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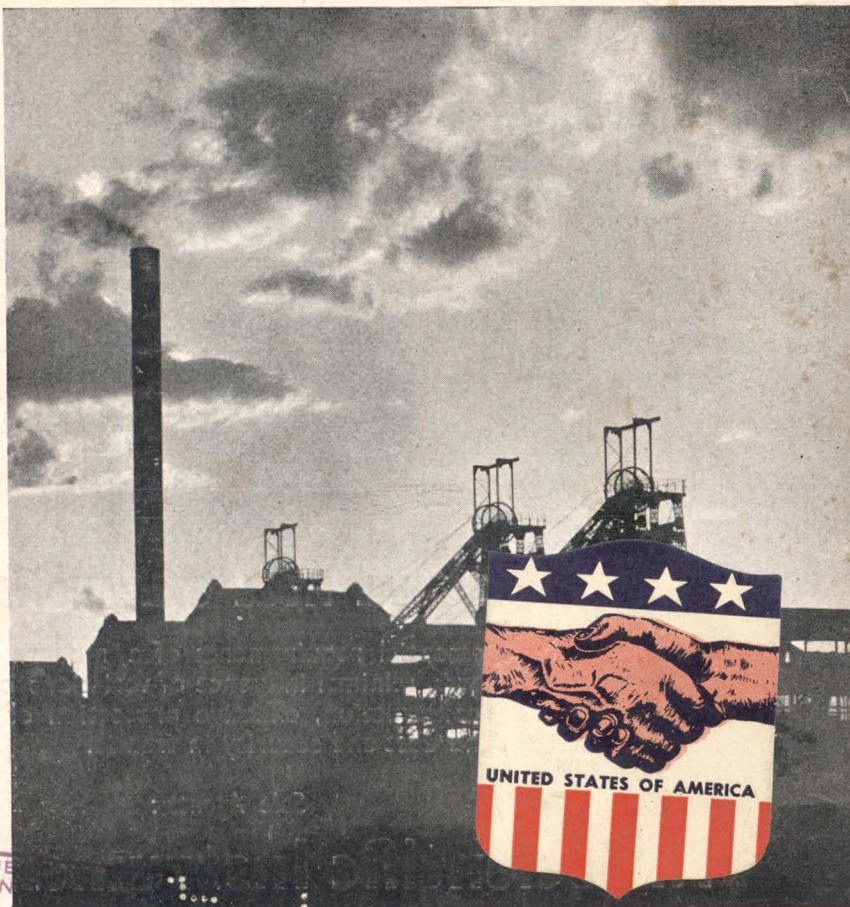


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- Atomic Nuclear Models & Beta Decay
- Chemical Examination of Kutki (*Picrorhiza kurooa*)
- Chemical Composition of Flowers of *Butea superba*
- Wax-like Products from Castor Oil
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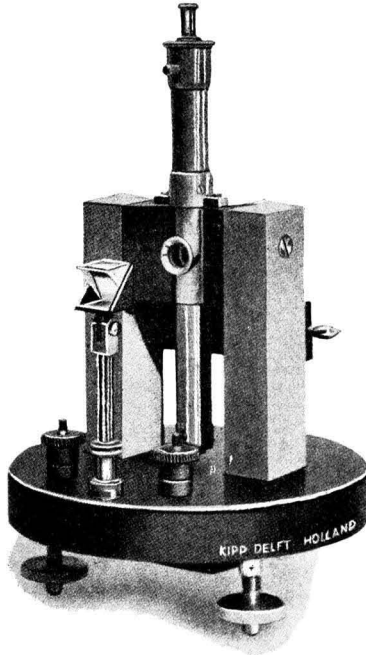
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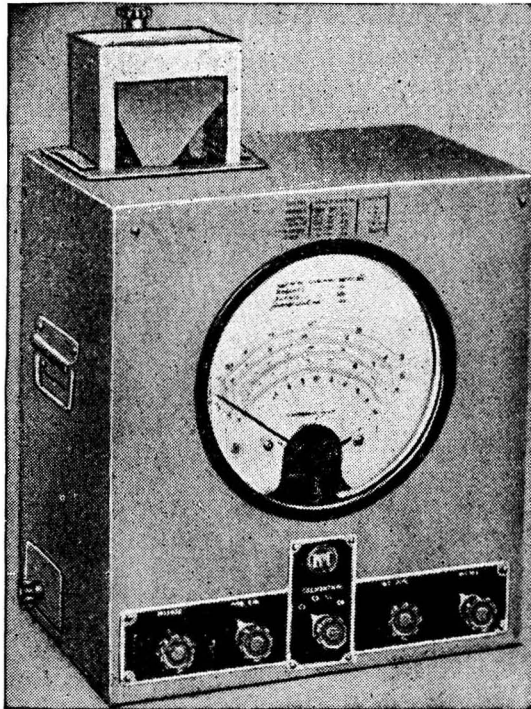
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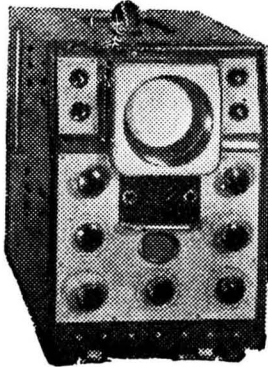
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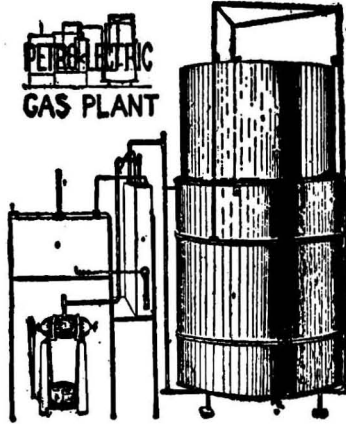
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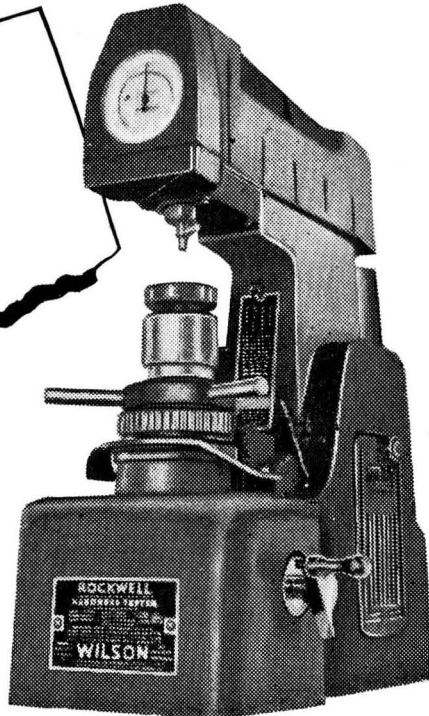


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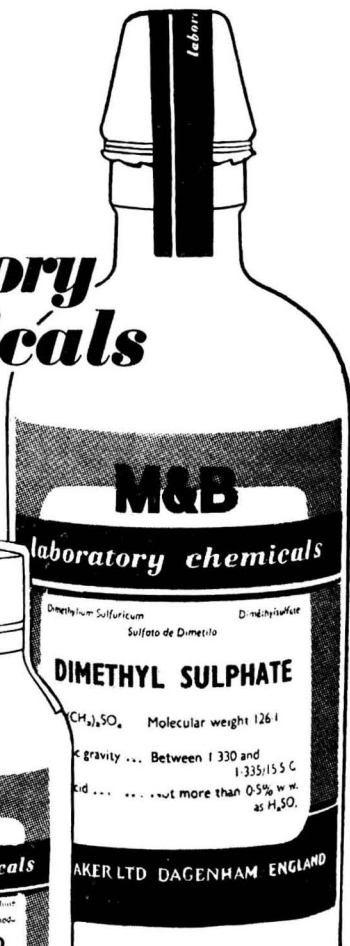
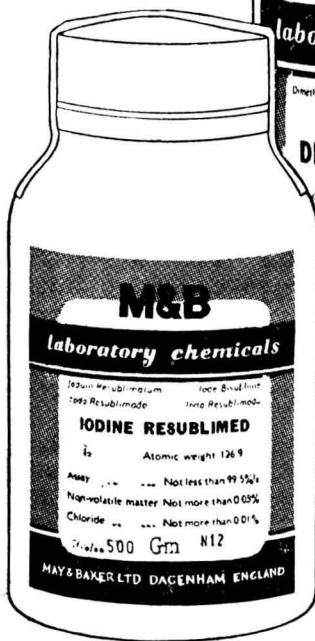
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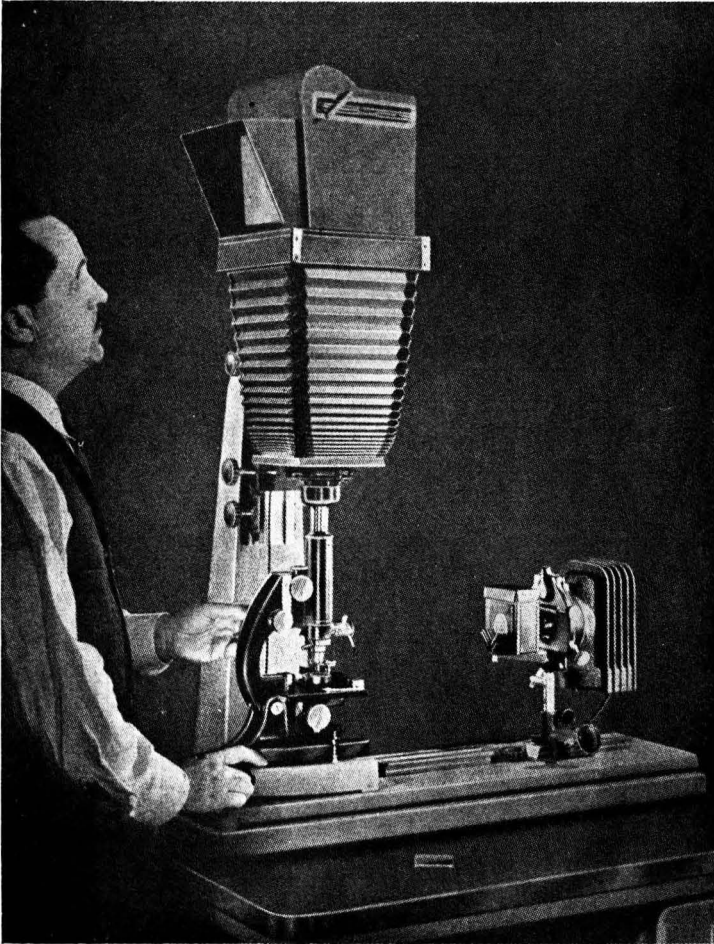
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**M**ETALS form one of the earth's most important resources in the support and enrichment of human life. In ancient and medieval times, metals helped man directly in his various avocations, his arts and crafts, raised his standard of living and lent grace to life by their use in the fine arts. Modern man today needs metals as much for the munitions of his destructive wars as for implementing human welfare in daily life.

The depletion of the earth's store of metals has increased progressively since 18th century but, with the 20th century era of world wars, it is going to become one of the biggest problems of the next century. Since 1914 more basic metals have been used up ( a large part either destroyed or irreclaimably locked up ) than during the whole of human history. The diminishing reserves of metals such as tin, lead, zinc have already become a problem. The 18th International Geological Congress, held at London in August 1948, devoted a special section to the discussion of " The geology, paragenesis and reserves of the ores of lead and zinc " which brought out this feature in relief. Doubtless, in some measure, the substitution of plastics and alloys of the light metals which are in more abundant supply and the discovery of new reserves in the remaining unexplored countries, together with resort to deeper mining and developments in the practice of ore-refining and upgrading, may put off the scarcity for two or three generations, but the lesson of metal depletion from the accessible parts of the earth's crust should be taken as nature's grim warning that man is spending away a prime treasure which is non-replenishable.

The critical shortage in metals is most felt up to now only in respect of tin, lead and zinc, but signs are apparent that accessible deposits of copper, nickel, manganese, wolfram and antimony are diminishing and new discovery is not keeping pace with demand. The extractable stock of platinum, silver and gold, left for future needs within minable depth, is becoming meagre. The situation, though general for the whole world, however, is becoming acute in the Western Hemisphere, chiefly the countries around the North Atlantic border where modern standards of life demand an inordinate use of metals ; for the Eastern countries whose consumption of metals is comparatively insignificant and who have only played the rôle of miners of tin, manganese, wolfram, antimony and their accessories for wholesale export to the West, the situation is not so grave. This disequilibrium in the world's mineral economics calls for adjustment by wise and equitable solution and not by resort to the old policy of colonial exploitation, tariff barriers, etc., against weaker nations.

### Trends in the Use of Metals

Since the advent of the industrial era, the under-developed countries of the world have been exploited for their metals and ores by the industrially developed countries. Tin, manganese, wolfram and antimony have thus flowed in increasing volume from Malaya, Siam, India, Burma, China, Indonesia to Europe and North America. The *per capita* consumption of these metals in the producing countries has been negligible, and to the extent to which their mineral production has helped the development of metal industries of the world since

---

\* Paper by D. N. Wadia read at the United Nations Conference on Conservation and Utilization of Resources at Lake Success, August 1949.

