable for comparing various tannages and leather lubricants, and is useful for detecting deterioration caused by various kinds of exposure. The equipment consists of two hemispherical tipped plungers attached to reciprocal shafts and supported horizontally in the same axial plane on a 2×4 ft. stand. The leather specimen is held in an upright position between the plungers by serrated steel plates provided with central apertures 1½ in. in diameter. Identical cams driven in opposite directions of reaction by an electric motor alternately activate the plungers. Force is applied to the specimen by 27 lb. weights attached through openings in the stand to the two plunger assemblies. During a test, the plungers break down the leather specimen by striking the two sides alternately at the rate of 44 impacts a minute.

A comparative study of the durability, as judged by the impact tester, of a typical upper leather of a shoe showed that the resistance to breakdown is greatest for straight chrome-tanned leather containing stuffing mixture, and lowest for chrome-vegetable tanned leather containing grease. Straight chrome-tanned leather containing fat-liquor is less resistant to impact breakdown than chrome-tanned leather containing stuffing mixture [*Tech. News Bull., U.S. Bur. Stand.*, **42** (1958), 52].

INSTRUMENTS FOR MEASURING HUMIDITY AND EVAPORATION

Two simple devices, useful in field work, for determining the humidity of air and the rate of evaporation from a surface, have been developed at the National Mechanical Engineering Research Institute of the Council for Scientific & Industrial Research, Pretoria, Union of South Africa.

One of them is based on the fact that the rate of evaporation of water and, therefore, its cooling effect on the wet bulb depends on the amount of water vapour already in the air. The principal feature of the new instrument is a pair of concentric tubes with an intervening vacuum. The outer surface of the outer tube is silvered to prevent loss of heat. Air is drawn slowly through the inner tube which is packed with a porous medium kept moist. The air passing through the tube becomes saturated with water vapour and reaches a temperature (the so-called adiabatic saturation temperature) dependent solely on the atmospheric temperature and the amount of moisture originally present in the air. Humidity can thus be calculated without the use of calibration curves or empirical tables.

The other instrument developed is for the direct determination of evaporation from the human body. This involves the measurement of minute changes in the humidity of a stream of air in which the body is placed. The instrument uses the property that the electrical resistance of a crystal of pure salt changes sharply when the humidity of the air reaches a point at which the pressure of the water vapour it contains corresponds to the vapour pressure at the surface of the salt at a particular temperature. This equilibrium position is extremely critical and can be determined with the same degree of accuracy as temperature and resistance measurements. Trials on experimental models resembling to scale the evaporation from a human being gave values which compared favourably with the evaporation values determined by other means. The instrument finds practical application in research into the physiology of human beings who have to work under difficult environmental conditions such as in mines and factories [*C.S.I.R. Res. Rev.*, **7** (1957), 122].

GALLENKAMP SEDIMENTATION BALANCE

The basic principle employed in the sedimentation balance manufactured by A. Gallenkamp & Co. Ltd., London, is that powders uniformly dispersed in a suitable liquid are allowed to settle from a cylindrical column on to the pan of a special torsion balance. The pan is always suspended in clear liquid and the weight recorded is not affected by changes in the density of the suspension. This is an important feature of the design because serious errors may occur if the balance pan were surrounded by dispersed particles of unknown and continuously changing concentration.

The instrument has a range of 5-75 μ but particles down to 2 μ can be measured if the top size does not exceed 20 μ. Its maximum sensitivity is full scale deflection with 0·5 g. of sample of sp. gr. 1·5 in liquid of sp. gr. 1·0.

A jacketed settling column and sample introduction system are mounted on a very rigid stand with a robust height-adjusting mechanism. The bottom of the column is immersed in a receiving tank, and a torsion balance pan fits closely over the end of the column. The balance is built into a metal housing and is read on an optically projected scale.

Dispersed samples are introduced easily, and readings are taken at intervals which increase by a factor of $\sqrt{2}$ in order to plot an accurate weight/time curve. So far automatic recording has not been applied because plotting on a linear time basis would either lead to considerable inaccuracy in calculating the distribution of the larger particles or require a very long chart.