1850 must be measured by their share in the spread of the industrial civilization of the last century. But the material return obtained by these countries for this service has been disproportionately small in most instances. This factor will tend to restrict, in the coming era of self-determination for each nation, the flow of minerals and metals in future, except for more substantial gains. A growing trend towards conservation of mineral resources in hitherto backward countries and their utilization (or barter) for national benefit will be the most significant development of the decade. The natural result of this may be that the large engineering and manufacturing industries of the world will not be able to draw, as unrestrictedly as in the past, on ores and raw materials except on the basis of reciprocity and on more liberal terms of exchange with manufactured goods which these backward countries need in building up more healthy national economy and standard of living.

India has supplied 40 million tons of manganese ores of the higher grade since 1900 at a price but little above the cost of mining and transport to the consumer; Malaya, Indonesia, Siam and China have supplied  $5\frac{1}{2}$  million tons of primary pig-iron during the same interval, and continues still to furnish the major part of the world's demand for this essential metal, for which no substitute is in sight. The largest supply of the strategic metal, tungsten (78 per cent), has come from China, Burma and Malaya ever since the discovery of its high potential in war and defence. China's mines supply 71 per cent of antimony metal, maintenance of which is vital to a number of industries. In a similar category must be placed the regular pre-war exports of over 2 million tons of iron ore to Japan, an industrially developed country, by Malaya and other undeveloped countries. Ceylon has supplied  $1\frac{1}{4}$  million tons of superior graphite during the last 60 years.

#### Lack of Policy of Conservation

None of these countries had any mineral conservation policy in the past and mining practice was unscientific, uneconomic and inefficient. A national conservation policy must imply complete and accurate appraisal of resources by organized resource surveys. With the exception of India, where an official geological survey has been in existence for nearly a century and which has since the war been quadrupled, and to a less extent in

China, Burma and Malaya, no scientific agency for mineral resource estimation, sampling and geological exploration on modern lines has been in working in Asian countries till very lately. The technique of aerial survey and mapping, geophysical investigation at depth, statistical control in resource assessment and mining, are being gradually applied in India after decades of experience of unsound mining and despoiling of depletable and non-renewable resources for export purposes.

But new trends are appearing in the post-war years. India has now a Bureau of Mines which is planning a programme of detailed appraisal of mineral deposits with systematic testing, stoppage of haphazard mining, ore beneficiation and use of statistics in enforcing conservation by local processing and utilization. Education and training of technical and mining personnel for putting into practice the chief elements of this programme is undertaken since 1945. It is only a matter of time for other countries of south-east Asia, which have metalliferous minerals, to follow in the wake of this programme. In these countries the pressure of increasing population, rising living standards, especially of food and public health, is creating an insistent desire for proper development, conservation and utilization of their mineral wealth, viz. use of new techniques and effective controls on export of raw products in place of the *laissez faire* attitude of the past. Everywhere there is a sustained drive to bring into use mineral resources as yet untapped to build up, with the revenues obtained from this source, more productive and diversified economies. The idea has slowly dawned that national resources must be cultivated for enriching the life of the people by judicious use and conservation.

The general lack of trained geological and mining personnel, technicians, engineers and other experts in the specific fields is being made good slowly by an intensive educational effort in training in local institutions, overseas scholarships and engagement of foreign experts on contract.

#### Extent of Metal Resources of India & Countries of the Far East

The resources of India in certain metals and ores are fully adequate to support higher living standards for the masses which is the aim of national planning. They are of a scale to ensure supply of indigenous needs for centuries and leave an exportable surplus

margin. The estimated deposits of iron, manganese, aluminium, titanium-ores and of the ferro-alloy metals, chromium and vanadium, give promise of India becoming, in the foreseeable future, an important metallurgical centre for ferrous metals and alloys and for the light-metal alloys. The strategic metals, thorium and beryllium, have been located in deposits of considerable size. Against this, the position in respect of non-ferrous metals is one of scarcity. The position with regard to lead, zinc, copper, cobalt and nickel may, in part, be improved by a more detailed exploration in the future. But, in respect of tin, silver, platinum and mercury, however, the deficiency is of a more permanent nature. A few notes on the existing proved and prospective metal resources of India with statistics of production and reserves are given in a following paragraph.

Burma has notable resources of wolfram and ores of lead, zinc, tin and nickel; China is believed to have the biggest iron reserves in the Western Pacific region, next only to the vast reserves of Singhbhum-Orissa in India, while her known tungsten and antimony reserves are yet of considerable magnitude. 60 per cent of tungsten and 71 per cent of antimony of the world is mined from these two countries from deposits which are regarded as ample for future needs. Malaya has moderate supplies of magnetic iron-ores, the chief source of supply to Japan. In tin, the south-east Asian countries — Malaya, Indonesia, Siam, China and Burma — virtually dominate the world production, though both production and distribution of tin is long controlled by the International Tin Cartel, which has prevented some of these countries from reaping the full benefit of their valuable natural asset. An indispensable accessory to metallurgical industries is graphite, for which Ceylon holds big reserves of pure crystalline and flake graphite, enough to last the world for a long period.

#### Outlook for Development & Potentialities

Expansion of these resources by discovery of new, or partly explored, or suspected deposits in the industrially under-developed countries named above is distinctly promising. On the other hand, the promise is limited by the difficulty and cost of the new methods of exploration, geophysical and geochemical, and insufficiently trained personnel. These features will retard the rate of discovery of new resources. These adverse

factors, however, have, in the past, proved to be not wholly disadvantageous, as they were the very factors that tended to conserve the underground resources of many undeveloped countries from too rapid depletion. In the race for export of ores, for which no indigenous market existed and the inducement of small immediate gain, the majority of the metal resources of these countries were mined haphazardly and unsystematically, the last consideration being given to any aspect of conservation. In countries which mine principally for export, this is the only aspect of conservation that needs effective safeguarding today, as it is the most serious threat to the world's mineral economy. The other aspects of metal resource conservation, viz. conservation of ore milling and concentration and conservation by substitution, reclamation, utilization of scrap, etc., are problems of the future when the mining industry has reached the second stage, viz. manufacture of the products of mines.

#### Metal Requirements for Industrial & Agricultural Developments

Accurate data and statistics of metal and ore production for export as well as for local consumption are unavailable for most Asian countries. Whatever ore is mined (and a considerable aggregate quantity as well as variety is mined) is mostly for trade purposes, for export overseas. The return for this is principally in the shape of consumer goods of general description and very subordinately in the form of processed or fabricated metals. The *per capita* use of metals, thus, in the every-day life of the people of these countries is not ascertainable through official statistics, beyond the knowledge that it is yet abnormally small. A trend for increased use of metals is slowly setting in, not for domestic or private life of the citizens, but for such general utility purposes as public works, engineering, railways, river navigation, etc.

The use of metals in agriculture in all the Asian countries is limited to a few primitive iron tools which the village smith fashions out of the local ores with forest fuel. All through the ages, man in Asia has made little use of metals in industry and agriculture, but he developed a remarkable skill in fashioning and alloying the metals, iron, copper, zinc, silver and tin, for his domestic utensils, the most obvious military weapons and articles of fine art. Ancient India acquired high reputation in the metallurgy

of iron and steel, copper, zinc and bronze. The fame of the ancient Indian steel *wootz*, in demand in Europe for sword blades, the 4th century iron pillar of Delhi, a solid shaft of rustless wrought iron, 16" diameter and 23' 8" high, and the ornamental bronzes and statuary of the 5th to 10th centuries are examples.

It would be, perhaps, too much to say that the comparatively small productivity of Indian agriculture in cereals, livestock and commercial crops is related to the small use of metals, or that more use of metals would mean better tillage. The age-old peasant traditions in India lend no support for these presumptions; at the same time, on a broad view of the problem, metals and machines are a *sine qua non* of large-scale agricultural practice, collective farming, reclamation of waste land, swamps and forests. Mechanized transport would bring fields, farms and factories nearer to markets. These are obvious facts, but rural and agricultural India, divided into 700,000 villages, reacts slowly to new forces.

Signs of mechanization of agriculture in some Asian countries are already becoming visible and the tractor-plough might become as popular in time as the motor-bus in the less remote parts, at any rate, of rural India. In industry, the pace is much quicker and the demand in India for pig-iron and steel, which used to be satisfied with about 2 million tons of the former and 1 million tons of the latter, put out by the Indian iron and steel plants in pre-war days, has increased fourfold at the present day. Better housing, road-building, motor transport, electrification programmes, cottage industries and the spread of air-conditioning in tropical climates will continue to swell these demands for metals in time. These, with heavy engineering plant and machinery required for the various multi-purpose water-power, irrigation and industrialization schemes which India and several Asian countries have launched since the war, will bring about a revolutionary rise in the East's demand for fabricated metals from a few hundred thousand tons to several million tons. To a limited extent, the indigenous resources of the Asian countries will be able to cope with these steeply rising needs in metals but a good part will have to be satisfied with imports from abroad. This demand will have to be adjusted against their capacity and willingness to continue to supply raw materials to the more advanced industrial

countries. To foster such interdependence of countries on the world's material resources and thus attempt to establish an equilibrium between these two sets of countries into which the world is unhappily divided today should be the goal of the United Nations Economic and Social Council. A desire for a friendly and satisfactory adjustment of this demand and supply for metals and minerals in meeting each other's wants and deficits in the coming years will be the best guarantee for the war-damaged world's peace.

#### Principal Metal Reserves in India, Production Statistics, etc.

Data and statistics regarding the following twenty metals found in deposits of workable size in India are given below:

**Aluminium** — Ore reserves are computed at about 250 million tons ( $Al_2O_3$ , 50 per cent); the deposits are fairly widespread throughout the country, in Bihar, Central Provinces, the Western Ghats, Kashmir and Madras. The annual production of aluminium in India is yet small; 4,000 tons, against a requirement of 15,000 tons.

**Antimony** — A deposit in Pakistan (Chitral) supplied about 1,000 tons of ore per year during the war. The smelting and refining was done at Bombay. China is the world's largest producer of antimony (about 16,000 tons in pre-war years), the largest deposit being found in the province of Hunan. The ore is smelted near the mines.

**Beryllium** — During the war, India exported considerable quantities of beryl. Beryl from Rajputana and Bihar is prized because it contains 11 to 14 per cent of  $BeO$ . The exports of beryl have been prohibited since 1946. Research on the isolation of beryllium and beryllium oxide is being carried out by a committee of the Board of Scientific & Industrial Research.

**Chromium** — Chromite is produced in Bihar, Orissa and Pakistan (Baluchistan); the last locality possesses large reserves. The production has been around 50,000 tons per year, the bulk of which was exported. Possibilities of upgrading lean chromite ores and manufacture of ferrochrome are being investigated.

**Cobalt** — The only area within the geographical limits of India which has reported deposits of cobalt is Nepal but the geology and probable reserves are not yet investigated.

**Copper** — The chief producing area is Singhbhum (Bihar) which has an annual

output of about 6,500 tons of metallic copper. Other reported deposits are under investigation. A number of abandoned sites where a copper industry flourished till a century or two ago are known; these supplied all the needs of India in copper and brass in medieval times. Burma produced copper matte (about 13,000 tons in the pre-war years) but the ore reserves are not large. Annual consumption of copper in India is 40,000 tons.

*Gold* — Annual production averages 300,000 fine oz. The gold mines of Kolar (Mysore) are now 9,600' deep, the deepest metal mine in the world. The ore persists at this depth, but the problem of rock-bursts is increasing in severity.

*Iron* — India's resources in iron-ore are of vast extent. In the district of Singhbhum and adjoining parts of Orissa, the reserves occurring at surface are computed at over 8,000 million tons (metal content 60 to 65 per cent). The reserves in the Central Provinces and parts of Madras are also large. In contrast with the vastness of ore reserves, however, the annual production of pig-iron and steel yet remains small (2 million tons of pig-iron and about 900,000 tons of steel). New steel plants are being installed and the manufacture of a range of ferro-alloys is being considered.

China's iron-ore reserves are only next to India's, though they have not been fully investigated and the production yet remains small.

*Lead* — Production of lead in India at the present day is insignificant, though two centuries ago considerable amount of ingot-lead was produced in several parts of the country, mainly for military requirements. India's annual lead import is about 8,000 tons.

Burma produced 75,000 tons of ingot lead in pre-war years. Reserves of lead-zinc-silver ores at the Bawdwin Mines are calculated at 4 million tons, containing 16 oz. of silver to the ton.

*Magnesium* — Magnesite deposits of large extent and high degree of chemical purity occur in Madras. In Salem the estimated reserve is 90 million tons, analysing over 96 per cent of magnesium-carbonate. A considerable part of the annual output is exported. Research on the preparation of metallic magnesium and light-metal alloys, principally with aluminium, is under investigation by the Board of Scientific & Industrial Research.

*Manganese* — Annual exports from India of high-grade manganese have often touched the 1 million ton mark during the last 20 years. Proved reserves of the richer grade ores (48 to 70 per cent) are computed at only about 15 to 20 million tons; the reserves of lower-grade ores (Mn 40 to 30 per cent) are several times this magnitude. Beneficiation of the latter by simple mechanical means will greatly add to the country's resources in this metal. Manufacture of ferro-manganese in India is being investigated and the unrestricted exports of manganese ore has now been controlled by the Government.

*Nickel* — There is a paucity of nickel-ore in India; the only notable occurrence of nickel-ores is reported from a locality in Nepal, but detailed prospecting and geological examination have not been done to ascertain the quantity and extent of the deposit.

The Bawdwin Mines of Burma produced annually nickel speiss averaging 3,000 tons, during pre-war years, containing 25 to 30 per cent of nickel. Annual consumption of nickel in India is about 2,000 tons.

*Strontium* — Large deposits of remarkably pure celestite, aggregating to about a million tons each, occur in the Trichinopoly district of Madras and in a West Punjab district (Pakistan). The mineral has not found industrial use in India yet.

*Thorium* — The thorium resources of India are considerable.  $\text{ThO}_2$  is a constant ingredient of the mineral monazite occurring in the form of beach sands, covering large areas of the Travancore coast. These beach deposits constitute a large potential source of thorium, which has now assumed strategic importance as an atomic fuel or a source of atomic energy. The export of monazite, for strategic reasons, is now prohibited by Government.

*Titanium* — The mineral ilmenite is widely distributed in India and constitutes an important mineral asset of the country. It occurs as highly concentrated black sand along a hundred-mile stretch of the Travancore coast. About 75 per cent of the world's requirements in titanium, ranging between 200,000 to 300,000 tons per annum were, prior to 1940, supplied by Travancore. The percentage of  $\text{TiO}_2$  in Travancore ilmenite runs to 52 to 62; the total reserves of ilmenite are estimated at over 300 million tons. Rutile is associated with ilmenite sands.

A considerable accumulation of ilmenite occurs in association with the alluvial deposits of tin-ore in Malaya from which it is separated by magnetic concentration.

*Tin* — This metal does not occur in India in any appreciable quantity. Malaya is the world's largest supplier of tin-ore, the quantity varying from 33,000 to 77,000 tons per annum (pre-war production), being about one-third the world's basic tonnage in this metal. This figure represents the entire output of Malayan mines. During the last 20 years, many of the richer placers have been exhausted. The largest proportion of the ore is won by dredging the alluvial and eluvial deposits. The remaining ore reserves are considered to be large. Indonesia and Siam are the next largest producers of tin-ore in south-east Asia, their share being 19,000 and 10,500 tons respectively per annum. China comes next with 6,500 tons. All the ore is mined by more or less primitive methods from shallow alluvial flats.

*Tungsten* — A small deposit of this metal was worked in India during war years. The world's largest producer of this valuable alloy metal is China which has yet maintained its lead in this metal in spite of her disturbed political conditions. The production in pre-war years was 11,000 to 17,000 tons, the present output being much smaller. The ore is worked from large deposits of detrital stream-tin. The next largest exporter is Burma with 5,000 to 6,000 tons (pre-war) followed by Chosen, 1,500 to 2,000 tons, and Malaya, from a few hundred to 2,000 tons. No part of

the ore won is used indigenously in these countries.

*Uranium* — Sporadic deposits of uranium-bearing minerals have been found in various localities in India, the largest being segregations of pitch-blende in mica-pegmatites in Bihar, from which a few tons were obtained some years ago. Geological work has revealed that such deposits are incapable of supporting continuous mining. Minerals containing the rare earths and metals have been observed in pegmatites traversing the mica fields of Bihar and Madras, viz. samarskite, gadolinite, columbite, tantalite, allanite, triplite, torbernite and thorianite.

*Vanadium* — Considerable reserves of this metal have been localized in parts of Bihar and Orissa as vanadium-bearing iron-ores; the vanadium occurs in these areas in amounts varying from 0.8 to 3 per cent. The total reserves of vanadiferous iron-ore is estimated at 3 to 5 million tons. The utilization of these ores for manufacture of vanadium-steel is under consideration.

*Zinc* — Zinc lodes have been observed at a few localities in India but no smelting of zinc is practised in India today. Slag deposits of large size in several districts suggest a flourishing zinc industry in medieval times, the zinc being refined for manufacture of brass and bronze for local consumption. The annual consumption of zinc in India, at present about 30,000 to 35,000 tons, is wholly met from imports.

*Zirconium* — Reserves of this metal are ample, but at present a fitful output of only 100 to 1,500 tons of the mineral zircon is worked for purposes of export.



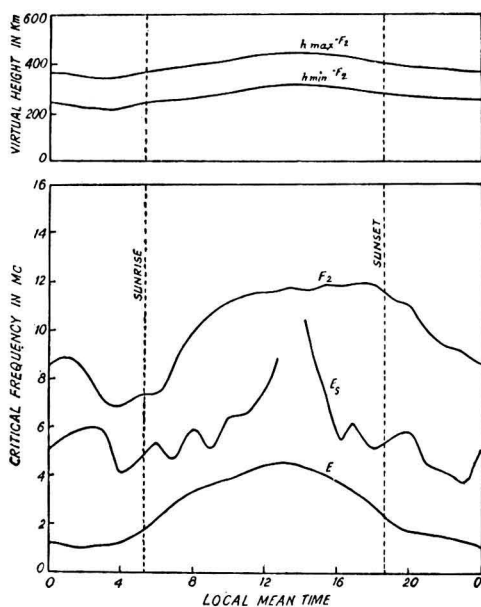
# Characteristics of the Ionosphere over Calcutta (July 1949)

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THE following are the ionospheric data observed at Calcutta for the month of July 1949.

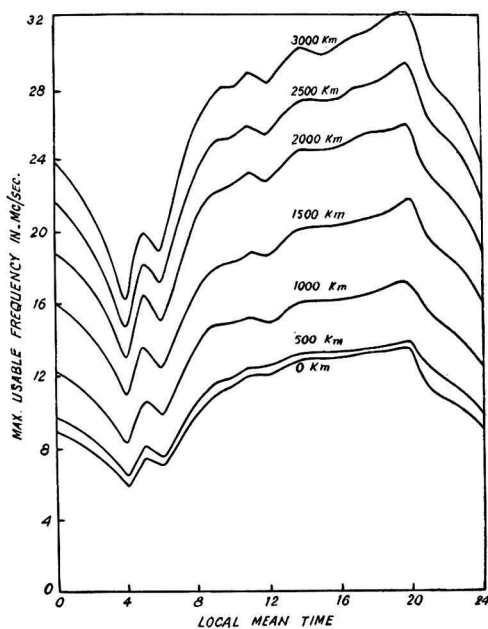
Fig. 1 represents the mean hourly values of the penetration frequencies of the E and E<sub>s</sub> regions and the penetration frequencies and virtual heights of region F<sub>2</sub>. The figures are obtained from average values of the data taken for each hour of the day for 5 days a week. Fig. 2 gives the prediction of the maximum usable frequencies which can be used for different distances of transmission by reflection at the F region over Calcutta for the month of October 1949. Table I gives the different occasions during routine observation when sporadic E ionization was observed and the values of the corresponding penetration frequencies and heights.



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

FIG. 1 — JULY 1949.

Sporadic E ionization was found to occur frequently during early morning hours but less in the afternoon unlike the previous month. Absorption of normal E echoes at mid-day was less than in the previous month. The behaviour of region F<sub>2</sub> was found to be normal in this month.



AT POINT OF REFLECTION.  
FIG. 2 — PREDICTED M.U.F. VIA F<sub>2</sub> LAYER DURING THE MONTH OF OCTOBER 1949.

| TABLE I      |      |       |                |           |
|--------------|------|-------|----------------|-----------|
| MONTH & YEAR | DATE | HOUR  | $f^{\circ}E_s$ | $h_{E_s}$ |
|              |      |       | Mc.            | Km.       |
| July 1949    | 1    | 05.00 | 2.70           | 105       |
|              |      | 08.00 | 7.45           | 120       |
|              |      | 12.00 | 5.40           | 120       |
|              |      | 14.00 | 10.00          | 135       |
|              |      | 16.00 | 6.00           | 120       |
|              |      | 17.00 | 4.25           | 120       |
|              |      | 18.00 | 7.00           | 120       |
|              |      | 20.00 | 3.25           | 105       |
|              |      | 21.00 | 3.00           | 105       |
|              |      | 22.00 | 3.95           | 90        |

TABLE I-- contd.

| MONTH & YEAR | DATE  | HOUR  | $f^{\circ}E_s$<br>Mc. | $hE_s$<br>Km. |
|--------------|-------|-------|-----------------------|---------------|
| July 1949    | 2     | 00.00 | 2.80                  | 90            |
|              |       | 10.00 | 6.00                  | 120           |
|              |       | 11.00 | 6.20                  | 120           |
|              | 4     | 18.00 | 3.30                  | 120           |
|              |       | 19.00 | 4.00                  | 120           |
|              |       | 20.00 | 4.75                  | 120           |
|              |       | 23.00 | 4.05                  | 105           |
|              | 5     | 00.00 | 4.25                  | 105           |
|              |       | 01.00 | 3.90                  | 105           |
|              |       | 06.00 | 5.00                  | 120           |
|              |       | 07.00 | 5.20                  | 120           |
|              |       | 08.00 | 6.10                  | 135           |
|              |       | 10.00 | 6.50                  | 135           |
| 21.00        |       | 3.50  | 135                   |               |
| 22.00        |       | 3.00  | 120                   |               |
| 23.00        |       | 2.50  | 105                   |               |
| 6            |       | 05.00 | 5.75                  | 120           |
|              | 06.00 | 8.20  | 135                   |               |
|              | 07.00 | 4.70  | 135                   |               |
|              | 09.00 | 5.00  | 135                   |               |
|              | 10.00 | 6.15  | 135                   |               |
|              | 17.00 | 5.30  | 135                   |               |
|              | 18.00 | 5.40  | 120                   |               |
|              | 7     | 08.00 | 7.00                  | 120           |
| 09.00        |       | 5.50  | 120                   |               |
| 16.00        |       | 4.25  | 120                   |               |
| 17.00        |       | 4.70  | 120                   |               |
| 21.00        |       | 2.30  | 105                   |               |
| 22.00        |       | 2.50  | 105                   |               |
| 23.00        |       | 2.00  | 90                    |               |
| 8            |       | 07.00 | 4.25                  | 105           |
|              | 10.00 | 4.50  | 105                   |               |
|              | 16.00 | 6.10  | 120                   |               |
|              | 20.00 | 6.00  | 120                   |               |
|              | 21.00 | 4.25  | 120                   |               |
|              | 22.00 | 4.00  | 105                   |               |
|              | 9     | 06.00 | 6.00                  | 120           |
| 07.00        |       | 4.25  | 120                   |               |
| 09.00        |       | 5.00  | 120                   |               |
| 12.00        |       | 4.70  | 120                   |               |
| 11           |       | 16.00 | 4.70                  | 120           |
|              | 17.00 | 10.40 | 135                   |               |
|              | 18.00 | 7.85  | 135                   |               |
|              | 19.00 | 7.00  | 120                   |               |
|              | 21.00 | 6.75  | 120                   |               |
|              | 22.00 | 3.25  | 105                   |               |
|              | 23.00 | 4.70  | 105                   |               |
|              | 12    | 00.00 | 7.95                  | 105           |
| 01.00        |       | 7.95  | 105                   |               |
| 02.00        |       | 7.95  | 105                   |               |
| 03.00        |       | 6.10  | 105                   |               |
| 04.00        |       | 4.70  | 105                   |               |
| 07.00        |       | 4.70  | 120                   |               |
| 08.00        |       | 5.35  | 120                   |               |
| 09.00        |       | 6.00  | 135                   |               |
| 11.00        |       | 6.00  | 135                   |               |
| 19.00        |       | 4.20  | 120                   |               |
| 20.00        |       | 4.00  | 120                   |               |
| 21.00        |       | 4.25  | 105                   |               |
| 22.00        |       | 4.20  | 105                   |               |
| 23.00        |       | 4.00  | 105                   |               |
| 13           |       | 00.00 | 4.00                  | 90            |
|              | 02.00 | 5.30  | 90                    |               |
|              | 06.00 | 5.20  | 120                   |               |
|              | 09.00 | 4.20  | 120                   |               |
|              | 18.00 | 5.30  | 120                   |               |
|              | 19.00 | 4.40  | 120                   |               |

TABLE I— contd.