Progress Reports

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, INDIA

THE ANNUAL REPORT OF THE COUNCIL OF SCIENTIFIC & Industrial Research, New Delhi, for the year 1956-57 records considerable expansion in its activities. Eighteen National Laboratories were functioning during the year under report. Of these, the Indian Institute for Biochemistry and Experimental Medicine, Calcutta, was taken over by the Council on 1 April 1956; the Central Laboratories for Scientific and Industrial Research, Hyderabad, were taken over on 13 April 1956 and renamed Regional Research Laboratory, Hyderabad; the Central Mining Research Station, Dhanbad, and the Birla Industrial and Technological Museum, Calcutta, were in the planning stage; the building of the Central Electronics Engineering Research Institute, Pilani, was completed in October 1956. The Regional Coal Survey Station at Hyderabad started functioning during the year.

The number of research projects sponsored by the Council and in operation during the year was 282. The investigations covered a wide range of subjects, such as radio physics, instrumentation, windpower technology, essential and vegetable oils, dyestuffs, pharmaceuticals and drugs, plastics and cellulose products. Two research units, namely the Rain and Cloud Physics Research Unit, New Delhi and the Ionospheric Field Station, Haringhata, continued to function during the year.

Sixty-four applications for patents were filed during the year 1956-57. The number of patents accepted was 47 and the number of patents sealed was 25.

Work on the development of stainless steels at the National Metallurgical Laboratory has resulted in the production of a stainless steel in which a part of the nickel is replaced by much cheaper and readily available manganese. The melting is carried out in a high frequency furnace and the raw materials used are Armo iron, mild steel scrap, low carbon ferro-chrome, low carbon ferro-manganese, electrolytic manganese, copper, ferro-silicon and misch metal. Nitrogen is introduced by blowing the gas on the melt, by the addition of calcium cyanamide and by the use of high nitrogen, low-carbon ferro-chrome.

Studies on the production of refractories have shown that forsterite and sillimanite, which are abundantly available in India, can be profitably utilized as raw materials for the manufacture of refractories. Super-duty hot-face insulation materials have been produced from indigenous raw materials.

The Regional Research Laboratory, Hyderabad, has developed two processes for the production of high grade active carbon and a pilot plant for its production has been set up. Two processes, namely production of manganese sulphate from low-grade manganese ores and of cuprous oxide from copper scrap, have been leased out for commercial development.

A process for recovering ceria of 99 per cent purity from mixed chlorides of rare earths accumulating as a byproduct in the Rare Earths Factory at Alwaye has been developed by the National Chemical Laboratory. An anion-exchange method developed at the laboratory has proved effective for the separation of zirconium from hafnium. Zirconia of spectroscopic purity, required for atomic energy work, has been obtained by the above process. A new process for the biosynthesis of labelled formate has been developed, which involves the reduction of $C^{14}O_2$ by hydrogen in the presence of *Escherichia coli* grown on formate.

The survey of the coal resources of the country, which is being pursued by the Central Fuel Research Institute, has led to the discovery of large reserves of coal for coking and blending in Jharia coalfields, Jhilinito coalfields in Madhya Pradesh and Raniganj coalfields.

A résumé of some of the other important researches carried out in the National Laboratories and under the sponsored research projects is given below.

Physics — The influence of polarization fields on restrahlen frequencies of alkali halides has been studied. It was found that a small displacement of the lattice of negative ions in the crystal produces an electric polarization in the medium which, when the displacement is sufficiently small, may be regarded as produced by point dipoles situated at the lattice points.

Zinc deposited on stearic acid crystals revealed growth spirals and interlaced spirals; this shows that two forms of stearic acid crystals exist. The technique has been extended to the study of the surface texture of crystals of substituted long chain compounds. A new superstructure of nickel oxide has been discovered; it is a metal-excess spinel.

An improved thermal expansion apparatus has been constructed to carry out measurements of the coefficient of expansion of various metals at low temperatures. A spectrograph for measuring the size of rain drops has been constructed and is based on the consideration that light scattered by rain drops depends on the size of the drops. A new instrument for recording heat radiations from the atmosphere during day time, an electronic integrating solariograph and a micro radiation balance meter and some accessories for the Beckmann infrared spectrophotometer have been designed.