| MONTH & YEAR | DATE  | HOUR  | $f^{\circ}E_s$<br>Mc. | $hE_s$<br>Km. |
|--------------|-------|-------|-----------------------|---------------|
| July 1949    | 14    | 08.00 | 4.60                  | 105           |
|              |       | 09.00 | 5.00                  | 120           |
|              |       | 10.00 | 4.90                  | 120           |
|              |       | 23.00 | 2.70                  | 90            |
|              | 15    | 01.00 | 6.65                  | 90            |
|              |       | 02.00 | 6.00                  | 90            |
|              |       | 03.00 | 5.50                  | 90            |
|              |       | 04.00 | 4.95                  | 90            |
|              |       | 05.00 | 4.95                  | 105           |
|              |       | 07.00 | 4.95                  | 105           |
|              |       | 09.00 | 5.40                  | 105           |
|              |       | 19    | 22.00                 | 4.60          |
|              | 23.00 |       | 3.20                  | 90            |
| 20           | 00.00 | 3.00  | 90                    |               |
|              | 01.00 | 3.20  | 90                    |               |
|              | 02.00 | 4.00  | 90                    |               |
|              | 06.00 | 5.00  | 105                   |               |
| 21           | 10.00 | 4.70  | 120                   |               |
|              | 22    | 04.00 | 2.50                  | 90            |
| 05.00        |       | 2.50  | 90                    |               |
| 06.00        |       | 2.50  | 105                   |               |
| 07.00        |       | 4.65  | 105                   |               |
| 08.00        |       | 4.75  | 105                   |               |
| 09.00        |       | 4.75  | 120                   |               |
| 16.00        |       | 4.60  | 120                   |               |
| 17.00        |       | 4.25  | 120                   |               |
| 23           | 10.00 | 4.70  | 120                   |               |
|              | 11.00 | 4.80  | 120                   |               |
| 25           | 18.00 | 4.25  | 120                   |               |
|              | 19.00 | 4.00  | 120                   |               |
| 26           | 08.00 | 4.55  | 120                   |               |
|              | 10.00 | 11.25 | 135                   |               |
|              | 12.00 | 12.50 | 135                   |               |
|              | 15.00 | 7.20  | 120                   |               |
|              | 16.00 | 7.00  | 120                   |               |
|              | 17.00 | 4.70  | 120                   |               |
|              | 18.00 | 3.85  | 120                   |               |
|              | 19.00 | 11.75 | 135                   |               |
|              | 20.00 | 11.00 | 135                   |               |
|              | 21.00 | 10.80 | 135                   |               |
| 27           | 22.00 | 8.00  | 120                   |               |
|              | 23.00 | 6.70  | 120                   |               |
|              | 00.00 | 8.50  | 105                   |               |
|              | 01.00 | 6.90  | 105                   |               |
|              | 09.00 | 4.50  | 105                   |               |
|              | 10.00 | 11.25 | 120                   |               |
|              | 11.00 | 10.00 | 135                   |               |
|              | 13.00 | 13.10 | 150                   |               |
| 28           | 14.00 | 10.80 | 150                   |               |
|              | 17.00 | 9.60  | 135                   |               |
|              | 08.00 | 8.20  | 120                   |               |
|              | 11.00 | 6.00  | 120                   |               |
|              | 12.00 | 7.50  | 135                   |               |
|              | 17.00 | 3.40  | 120                   |               |
| 29           | 18.00 | 3.90  | 120                   |               |
|              | 00.00 | 5.25  | 120                   |               |
|              | 10.00 | 6.00  | 135                   |               |
|              | 14.00 | 12.00 | 150                   |               |
| 30           | 18.00 | 4.70  | 135                   |               |
|              | 19.00 | 3.80  | 120                   |               |
|              | 01.00 | 4.50  | 105                   |               |
|              | 05.00 | 7.00  | 120                   |               |
|              | 07.00 | 4.50  | 120                   |               |
|              | 09.00 | 5.25  | 120                   |               |



# Atomic Nuclear Models & Beta Decay

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## 1. Radioactivity & Nuclear

### Model for the Atom

**R**ADIOACTIVITY, which is characterized by spontaneous emission of unique radiations, was discovered by Becquerel in 1896. Soon after the discovery of the radioactivity of uranium, a number of other heavy elements were also found to be radioactive. An interpretation of this phenomenon was made on the basis of Rutherford nuclear model for the atom. The atom consists of a central heavy particle or nucleus in which most of the mass and the total positive charge are concentrated. The nucleus is surrounded by a certain number of moving electrons in configuration whose total negative charge compensates the nuclear charge giving rise to neutral atom. The charge of the nucleus is thus an integral multiple  $Z$  of the electronic charge.

Chemical and spectroscopic properties are determined by the nuclear charge which determines the number of electrons and their distribution in the extra nuclear electronic configuration of the atom while mass properties exhibited by the atom are due to the nuclear structure, the mass being given by the mass of the nucleus. Thus, a clear and distinct separation of the chemical and spectroscopic properties from the mass properties are provided by the nucleus. Hence there can be, as there actually are, isotopic atoms, that is, atoms of different masses having the same chemical properties and practically the same spectrum.

The phenomenon of radioactivity is due to instability in the very structure of the nucleus of the atom which leads to a change in either the mass and charge properties together or in only the charge properties with the mass properties practically unaffected. In other words, the spontaneous emission of radiations gives rise to a change in either the mass and charge properties jointly or in only the charge properties. These radiations emitted by radioactive elements, as a result of careful investigation by

absorption and other methods, are found to be of three different and clearly defined groups, alpha, beta and gamma.

Of these, the alpha rays are found to be doubly charged atoms of helium. In other words, they are particles with a positive charge of two units and a mass of four atomic mass units,  ${}_2\text{He}^4$ . They, as positively charged particles, are deflected by magnetic and electric fields. The beta rays are positively or negatively charged, high-speed positrons or electrons and, as such, are also deflected by these fields. The gamma rays, on the other hand, are electro-magnetic radiations of very high frequency and they are unaffected by magnetic and electric fields.

## 2. The Proton-electron Nuclear

### Model & Emission of Beta Rays

The emission of radiations accompanied by a change of mass properties indicates that the atomic nucleus is not a simple particle, rather it is composite, and properties of nuclei such as momentum or spin which can be deduced from optical spectra, confirm the assumption that this composite consists of elementary particles. In the earliest development of the nuclear theory, the nucleus was assumed to be built up of protons and electrons. The proton-electron nuclear model encountered many difficulties.

The emission of electrons as beta rays from radioactive nuclei was long regarded as direct evidence for the existence of electrons in nuclei. With the discovery of numerous artificially produced radioactive nuclei which emit positrons instead of electrons in their energy spectra, this argument proved to be untenable, since beta radioactive substances may emit either positive or negative electrons from their nuclei.

The electron and proton, by direct measurement, are each found to have a spin of  $\frac{1}{2}$ . Hence the angular momentum of a nucleus, composed of an odd total number of particles, must be half integral. Band

spectra show that the nuclear spin of  ${}^7\text{N}^{14}$  is 1. Therefore, it cannot contain an odd number of particles, 14 protons and 7 electrons. Similarly  ${}^1\text{H}^2$ ,  ${}^3\text{Li}^6$  and  ${}^5\text{B}^{10}$ , each with spin 1, cannot contain odd number of particles as required by nuclear proton-electron hypothesis.

${}^7\text{N}^{14}$ , as shown by band spectra, follows Bose-Einstein statistics and hence must contain an even number of nuclear particles. If the nitrogen nucleus contained 14 protons and 7 electrons or 21 nuclear particles in all, it would have to obey Fermi-Dirac statistics, since the total number of particles is odd. Also  ${}^1\text{H}^2$ ,  ${}^3\text{Li}^6$  and  ${}^5\text{B}^{10}$  which would have to follow Fermi-Dirac statistics actually follow Bose-Einstein statistics.

Thus the spin and statistics properties, which the proton-electron nuclear model is expected to have, come into conflict with those actually observed in a nucleus.

Measured nuclear magnetic dipole moments are all of the order of the nuclear magneton  $e/h/2MC$ . The Bohr magneton containing the electron mass  $m$ , instead of the proton mass  $M$ , is  $M/m$  which is equal to 1,840 times as large as the nuclear magneton. The measured magnetic dipole moment of the electron is one Bohr magneton and is negative in sign, corresponding to the rotation of a negative charge in the spin direction. The presence of electrons in the nucleus would, therefore, require nuclear magnetic moments nearly 2,000 times greater than what is actually observed and these would be negative in sign.

If the beta particles emitted by radioactive elements, as the proton-electron hypothesis implies, be high-energy constituents of their nuclei, which have mean life from incredibly small fraction of a second up to a million years or more, then there must be some power to hold these electrons for this length of time inside the nuclei. In analogy to alpha particles, one may attempt to devise a potential barrier. But it is impossible, as Bethe and Bacher<sup>1</sup> have shown, to devise a nuclear potential barrier to hold electrons inside the nucleus unless the height of the barrier is of the order of 13 times the height of the  $k$ -level in heavy atoms. Such a nuclear potential would cause large perturbations of the optical and X-ray levels which are definitely not observed.

The electrons observed in beta disintegration cannot exist in the nucleus prior

to their emission as beta rays. This inability can be judged quantitatively from the fundamentals of wave mechanics. The De Broglie<sup>2</sup> wavelength  $\lambda$  associated with a particle of momentum,  $P=mv$ , is given by

$$\lambda = \frac{h}{P} \quad (1)$$

where  $h$  is Planck's constant. Now, to derive the relativistic expression for momentum  $P$  from equation for kinetic energy, let  $C$  be the velocity of light,  $m$  the mass of the particle moving with a velocity  $v$ ,  $m_0$  its rest mass and  $E$  the kinetic energy of motion, given by

$$\begin{aligned} E &= m_0 C^2 \left( \frac{1}{\sqrt{1 - \frac{v^2}{c^2}}} - 1 \right) \\ &= (mC^2 - m_0 C^2) \\ &= \Delta m C^2 \end{aligned}$$

Then the momentum, expressed in terms of the kinetic energy, has the form

$$P = mv = \frac{1}{C} \sqrt{2Em_0 C^2 + E^2} \quad (2)$$

Beta rays from most of the naturally radioactive elements have energies of 1.5 MEV or less, and an electron of this energy whose wavelength  $\lambda$ , from equations 1 and 2, is  $630 \times 10^{-13}$  cm. could not exist as a standing wave even in the heaviest nucleus of uranium since its diameter is  $25 \times 10^{-13}$  cm.

This fact becomes more obvious when the quantization of moment of momentum of an electron is considered. Heisenberg<sup>3</sup> has shown that this quantization implies that simultaneous knowledge of the position and momentum of a particle cannot have an accuracy greater than that implied in the expression

$$\Delta P \times \Delta q = \frac{h}{2\pi} \quad (3)$$

where  $\Delta P$  and  $\Delta q$  are simultaneous indeterminacies in momentum and position. To consider minimum indeterminacy of the position of an electron, a maximum indeterminacy in its momentum may be assumed. Let this indeterminacy be its entire momentum  $P$ . Then the minimum indeterminacy in position is

$$\Delta q_{\min.} = \frac{h}{2\pi P} = \frac{\lambda}{2\pi} \quad (4)$$

i.e. the minimum indeterminacy is about De Broglie wavelength  $\lambda$  divided by  $2\pi$ .

Therefore, the minimum indeterminacy in position of an electron with an energy of 1.5 MEV is about  $100 \times 10^{-13}$  cm., or 4 times the diameter of the largest nucleus. Hence it could not be stated that the beta particle had ever existed in the nucleus.

From the considerations of the principle of indeterminacy, it can be found that if the position of an electron is to be known within  $\Delta q = 10 \times 10^{-13}$  cm. or about the diameter of the nitrogen nucleus, the energy of the electron must be of the order of 19 MEV. The total binding energy, however, of the  ${}^7\text{N}^{14}$  nucleus is only about 100 MEV, and if this were to be shared between 14 protons and 7 electrons in this nucleus, as the proton-electron nuclear model implies, then there would be too little energy per particle to meet the minimum energy requirement and, therefore, these electrons could not be confined to the nuclear volume.

Thus, these considerations show that the proton-electron nuclear model is untenable and that the fundamental difficulties associated with this hypothesis can be surmounted when the electron is, as proposed by Heisenberg and Majorana, replaced by a heavy particle without charge.

### 3. The Neutron-proton Nuclear Model & Beta Decay

With the break down of the proton-electron hypothesis, there came the neutron-proton nuclear model with the proof of the existence of another fundamental nuclear particle, the neutron. Chadwick's discovery of neutron<sup>4,5</sup> which threw great light on the structure of atomic nuclei, confirmed the hypothesis of a nucleus composed of neutrons (neutral particles) and protons (hydrogen nuclei). According to this hypothesis, the atomic number  $Z$  of an element is the number of protons in its nucleus while its mass number  $A$  is the total number of protons and neutrons in the nucleus, or

$$Z = N_p, \text{ and } A = (N_p + N_n) \quad (5)$$

where  $N_p$  and  $N_n$  are the number of protons and neutrons respectively.

The neutron-proton nuclear model, consisting of heavy particles, surmounts some of the principal difficulties which the former encountered. On this hypothesis, since neutron, like proton, follows the Fermi statistics, it is expected that nuclei of odd mass number obey Fermi-Dirac statistics while those of even mass number obey Bose-

Einstein statistics. Also, since the spin of either one of the elementary nuclear constituents is half integral, the nuclear spin is expected to be half integral or integral according to whether the mass number is odd or even. The nuclear spin and statistics properties for stationary states, expected on this neutron-proton hypothesis, have been actually verified by experiments. Further, the nuclear magnetic dipole moments which, on this hypothesis, are all expected to be of the same order of magnitude as those of the proton and neutron, are also confirmed by experimental evidence.

In discussing the proton-electron nuclear hypothesis, it has been shown that the presence of an electron in a nucleus is an objectionable feature both from the point of view of the nuclear dynamical and statistical properties as well as from that of beta decay. If the presence of electrons in the nuclear structure be excluded, then it is natural to suppose that in the process of beta decay, the electrons are formed at the moment of their actual emission in a manner analogous to the emission of light quanta from atoms. This assumption solves the difficulties of the nuclear magnetic moments, the magnitude of wave function in relation to the nuclear dimensions and assumption of potential barrier encountered by the proton-electron nuclear model. There, however, remain the difficulties of statistics and spin. In addition to these, a still more serious difficulty is that beta disintegration electrons have energies distributed over a wide range from zero to a fairly defined maximum  $E_0$  in spite of the fact that the initial nuclei, before the emission of beta rays and, after their emission, the residual product nuclei have quite exactly determined energies.

Thus the situation to be confronted with is that the parent nucleus, in a quantized state of definite energy, emits an electron leaving a product nucleus also in a quantized state of definite energy. The emitted electron, however, does not carry away the energy difference  $\Delta E$  between these two quantized states but any energy between this value and the energy value which corresponds to the rest mass of an electron, the exact energy in each individual case being apparently determined by chance. To account for this, Bohr proposed the theory of non-conservation of energy in the process of beta disintegration.

This hypothesis, however, is in conflict with the observation that the total energy is conserved in the process of beta emission as far as the upper limits of beta-ray spectra are concerned. It encounters, also other serious theoretical and experimental difficulties.

Then Pauli pointed out that the principle of conservation of energy is valid and the conservation laws may be retained if it be assumed that in beta disintegration a new particle which is escaping observation is emitted along with the beta particle, carrying away the missing energy, momentum and spin. According to Pauli, this new kind of particle which he calls neutrino is devoid of electric charge and is of very small mass. It has practically no interaction with matter.

In explaining the facts of continuous energy spectra, Pauli assumes that in each beta decay, an electron and a neutrino are simultaneously emitted, that the maximum energy of beta rays occurs when the neutrino is emitted with zero velocity and that the energy difference  $\Delta E$  between the initial and the product nuclei is distributed among the electron and the neutrino so that the electron receives a part of  $\Delta E$ , varying from case to case. Then the maximum kinetic energy, which the electron may receive, is given by

$$E_0 = \Delta E - (m_e + m_\nu)c^2 \quad (6)$$

where  $m_e$  and  $m_\nu$  are the rest masses of the electron and neutrino respectively and  $E_0$  is the disintegration energy, represented by the upper limit of the energy spectrum.

The difficulties of spin and statistics for a stationary state of a nucleus are, on the basis of neutron-proton nuclear model, overcome, but we are confronted with these difficulties for nuclear instability state with beta emission. Since in the beta transformation the atomic weight of the nucleus does not change, it is expected that the properties of a nucleus having an integral or half integral spin and Fermi or Bose statistics would be reversed by the emission of an electron—a particle with half spin and Fermi statistics. These properties, however, are not changed by this transformation of initial nucleus into product nucleus. The unchangeability of these properties can be accounted on the assumption of the emission of the second particle, the neutrino of half spin and Fermi statistics, inasmuch as the resultant spin of the

simultaneously emitted electron and neutrino is integral (1 or 0) and the neutrino and electron, both being elementary particles, each with Fermi statistics, the initial statistics properties of the parent nucleus are retained by the product nucleus.

From the considerations outlined above and from the results of experience, it may be stated that a neutrino has no charge and has a very small mass, only a fraction of the electron rest mass. It follows Fermi statistics and its spin  $\frac{1}{2}\hbar = \frac{h}{4\pi}$  where  $h$  is

Planck's constant. Its magnetic moment, if any, must be less than  $\frac{1}{7000}$  magneton. Nahmias' search<sup>6</sup> for ionization by neutrino, using strong radioactive sources shielded by large amounts of lead (about 1 metre lead) in order to absorb alpha, beta and gamma rays and leave only the neutrinos, shows that neutrinos cannot form more than 1 ion in about 500,000 kilometres path in air, indicating that their magnetic moment, if any, must be smaller than  $\frac{1}{7000}$  magneton. It has no detectable effects in the free state except the momentum recoil of the initial nucleus. The data of the automatic Wilson cloud chamber experiments with  $^{38}\text{Cl}$ , conducted by Crane and Halpern<sup>7,8</sup>, and Allen<sup>9</sup>, show that the momentum of the recoil nucleus is greater than what it could have received from beta ray alone, thus indicating the existence of a third particle taking part in the disintegration. So the momentum of the recoil nucleus, over and above what it could have received from the beta particle, must be considered to have received from the third particle, the neutrino.

#### 4. Beta Decay & the Theory of Allowed Transitions

The phenomenon of beta emission is in many respects analogous to the phenomenon of emission of a light quantum in the transition of an electrically charged particle from a higher to a lower quantum state. A photon is produced in the quantum transition effected by the coupling of the electron in atom. Similarly, an electron (negative or positive) and neutrino or antineutrino are formed by the transition of heavy nuclear particle (nucleon) caused by the coupling of the nucleons with the electron-neutrino field.

Hence, neutrinos or antineutrinos may obey a wave equation similar to Dirac equation, the charge being zero and mass negligible. The solutions of this equation

give values with positive as well as negative energy. All states of negative energy must be assumed to be ordinarily filled in order to avoid difficulties associated with the possibility of transitions from positive to negative energy. In the case of an electron, an empty negative energy state is equivalent to a particle analogous to positron, while in the case of neutrino, the empty negative energy state is equivalent to a particle analogous to antineutrino. Thus a neutrino is emitted along with the electron and an antineutrino along with the positron. Antineutrino has no property such as charge or magnetic moment that distinguishes it from the neutrino except for the difference in connection with positron and electron emissions and the associated theoretical values.

In beta disintegrations, the nuclear charge is changed by one unit while the mass number remains the same. So the phenomenon of beta disintegration can be considered as the transition of the nuclear heavy particle (nucleon) from neutron eigen state to the proton eigen state in the case of the emission of electron and neutrino or from proton eigen state to the neutron eigen state in the case of the emission of positron and antineutrino which, when symbolically expressed, has the forms :

$${}_0n^1 \longrightarrow {}_1P^1 + e^- + \overset{\rightarrow}{\nu}_0 \quad (7)$$

$${}_1P^1 \longrightarrow {}_0n^1 + e^+ + \overset{\leftarrow}{\nu}_0 \quad (8)$$

Following the lines of analogy outlined above, Fermi<sup>10</sup> developed a theory of beta disintegration on the basis of an analysis of interaction between neutrons and protons, and electrons and neutrinos. The theory calculates the disintegration probability and predicts the shape of the continuous spectrum in which the two light particles, electron and neutrino or positron and antineutrino, share the available energy.

The calculations of the theory are rendered easier by simplifying assumptions in which the wave functions of light particles are considered as constant over the whole nucleus and equal to their value at the nuclear radius  $R_0$ , the velocity of the heavy particles is assumed as negligible in comparison with the velocity of light, and the forces of interaction are treated as those of short ranges and, therefore, the interaction as one depending mainly on the values of the wave functions in the neighbourhood of nucleons.

Now, to proceed into the actual working of the theory of allowed transitions, consider two light particles, an electron and a neutrino or a positron and an antineutrino. Let the wave function of the electron be denoted by  $\phi$  and that of neutrino by  $\psi$  and  $X$ - be co-ordinates of both particles. Then the interaction will be, in general, a function of

$$\phi(x) \frac{d}{dt} \phi(x) \frac{d^2}{dt^2} \phi(x), \text{ etc., and}$$

$$\psi(x) \frac{d}{dt} \psi(x) \frac{d^2}{dt^2} \psi(x), \text{ etc.} \quad (9)$$

As the simplest hypothesis, the interaction may be assumed as one which depends on the wave functions of the light particles but not on their derivatives and, on this assumption, the Hamiltonian of this interaction may be expressed in the form :

$$H \sim g \int \Phi^*(x) \Psi(x) \phi^*(x) \psi(x) \quad (10)$$

where  $g$  is the constant of coupling between the nucleons and electron-neutrino field and  $\Phi$  and  $\Psi$  are the wave functions of proton and neutron respectively.

The forms of experimentally obtained continuous energy spectra are found to have a better fit with the calculated distribution curves if the mass of neutrino be assumed to be vanishingly small, i.e. of the order of zero compared with the mass of an electron. Under the simplifying assumptions mentioned and the condition that  $\mu \sim 0$ , the transition probability  $P_{(E)}$  in which an electron is emitted with an energy between  $E$  and  $E+dE$  is given by

$$P_{(E)} dE = G^2 |\chi|^2 E \sqrt{E^2 - 1} (E_0 - E)^2 dE \quad (11)$$

where  $E_0$  is the total available energy in units of  $MC^2$ ,

$$\chi = \int \Phi \Psi dW,$$

$\Phi$  and  $\Psi$  being the wave functions for proton and neutron respectively and the integration being a summation over all possible spins and directions of the nucleons in the field of nuclear forces,

$$G = g \frac{m^2 C}{\sqrt{2\pi \hbar^3}}$$

is the universal constant indicating the forces of interaction and

$$E \sqrt{E^2 - 1} (E_0 - E)^2 dE$$

is the statistical factor of the transition probability. The expression (11) is a good approximation for  $Z \sim O$ . In heavier elements, the influence of Coulomb field is appreciable

and for these elements the allowed transition probability expression (11), however, will be of the form :

$$P_{(E)}dE = G^2 \chi^2 F_{(Z,E)} E \sqrt{E^2 - 1} \times [E_0 - E]^2 dE \quad (12)$$

where  $F_{(Z,E)}$  is a factor which determines the influence of Coulomb field on the emission of electrons. This factor is given by

$$F_{(Z,E)} = \frac{4}{[\Gamma(1+2S)]^2} \times (2\sqrt{E^2 - 1}R_0)^{2S-2} e^{\frac{\pi\alpha ZE}{\sqrt{E^2 - 1}}} \times \left[ \Gamma \left( S + i \frac{\alpha ZE}{\sqrt{E^2 - 1}} \right) \right]^2 \frac{1+S}{2} \quad (13)$$

where  $S = \sqrt{1 - \alpha^2 Z^2}$ , the fine structure constant  $\alpha = \frac{1}{137}$ ,  $R_0$  is the nuclear radius and  $\Gamma$  is gamma function. The allowed transition probability expression (12) did not agree with the available experimental data in that it did not predict as much electron density at low energies as an experimentally obtained spectrum actually indicates. The discrepancy between theory and experiment could be minimized if the theory would be modified so as to permit a higher probability for the emission of high energy neutrinos. Konopinski and Uhlenbeck<sup>11</sup> found a way to effect this modification by showing that although Fermi had made the most plausible assumption concerning the interaction of electron and neutrino with the nucleus, other assumptions were nevertheless possible which could lead to different probability expressions. They worked the theory for several different types of interaction and found that one, in particular, in which not only the eigen functions of electron and neutrino but also their derivatives with respect to co-ordinates were introduced into the expression for interaction, led to a modified form of probability expression. This new expression which seemed to have agreed with the available experimental data, differed from that of Fermi in that the factor  $(E_0 - E)^4$  appeared in the place of  $(E_0 - E)^2$ . The fourth power of neutrino as given by Konopinski-Uhlenbeck's expression instead of the second power as in (12) increases the probability of fast neutrinos and slow electrons and shifts the most probable electron energy approximately to  $E_0/3$  for large  $E_0$ .

From the considerations outlined above, it is obvious that the energy spectrum of electrons is given by

$$N_{(E)}dE \sim E \sqrt{E^2 - 1} (E_0 - E)^2 dE \quad (14)$$

$$\text{and } N_{(E)}dE \sim E \sqrt{E^2 - 1} (E_0 - E)^4 dE \quad (15)$$

in accordance with the Fermi allowed transition probability and the Konopinski-Uhlenbeck modification respectively. In these expressions  $N_{(E)}dE$  are relative numbers of particles in equal intervals of energy. From the allowed transition probability expression (12) and the energy spectrum expressions (14) and (15), it follows that a plot

$$\left[ \frac{N_{(E)} dE}{P^2 F_{(Z,E)}} \right]^{\frac{1}{k}}$$

against energy  $E$  should yield a straight line with its intercept with energy axis representing the maximum available energy  $E_0$ , if the Fermi theory ( $K=2$ ) or Konopinski's theory ( $K=4$ ) is correct.

Early experimental data of certain continuous spectra appeared to satisfy Konopinski-Uhlenbeck allowed transition probability equation better than that of Fermi. But results of subsequent measurements carefully carried out with improved experimental technique had shown that Konopinski-Uhlenbeck modification was instigated by the influence of scattering of electrons caused by the thickness of the source and improper source mounting technique.

### 5. Allowed & Forbidden Transitions

Owing to the simplifying assumptions already dealt with, the matrix element  $\chi = \int \Phi \Psi dw$  is produced in the disintegration probability expression (12). This element will be near unity if the wave functions of the nucleons in the initial and final states are alike or very much alike. If  $\chi \sim 1$  or  $\chi \neq 0$ , the spin change at the disintegration  $\Delta I = 0$ . This condition constitutes the Fermi selection rule for the allowed transition probability, the theory of which is briefly outlined in the last section (section 4).

If the matrix element  $\chi$  vanishes for the transition ( $\chi = 0$ ) which occurs most usually with the change of total angular momentum, the transition will be forbidden. The forbidden transitions of the first, second and third orders are distinguished according to changes of  $I$  by  $\Delta I = 1, 2, 3 \dots$ . These forbidden transitions are comparatively much less probable than the allowed transitions with  $\Delta I = 0$ . The probability of forbidden

transitions of higher order decreases by a factor of approximately

$$\frac{R_o}{\hbar/mC} = \frac{4 \times 10^{-13}}{3.86 \times 10^{-11}} \sim \frac{1}{100}$$

per order. A forbidden transition is characterized by relatively long life, and by an energy distribution curve other than that for the allowed Fermi distribution. Fermi made approximate calculation and this calculation indicates that the energy distribution curve at low energies should be at a position beneath the allowed Fermi curve in the case of forbidden transitions. This indication, however, could not be reconciled with some of the available experimental data<sup>12</sup>.