A new method which eliminates the necessity of measuring densities of solutions has been worked out for the determination of dipole moment and relaxation time of the solutions from the measured dielectric constant and loss factor values at microwave frequencies.

An improved four-station method, in which a fourth aerial is introduced in east-west direction such that the four aerials occupy the corners of two adjacent and identical right-angled triangles, has been developed for measuring ionospheric wind drifts.

A technique for estimating the intensity of vertical currents in and around a convective cloud — an important basic parameter in determining the final sizes attained by rain drops and hence the intensity of rainfall — has been developed. The method is based on telemetering pressure levels reached during successive minutes by a liftless or balanced balloon after it gets detached at some predetermined height from the carrier balloon by means of a fine fuse device.

Chemistry — A paper chromatographic method has been developed for the separation of chromium and titanium in which thioglycollic acid and ammonium benzoate are used as the reagents. The chromium complex gets eluted leaving behind titanium which may be detected by the usual chromotropic acid method. A new microgravimetric method for estimating germanium, silicon and phosphorus as acridine heteropolymolybdates has been worked out. New spectrophotometric methods have been developed for the estimation of titanium with phenylfluorone and for uranium and molybdenum with pyrocatechol disulphonic acid.

A mixture of phosphorus oxychloride and anhydrous zinc chloride has been shown to be a powerful condensing agent in organic reactions and a number of substituted aryl allyl ketones, deoxybenzoins, phthaleins, chalkones, chromonones and sulphonic esters have been obtained directly from the acids in high yields using the new condensing agent. Among the industrially important synthetic drug intermediates prepared are hexyl resorcinol, *p*-methoxy propiophenone, *p*-hydroxypropiophenone and deoxyanisoin.

Munetone, isolated from the root bark of *Mundulea suberosa* Benth, on the basis of exhaustive degradation experiments, has been assigned the structure 2-methoxy-7:8-(2 *iso*propyl 4:5-furano) *iso*flavone. Stigmasterol and sitosterol have been isolated in sufficient quantity and attempts are being made to utilize them for the preparation of steroidal hormones.

Biochemistry — A tentative method has been developed for the estimation of nicotine in the presence of allied alkaloids; the method is based on the separation of the alkaloids by paper chromatography, elution with dilute hydrochloric acid and spectrophotometric estimation at 259 mμ. A new androgenic compound, 19-nortestosterone (19-NT), with low virilizing properties, has been found to delay conception in rats probably by deferring ovulation beyond the normal time. The drug has marked uterotrophic properties in immature rats, but has little effect on ovogenesis. Two new biosynthetic penicillins from *Penicillium chrysogenum* have been prepared by incorporating long chain fatty acids from chaulmoogra and hydnocarpus oils into the penicillin molecule. A new species of streptomyces, *Streptomyces caiusiae* sp.n., has been isolated from a soil sample from Madras. The active principle elaborated by the organism has been found to possess antibacterial and antifungal activities.

An economic method for the isolation of diosgenin from *Dioscorea prazeri* Wall has been perfected and a patent for the same has been filed. Diosgenin is a useful starting material for the preparation of steroid hormones. Progesterone has been synthesized using diosgenin.

Metallurgy — An inexpensive and simple electrolytic process for the preparation of beryllium oxide from beryl ores has been developed. Pure manganese dioxide, suitable for use as a depolarizing material in dry cells, has been obtained by electrolysing manganese sulphate between an antimony-lead anode and a stainless steel cathode. A process for electroplating on non-conducting materials, like glass, wood and plastics, has been developed. The process consists in producing a conducting metallic film on prepared and pretreated non-conducting surface by chemical reduction. A hot-dip aluminizing process for coating steel, involving the use of potassium fluoride as an aqueous flux, has been developed.

Systematic investigations have been undertaken on the utilization of low-grade manganese ores for the production of electrolytic manganese and manganese dioxide. The methods developed include: (i) regeneration of spent liquor from electrolytic cell with low-grade manganese ores; (ii) electrolysis of regenerated liquor or feed liquor to produce manganese metal and (iii) electrolysis of regenerated liquor or feed liquor to produce manganese dioxide.