**6. Life-time & Beta-disintegration Energy**

The theory of beta decay indicates that there is a relation between life-time  $\tau$  for beta-ray emission and maximum disintegration energy  $E_o$  available for electron and neutrino, since the reciprocal of the life-time is the total transition probability. This quantity may, therefore, be derived from the transition probability which is obtained by integrating the energy distribution equation (10) thus :

$$\frac{1}{\tau} = G^2 |\chi|^2 f_{(Z,E_o)} \tag{16}$$

where  $G$  is a constant denoting the strength of interaction between the heavy particles and light particles,  $\chi$  is the matrix element for the transition and  $f_{(Z,E_o)}$  is the integral of the energy distribution given by

$$f_{(Z,E_o)} = \int_1^{E_o} F_{(Z,E)} E \sqrt{E^2 - 1} (E_o - E)^2 dE \tag{17}$$

$E_o$  being in units of electron rest mass.

The reciprocal of the life-time  $\frac{1}{\tau}$  for beta emission is identical with beta disintegration constant  $\lambda$ .

If  $F_{(Z,E)} = 1$ , and this is certainly valid for all light elements, the disintegration constant  $\lambda$  may be expressed in this form :

$$\lambda = G^2 |\chi|^2 \int_1^{E_o} E \sqrt{E^2 - 1} (E_o - E)^2 dE \tag{18}$$

From the expressions (16) and (17) it is obvious that  $f_{(Z,E_o)}$  varies rapidly with  $E_o$ , and thus the life-time  $\tau$  of the beta-disintegrating element decreases rapidly with increasing kinetic energy of the particle, so that for small kinetic energy of the beta particles when  $E \sqrt{E^2 - 1} \sim E$

$$\frac{1}{\tau} = E^5 \tag{19}$$

and for large kinetic energy when  $E \sqrt{E^2 - 1} \sim E^2$

$$\frac{1}{\tau} = E^7 \tag{19a}$$

while for much higher kinetic energies when  $E \sqrt{E^2 - 1} \gg E^2$

$$\frac{1}{\tau} = E^9 \tag{19b}$$

$\frac{1}{\tau}$  and  $\lambda$ , as indicated by (16), (17) and (18), are identical. Therefore, from (19), (19a) and (19b),  $\lambda$  may be expressed in terms of beta-disintegration energy  $E_o$  by

$$\log \lambda = \log \frac{1}{\tau} = K \cdot \log E_o \tag{19c}$$

where  $E_o$  is the beta-disintegration energy in units of  $MC^2$  obtained from the value of the upper end point of the energy distribution curve and  $K=5$  or  $7$ , etc., according to the range of energy and to the nature and order of transition. The group of beta-decay processes to which the constant  $K=5$  holds good is interpreted as corresponding to allowed transitions while the second and the third groups to which the constant  $K=7$  and  $K=9$  hold are interpreted as corresponding to forbidden transitions of the first and the second orders respectively. The classification of beta-decay processes into two or three groups, each group belonging to allowed or one of various forbidden transitions, has been made by Sargent<sup>13</sup> on purely empirical basis which will be dealt in section 7.

**7. Sargent Diagrams & Classification of Beta-decaying Processes**

The Fermi theory of beta decay, as shown in the previous two sections, 5 and 6, has led to a classification of the beta-decay processes into allowed and forbidden transitions, and the forbidden into different orders. This theory also implies the relation between beta-disintegration energy  $E_o$  and its decay constant  $\lambda$  and thus offers, so to speak, an interpretation to the well-known Sargent empirical rule which, in a modified form, is similar to the theoretically obtained relation expressed by (19c).

Sargent found that plots of the logarithms of half lives or of the disintegration constants versus logarithm of the maximum disintegration energies  $E_o$  of the beta-decaying isotopes such as those given in Appendix I give different orders of curves

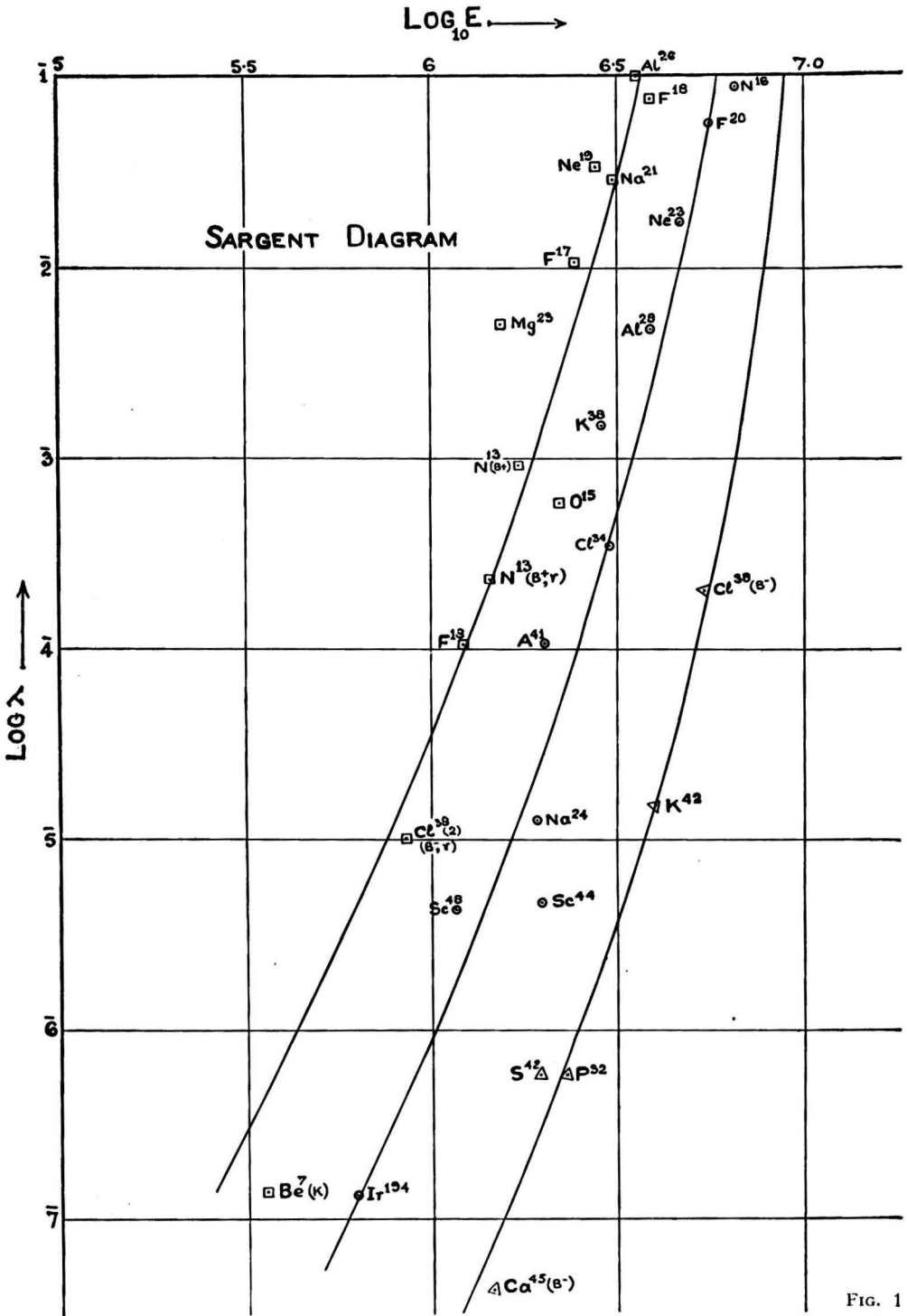


FIG. 1



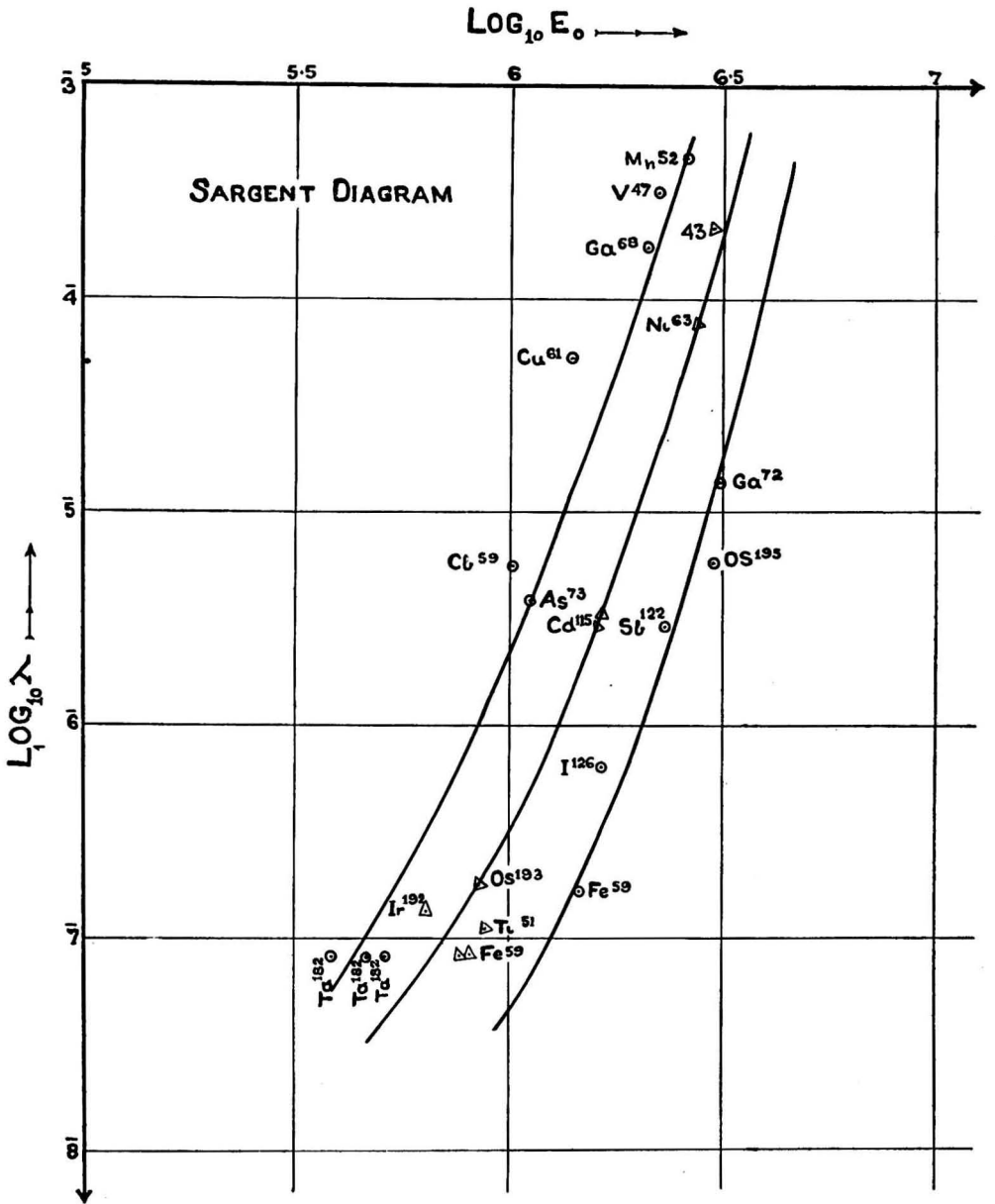


FIG. 2

as shown in Figs. 1, 2 and 3. In these three figures, the curves which represent the group of isotopes for which the constant K is a minimum, are curves of the group of isotopes of allowed transition while the other curves in each figure are of the groups of isotopes

of forbidden transitions. Thus, Sargent diagrams form an empirically obtained powerful graphical method of classifying the isotopes into allowed and forbidden transitions and those of forbidden transitions into different orders.