A rapid method has been developed by which the alternating load on a specimen can be steadily increased during the testing of plain carbon structural steels for fatigue resistance. A method for the estimation of titanium in cast iron, steel, red mud and ferro-titania, using tannic acid and cinchonine, has been developed.

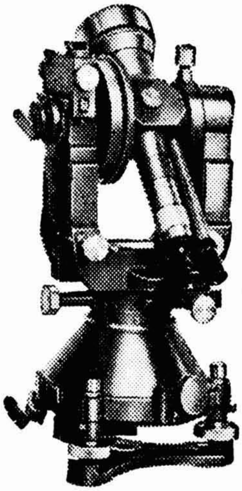
Fuels — Studies on coal carbonization have shown that weathering is responsible for the lowering of volatile matter, carbon, hydrogen, calorific value and tar yield of coals; the lowering is inversely proportional to the rank of coal.

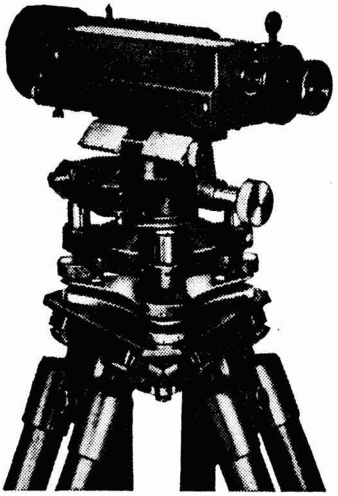
A 4 in. diameter, low temperature, fluidized carbonization pilot plant, with a capacity of 300 lb./sq. ft./hr. coal, feed, which can be operated both batchwise and intermittently, has been installed at the Central Fuel Research Institute, Jealgora, for studying the carbonizing characteristics of non-coking coals. A rapid modified Liebig's method for the determination of carbon and hydrogen in tar, pitch, anthracene and other high boiling point liquids has been developed.

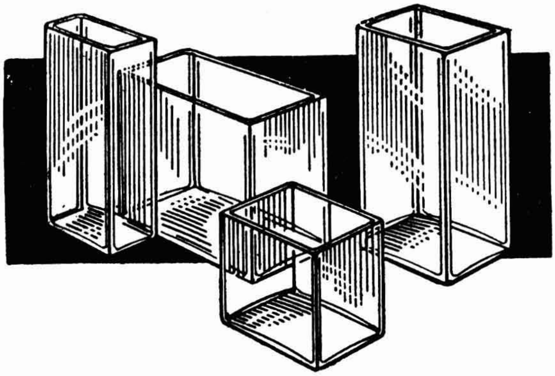
Palaeobotanical investigation of Indian coals from Samla Seam has revealed a number of new fossils; a new type of megaspore has been identified.

Buildings and roads — An ultrasonic tester for field and laboratory testing of building materials and structures has been built. The instrument is suitable for detecting and determining the depth of surface cracks and also locating weak and strong zones of concrete in a structure.

A standard pneumatic test which yields reliable results for abrasive resistance of aggregates and cement mortar has been developed. A test track has been constructed to facilitate testing of various specifications for cheap all-weather roads.







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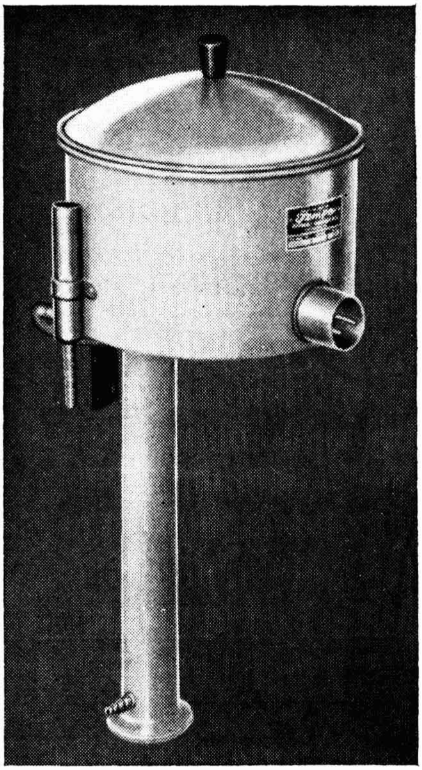

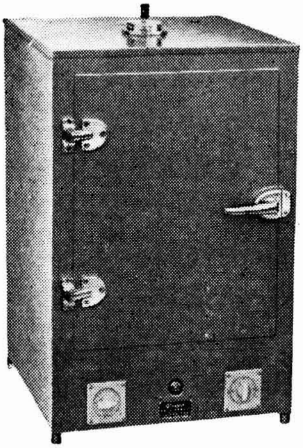

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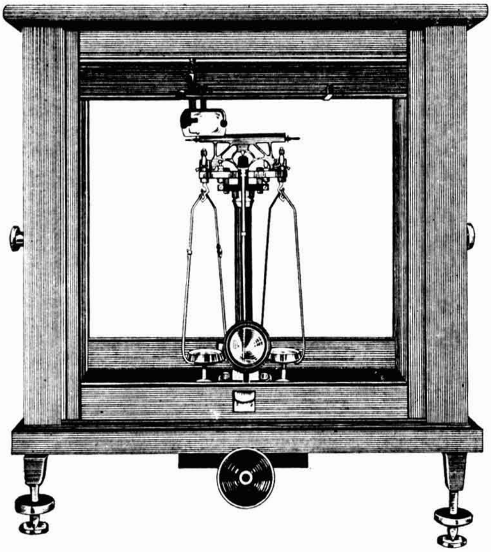

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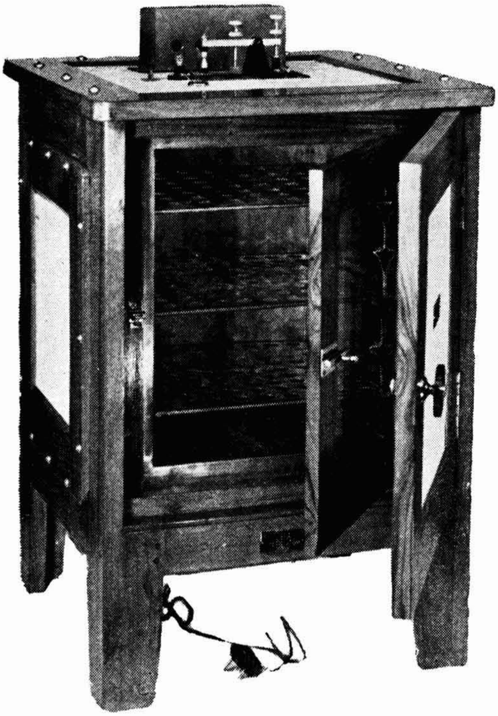

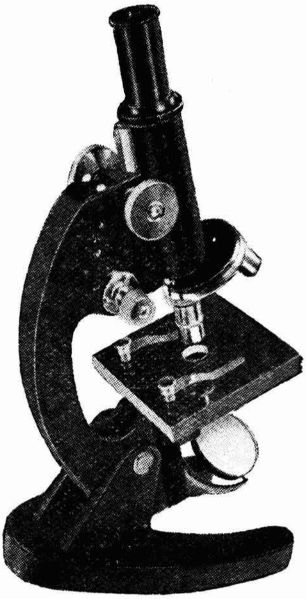

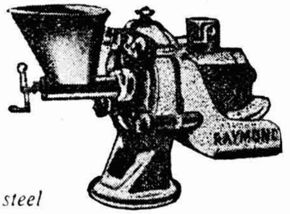
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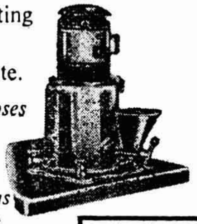