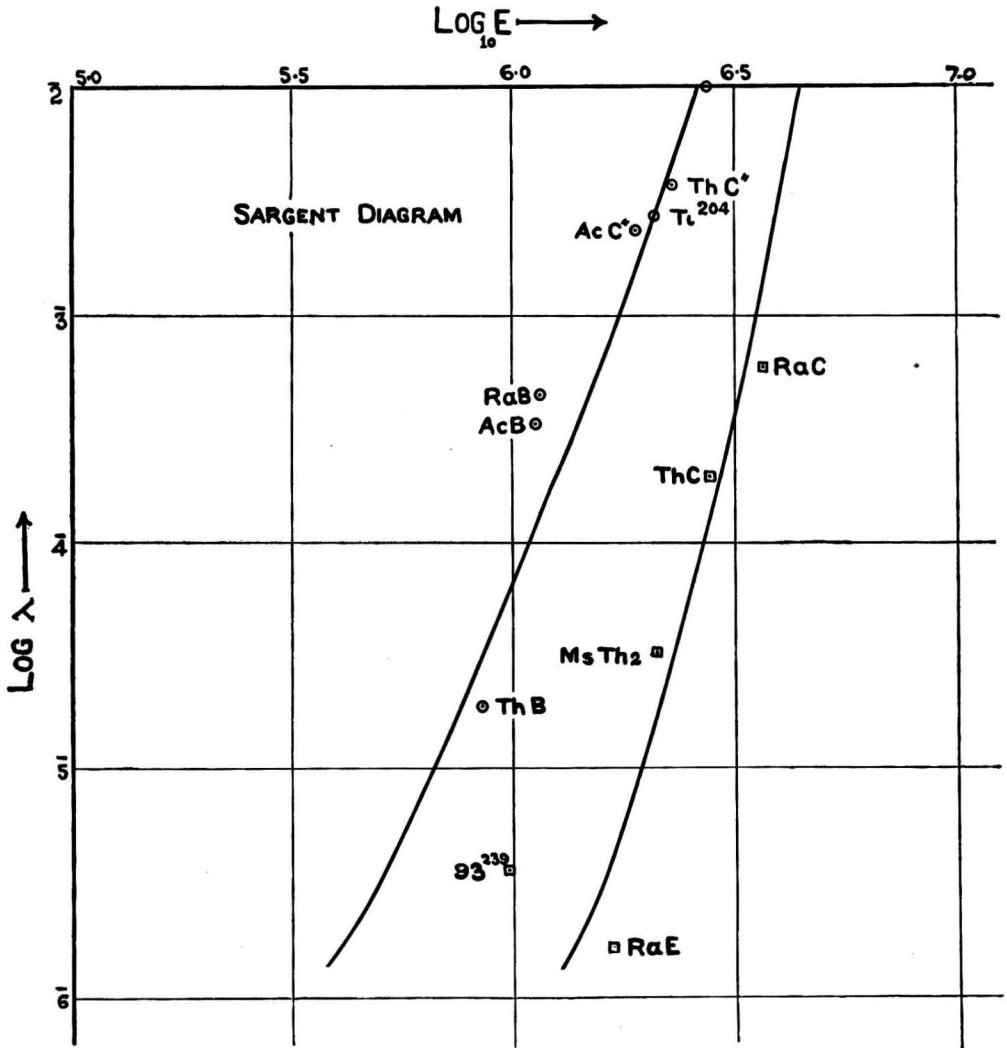


FIG. 3

8. Meson Theory & Beta Decay

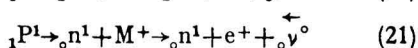
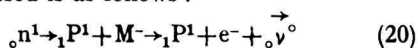
The matrix element  $\chi$  for allowed transition is of the order of unity and so the expression (18) for the disintegration constant  $\lambda$  readily gives the value of G. From the mean value so obtained, the coupling constant g may be found to be of the order of  $4 \times 10^{-50}$  cm.<sup>3</sup> Erg. The implication of this low value of the coupling constant is that Fermi field of forces would by far be too small to hold the nucleons together.

To surmount this difficulty, Yukawa<sup>14</sup> assumed that the force acting between

nucleons might be described by a meson field surrounding the one which acts on the other just as the Coulomb force between charged particles can be expressed in terms of the interaction of these particles with the electro-magnetic field. The quanta which surround a nucleon are mesons and if these are treated as having a finite mass m, the range of forces resulting from the meson field will be  $\hbar/mc$ , the Compton wavelength for the meson. From experiments on scattering of neutrons by protons, the range of forces is found to be of the order of  $2 \times 10^{-13}$  cm.

With this range of forces, the meson rest mass should be of the order of 200 electron masses. Particles having this order of rest mass were discovered in cosmic rays. This discovery confirmed the required range of nuclear forces as produced by the Yukawa scalar meson theory describing the nature of the meson field.

The meson is ascribed an integral spin, and as assumed and subsequently confirmed by its discovery in cosmic rays, it is either negative or positive or neutral. The field, though virtual in its nature, may be described by the meson particle which has, so to say, its formation at a beta disintegration as an intermediary between this disintegration and the emission of light particles. The meson is capable of exchange between a neutron and proton or vice versa between a proton and neutron or any pair of nuclear heavy particles depending upon whether the meson is negative or positive or neutral. It has a very short half-life period of the order of a micro-second and disintegrates into light particles, negatron and neutrino or positron and antineutrino. Thus the interaction symbolically expressed is as follows :



where  ${}_0n^1$ ,  ${}_1P^1$ ,  $M^-$ ,  $M^+$ ,  $e^-$ ,  $e^+$ ,  $\overset{\rightarrow}{\nu}^0$  and  $\overset{\leftarrow}{\nu}^0$  are neutron, proton, negative meson, positive meson, negatron, positron, neutrino and antineutrino respectively.

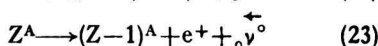
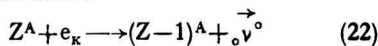
The meson may be described by a scalar function if it be of zero spin and by a vector function if it be of spin one. The scalar and vector meson theories predict distribution of the Fermi type.

**9. Inversion**

The phenomenon of inversion involves two variations of the beta process : either an electron of the extra-nuclear configuration, very usually a K-electron, of the atom may be captured by the nucleus with the emission of a neutrino, or an electron in negative energy state may be absorbed by the nucleus with the vacancy so caused being equivalent to a positron accompanied by antineutrino. In both these variations of the beta process, the nuclear charge is reduced by one unit.

In a nucleus which is unstable with respect to its lower isobar, a nucleon from a proton eigen state changes into a neutron eigen state by emitting a neutrino or an antineutrino

and absorbing an electron (a K-electron) or emitting a positron respectively. These variations of the beta process may be represented thus :



where  $Z$ ,  $A$ ,  $e_K$ ,  $e^+$ ,  $\overset{\rightarrow}{\nu}^0$  and  $\overset{\leftarrow}{\nu}^0$  are atomic number, mass number, K-electron, positron, neutrino and antineutrino respectively.

It is of great importance to determine the relative probability of these two processes taking place in this type of radioactive nuclei. In the process given by (22), let the energy released be denoted by  $E$ , the available energy in the process by  $E_0$  and the ionizing energy of a K-electron by  $E_K$ . Then assuming statistical conservation of energy,  $E = E_0 - E_K$ . The process, represented by (23), being the absorption of an electron in negative energy state, is analogous to the process represented by (22).

The probability of the absorption of an electron is proportional to the probability density of the electron in the neighbourhood of the nucleus, i.e. to  $|\phi|^2$ ,  $\phi$  being the wave function of the electron, and to the statistical weight of the final state which is proportional to the square of the energy of neutrino, assuming its rest mass is zero (negligibly small). Hence the capture probability  $P_K$  may be written as

$$P_K = A E_\nu^3 |\phi|^2 \quad (24)$$

where  $A$  is a constant depending upon the kind of transition and  $E_\nu$  the neutrino energy. In the process of K-electron capture, if  $Z$  is small, the wave function of the K-electron may be expressed by

$$\phi_K = \frac{Z^3}{\pi a^3} e^{-\frac{Zr}{a}} e^{-\alpha_K} \quad (25)$$

where the Bohr radius for K-electron

$$a_K = \frac{h^2}{4\pi^2 m e^2} = \frac{\hbar^2}{m e^2}$$

The completed K-level has two electrons and

$$|\phi|^2 = \frac{Z^3}{\pi a^3}$$

at the centre. Hence the expression (24) for the probability of the K-electron capture may be written in the form :

$$P_K = \frac{2AZ^3 E_\nu^2}{\pi a^3} \quad (26)$$

Now, to determine the probability of positron emission, consider an electron in negative energy state. There are two of them for each cell of volume  $h^3 = 8\pi^3 \hbar^3$  in a phase space. Hence, for positron emission, the probability of finding an electron in negative energy state per unit volume of momentum space is

$$\frac{2}{8\pi^3 \hbar^3} = \frac{1}{4\pi^3 \hbar^3}$$

Therefore, the probability of an electron in negative energy state being absorbed by the nucleus which is equivalent to the probability of the emission of a positron in the momentum range between  $P_x$  and  $P_x + dp_x$ ,  $P_y$  and  $P_y + dp_y$  and  $P_z$  and  $P_z + dp_z$  is given by

$$P_{\beta+} = \frac{AE_\nu^2}{4\pi^3 \hbar^3} dp_x dp_y dp_z \quad (27)$$

The total probability for positron emission may be obtained by integrating from  $p_x = p_y = p_z = p_0$  to  $E_\nu = 0$ . Since an energy of  $2mC^2$  is required for an electron in negative kinetic energy state to be removed, the energy of neutrino may be expressed in relativistic units by

$$E_\nu = E_0 - mC^2 - C\sqrt{p^2 + m^2C^2},$$

where  $p^2 = p_x^2 + p_y^2 + p_z^2$ . Consider  $\int dp_x dp_y dp_z = 4\pi p^2 dp$  and let  $p_0 = C\sqrt{mC^2 + p^2} = E_0 - mC^2$ . Then the ratio of probabilities of a K-electron capture and positron emission is

$$\frac{P_K}{P_{\beta+}} = 2\pi \left( \frac{2\hbar}{\alpha_K} \right)^3 \frac{E_\nu^2}{\int_0^{p_0} E_\nu^2 p^2 dp} \quad (28)$$

Since  $\frac{\hbar}{\alpha} = \frac{e^2}{\hbar C} mC = \frac{1}{137} mC$  the expression (28) becomes:

$$\frac{P_K}{P_{\beta+}} = 2\pi (Z\alpha)^3 \frac{E_\nu^2 m^3 C^3}{\int_0^{p_0} E_\nu^2 p^2 dp} \quad (29)$$

Thus (28) or (29) represents the Fermi expression for the ratio of probabilities of the K-electron capture and positron emission.

In the Konopinski-Uhlenbeck theory of inversion, the probability of electron capture contains the factor  $|\text{grad } \phi|^2$  instead of the factor  $|\phi|^2$ . With electrons in negative energy state, this factor introduces  $\frac{p^2}{\hbar^2}$  and with K-electrons a factor  $\frac{z^2}{\alpha^2}$ , so that the

ratio of probabilities of the K-electron capture and positron emission is

$$\frac{P_K}{P_{\beta+}} = 2\pi (Z\alpha)^5 \frac{E_\nu^2 m^5 C^5}{\int_0^{p_0} E_\nu^2 p^4 dp} \quad (30)$$

If the isotopes under consideration be of large  $Z$ , a correction factor arising from the effect of Coulomb field on the wave functions must be introduced in the Fermi expressions (28) and (29) and likewise in the Konopinski-Uhlenbeck expression (30) for the ratio of probabilities of K-electron capture and positron emission.

These theories of inversion for allowed transition, outlined above, give widely differing values for the ratio of probabilities of K-electron capture and positron emission for this type of radioactive nuclei.

In a nucleus which is unstable to its immediately lower isobar, if there is not sufficient energy to cause positron emission, an electron of the K-shell is absorbed by the nucleus and the K-shell will subsequently be filled giving rise to the emission of an X-ray quantum. This quantum is characteristic of the K-radiation of an atom whose number is one below that of the original one, inasmuch as the capture of negative charge reduces the nuclear charge and, therefore, the atomic number by one unit. This characteristic quantum is the distinguishing external effect produced by K-electron capture phenomenon and was the first evidence of this phenomenon.

This phenomenon was observed by Alvarez<sup>15</sup> and subsequently also by others<sup>16</sup>. Alvarez found that  ${}_{22}\text{Ti}^{48}$  bombarded by deuterons emitted a radiation whose absorption coefficient agreed with that of titanium K-characteristic radiation. The active isotope produced by the Ti-d-n reaction was an isotope of vanadium  ${}_{23}\text{Va}^{49}$ , the half life of which was 600 days<sup>17</sup>, while the radiation emitted was titanium K-characteristic X-radiation.

It was found that vanadium<sup>49</sup> was only a K-electron capture disintegrating isotope without positron emission. It was, however, found that there were a few isotopes disintegrating both by K-electron capture and positron emission. An experimental study of this type of active isotopes could prove a touch-stone to test the validity of the two theories of inversion which give very widely diverging values. Hence a measurement of the ratio of probabilities of K-electron capture processes and positron emission processes

of cadmium<sup>107</sup> (<sup>109</sup> <sup>2</sup>), the half life of which was 6.7 hours, was undertaken by a number of workers<sup>18</sup>. It was found that this isotope, which was disintegrating both by K-electron capture and positron emission, was of an allowed transition probability and that the ratio of K-electron processes to positron emission processes was

$$\frac{P_K}{P_{\beta^+} \text{ (observed)}} = 320 \pm 20$$

This observed value, which is in good agreement with the value predicted by the Fermi theory given by

$$\frac{P_K}{P_{\beta^+} \text{ (Fermi)}} = 342,$$

is at great variance with the value

$$\frac{P_K}{P_{\beta^+} \text{ (K.U.)}} = 19800$$

given by the Konopinski-Uhlenbeck theory, which, therefore, is ruled out.

The author wishes to record his appreciation of the active co-operation of his scientific assistants, U. C. Gupta, A. Sagar, P. N. Sundaram, R. Sundaram and S. K. Suri, who helped him in compiling and calculating the data given in Appendix I,

and plotting a number of Sargent diagrams, each covering a wide range of beta-decaying isotopes.