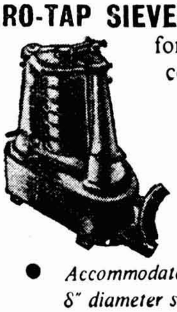

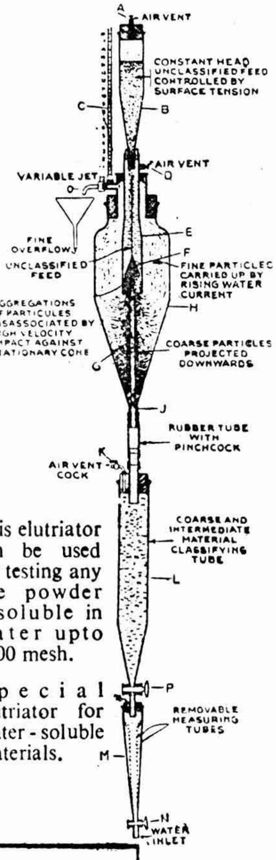
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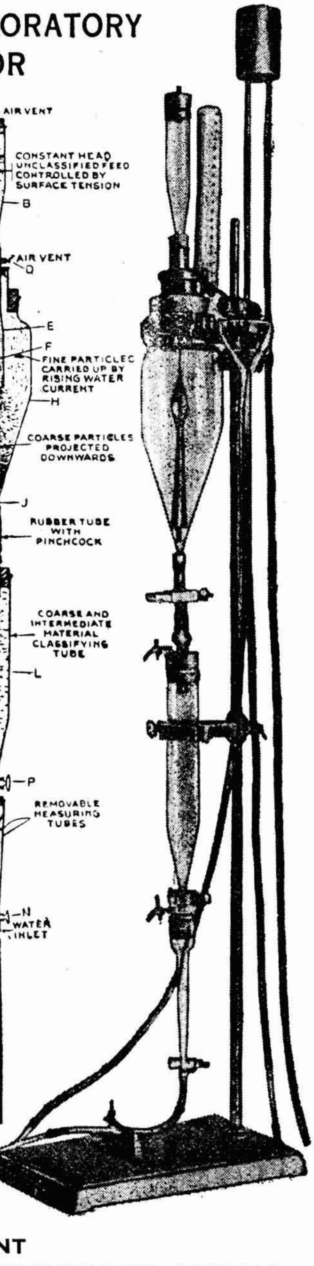

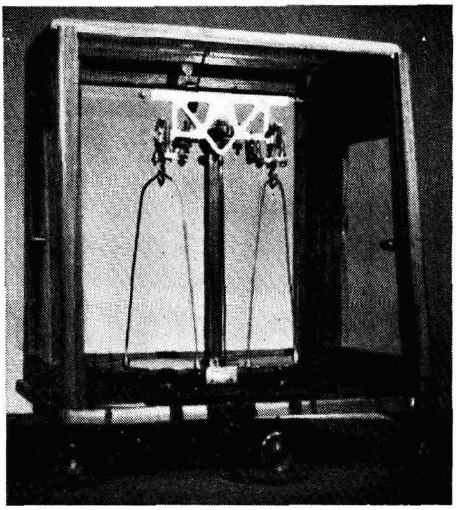

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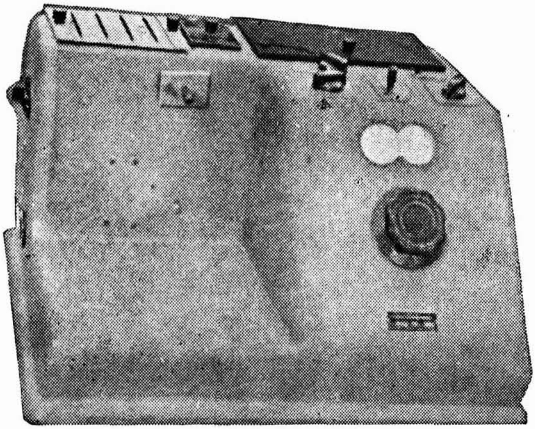

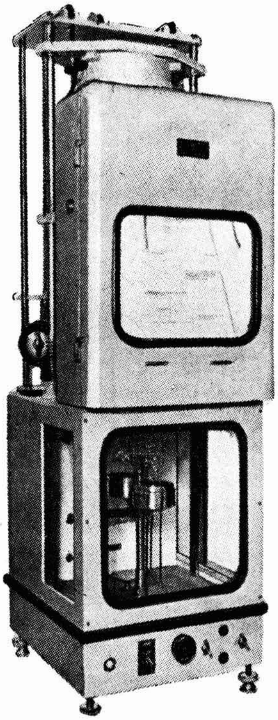

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