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## APPENDIX I

### BETA-DECAYING ISOTOPES

(See Section 7, Page 403)

| ISOTOPE   | SYMBOL | Z  | A  | HALF-LIFE<br>(Sec.) | DECAY CONST.<br>(Sec. <sup>-1</sup> ) | ENERGY<br>MEV. | RADIATION          |
|-----------|--------|----|----|---------------------|---------------------------------------|----------------|--------------------|
| Hydrogen  | H      | 1  | 3  | 10 <sup>9</sup>     | 6.9 × 10 <sup>-10</sup>               | 0.015          | β <sup>-</sup>     |
| Helium    | He     | 2  | 6  | 0.8                 | 8.622 × 10 <sup>-1</sup>              | 3.7            | β <sup>-</sup>     |
| Lithium   | Li     | 3  | 8  | 0.88                | 7.840 × 10 <sup>-1</sup>              | 12.0           | β <sup>-</sup>     |
| Beryllium | Be(1)  | 4  | 7  | 5 × 10 <sup>6</sup> | 1.380 × 10 <sup>-7</sup>              | ...            | K                  |
|           | Be(2)  | .. | 7  | 5 × 10 <sup>7</sup> | 1.380 × 10 <sup>-8</sup>              | ...            | K, γ               |
|           | Be     | .. | 10 | 10 <sup>13</sup>    | 6.902 × 10 <sup>-14</sup>             | 0.5            | β <sup>-</sup> , γ |
| Boron     | B      | 5  | 12 | 0.022               | 31.36                                 | 12.0           | β <sup>-</sup>     |
| Carbon    | C      | 6  | 10 | 8.8                 | 7.84 × 10 <sup>-2</sup>               | 3.4            | β <sup>+</sup>     |
|           | C      | .. | 11 | 1230                | 5.611 × 10 <sup>-5</sup>              | 0.95           | β <sup>+</sup>     |
|           | C      | .. | 14 | > 10 <sup>12</sup>  | ~ 6.9 × 10 <sup>-13</sup>             | 0.145          | β <sup>-</sup>     |
| Nitrogen  | N      | 7  | 13 | 3000(?)             | 2.3 × 10 <sup>-4</sup>                | ...            | β <sup>+</sup> , γ |
|           | N      | .. | 16 | 8                   | 8.628 × 10 <sup>-2</sup>              | 6.0(?)         | β <sup>-</sup>     |
| Oxygen    | O      | 8  | 15 | 126                 | 5.321 × 10 <sup>-3</sup>              | ...            | β <sup>+</sup>     |
|           | O      | .. | 19 | 31                  | 2.22 × 10 <sup>-2</sup>               | ...            | β <sup>-</sup>     |

APPENDIX I — BETA-DECAYING ISOTOPES — *continued*

| ISOTOPE    | SYMBOL   | Z  | A      | HALF-LIFE<br>(Sec.)  | DECAY CONST.<br>(Sec. <sup>-1</sup> ) | ENERGY<br>MEV.         | RADIATION             |
|------------|----------|----|--------|----------------------|---------------------------------------|------------------------|-----------------------|
| Fluorine   | F        | 9  | 17     | 70                   | $9.86 \times 10^{-3}$                 | 2.1                    | $\beta^+$             |
|            | F        | "  | 18     | 6720                 | $1.027 \times 10^{-4}$                | 0.7                    | $\beta^+$             |
|            | F        | "  | 20     | 12                   | $5.749 \times 10^{-2}$                | 5.0                    | $\beta^-, \gamma$     |
| Neon       | Ne       | 10 | 19     | 20.3                 | $3.399 \times 10^{-2}$                | 2.20                   | $\beta^+$             |
|            | Ne       | "  | 23     | 40                   | $1.723 \times 10^{-2}$                | 4.1                    | $\beta^-$             |
| Sodium     | Na       | 11 | 21     | 23                   | $3.000 \times 10^{-2}$                | ...                    | ...                   |
|            | Na       | "  | 22     | $> 10^8$             | $6.897 \times 10^{-9}$                | 0.58                   | $\beta^+, \gamma$     |
|            | Na       | "  | 24     | $5.3 \times 10^4$    | $1.302 \times 10^{-5}$                | 1.4                    | $\beta^-, \gamma$     |
|            | Na       | "  | 25     | 62                   | $1.11 \times 10^{-2}$                 | 2.8                    | $\beta^-, \gamma$     |
| Magnesium  | Mg       | 12 | 23     | 11.6                 | $5.950 \times 10^{-2}$                | 2.82                   | $\beta^+$             |
|            | Mg       | "  | 27     | 612                  | $1.127 \times 10^{-3}$                | 1.8                    | $\beta^-, \gamma$     |
| Aluminium  | Al       | 13 | 26     | 7.0                  | $9.860 \times 10^{-2}$                | 2.99                   | $\beta^+$             |
|            | Al       | "  | 28     | 144                  | $4.791 \times 10^{-3}$                | 3.3                    | $\beta^-, \gamma$     |
|            | Al       | "  | 29     | 402                  | $1.716 \times 10^{-3}$                | 2.5                    | $\beta^-$             |
| Silicon    | Si       | 14 | 27     | 4.92                 | $1.409 \times 10^{-1}$                | 3.74                   | $\beta^+$             |
|            | Si       | "  | 31     | 10200                | $6.9 \times 10^{-4}$                  | 1.8                    | $\beta^-$             |
| Phosphorus | P        | 15 | 29     | 4.6                  | $1.500 \times 10^{-1}$                | 3.63                   | $\beta^+$             |
|            | P        | "  | 30     | 153                  | $4.50 \times 10^{-3}$                 | 3.0                    | $\beta^+$             |
|            | P        | "  | 32     | $1.2 \times 10^6$    | $5.751 \times 10^{-7}$                | 1.69                   | $\beta^-$             |
| Sulphur    | S        | 16 | 31     | 3.18                 | $2.156 \times 10^{-1}$                | 3.90                   | $\beta^+$             |
|            | S        | "  | 35     | $7.52 \times 10^6$   | $9.075 \times 10^{-8}$                | 0.107                  | $\beta^-$             |
| Chlorine   | Cl       | 17 | 33     | 2.8                  | $2.46 \times 10^{-1}$                 | ...                    | $\beta^+$             |
|            | Cl       | "  | 34     | 1980                 | $3.484 \times 10^{-4}$                | 2.5                    | $\beta^+$             |
|            | Cl       | "  | 36     | $> 10^8$             | $6.902 \times 10^{-9}$                | 0.66                   | $\beta^+; K, \beta^-$ |
|            | Cl       | "  | 38     | 2220                 | $3.136 \times 10^{-4}$                | 1.1, 5.0               | $\beta^-, \gamma$     |
| Argon      | A        | 18 | 35     | 1.91                 | $3.63 \times 10^{-1}$                 | 4.38                   | $\beta^+$             |
|            | A        | "  | 39     | 240                  | $2.874 \times 10^{-3}$                | ...                    | $\beta^-$             |
|            | A        | "  | 41     | 6600                 | $1.046 \times 10^{-4}$                | 1.5                    | $\beta^-, \gamma$     |
| Potassium  | K        | 19 | 38     | 462                  | $1.494 \times 10^{-3}$                | 2.3                    | $\beta^+$             |
|            | K        | "  | 40     | $4.5 \times 10^{16}$ | $1.534 \times 10^{-17}$               | 0.40; 0.70             | $\beta^-$             |
|            | K        | "  | 42     | $4.5 \times 10^4$    | $1.534 \times 10^{-5}$                | 3.5                    | $\beta^-$             |
|            | K        | "  | 43, 44 | 1080                 | $6.388 \times 10^{-4}$                | ...                    | $\beta^-$             |
| Calcium    | Ca       | 20 | 39     | 270                  | $2.555 \times 10^{-3}$                | ...                    | $\beta^+$             |
|            | Ca       | "  | 41     | $7.34 \times 10^5$   | $9.4 \times 10^{-6}$                  | ...                    | K, $\gamma, e^-$      |
|            | Ca       | "  | 45     | $1.55 \times 10^7$   | $4.45 \times 10^{-8}$                 | 0.2                    | $\beta^-, \gamma$     |
|            | Ca       | "  | 49     | $9 \times 10^3$      | $7.66 \times 10^{-5}$                 | 2.3                    | $\beta^-, \gamma$     |
|            | Ca       | "  | 49     | 1800                 | $3.83 \times 10^{-4}$                 | ...                    | $\beta^-$             |
| Scandium   | Sc       | 21 | 41     | 0.87                 | $7.93 \times 10^{-1}$                 | 4.94                   | $\beta^+$             |
|            | Sc       | "  | 42     | $1.17 \times 10^6$   | $5.897 \times 10^{-7}$                | 1.4                    | $\beta^+$             |
|            | Sc       | "  | 43     | $1.44 \times 10^4$   | $4.791 \times 10^{-5}$                | 0.4; 1.4               | $\beta^+, \gamma$     |
|            | Sc       | "  | 44     | $1.87 \times 10^5$   | $3.689 \times 10^{-6}$                | ...                    | $e^-, \gamma$         |
|            | Sc       | "  | 44     | $1.48 \times 10^4$   | $4.67 \times 10^{-5}$                 | 1.45                   | $\beta^+, \gamma$     |
|            | Sc       | "  | 46     | $7.34 \times 10^6$   | $9.4 \times 10^{-7}$                  | 0.26; 1.5              | $\beta^-, \gamma, K$  |
|            | Sc       | "  | 47     | $2.27 \times 10^8$   | $3.039 \times 10^{-6}$                | 1.1                    | $\beta^-, \gamma$     |
|            | Sc       | "  | 48     | $1.58 \times 10^5$   | $4.34 \times 10^{-6}$                 | 0.64                   | $\beta^-, \gamma$     |
|            | Sc       | "  | 49     | $3.42 \times 10^3$   | $1.480 \times 10^{-4}$                | 1.8                    | $\beta^-$             |
|            | Sc       | "  | ?      | $2.94 \times 10^5$   | $2.346 \times 10^{-6}$                | 0.46                   | $\beta^-$             |
|            | Titanium | Ti | 22     | 45                   | $1.1 \times 10^4$                     | $6.275 \times 10^{-5}$ | 1.2                   |
| Ti         |          | "  | 51     | 174                  | $3.965 \times 10^{-3}$                | ...                    | $\beta^-, \gamma$     |
| Ti         |          | "  | 51     | $6.2 \times 10^6$    | $1.113 \times 10^{-7}$                | 0.36                   | $\beta^+$             |
| Vanadium   | V        | 23 | 47     | 1980                 | $3.487 \times 10^{-4}$                | 1.9                    | $\beta^+$             |
|            | V        | "  | 48     | $> 1.4 \times 10^6$  | $< 4.928 \times 10^{-7}$              | 1.0                    | $\beta^+, K, \gamma$  |
|            | V        | "  | 49     | $5.18 \times 10^7$   | $1.33 \times 10^{-8}$                 | ...                    | K                     |
|            | V        | "  | 50     | $1.33 \times 10^4$   | $5.187 \times 10^{-5}$                | ...                    | $\beta^+$             |
|            | V        | "  | 52     | 234                  | $2.968 \times 10^{-3}$                | 2.05                   | $\beta^-$             |
| Chromium   | Cr       | 24 | 49     | 2514                 | $2.748 \times 10^{-4}$                | 1.45                   | $\beta^+, \gamma$     |
|            | Cr       | "  | 51     | $2.29 \times 10^6$   | $3.01 \times 10^{-7}$                 | ...                    | K, $\gamma, e^-$      |
| Manganese  | Mn       | 25 | 51     | 2760                 | $2.50 \times 10^{-4}$                 | 2.0                    | $\beta^+$             |
|            | Mn       | "  | 52     | 1260                 | $5.321 \times 10^{-4}$                | 2.2                    | $\beta^+, \gamma$     |

APPENDIX I — BETA-DECAYING ISOTOPES — *continued*

| ISOTOPE   | SYMBOL | Z  | A      | HALF-LIFE<br>(Sec.)     | DECAY CONST.<br>(Sec. <sup>-1</sup> ) | ENERGY<br>MEV.                           | RADIATION                        |
|-----------|--------|----|--------|-------------------------|---------------------------------------|------------------------------------------|----------------------------------|
| Manganese | Mn     | 25 | 52     | $5.62 \times 10^5$      | $1.23 \times 10^{-6}$                 | 0.77                                     | $\beta^+$ , $\gamma$             |
|           | Mn     | "  | 54     | $2.68 \times 10^7$      | $2.574 \times 10^{-8}$                | ...                                      | K, $\gamma$                      |
|           | Mn     | "  | 56     | $9.32 \times 10^3$      | $7.367 \times 10^{-4}$                | 0.75 ; 2.86 ; 1.09                       | $\beta^-$ , $\gamma$             |
| Iron      | Fe     | 26 | 53     | 534                     | $1.3108 \times 10^{-3}$               | ...                                      | $\beta^+$                        |
|           | Fe     | "  | 55     | $\sim 1.26 \times 10^8$ | $5.321 \times 10^{-9}$                | ...                                      | K, $e^-$                         |
|           | Fe     | "  | 59     | $3.8 \times 10^6$       | $1.81 \times 10^{-7}$                 | 0.26 ; 0.46                              | $\beta^-$ , $\gamma$             |
| Cobalt    | Co     | 27 | 55     | $6.55 \times 10^6$      | $3.797 \times 10^{-4}$                | 1.50                                     | $\beta^+$ , $\gamma$             |
|           | Co     | "  | 56     | $6.22 \times 10^6$      | $1.109 \times 10^{-7}$                | 1.2                                      | $\beta^+$ , $\gamma$ , K         |
|           | Co     | "  | 57     | $2.33 \times 10^7$      | $2.98 \times 10^{-8}$                 | 2.6                                      | K, $\gamma$ , $e^-$ , $\beta^+$  |
|           | Co     | "  | 58     | $6.221 \times 10^3$     | $1.109 \times 10^{-4}$                | 0.470                                    | $\beta^+$ , $\gamma$             |
|           | Co     | "  | 60     | $1.67 \times 10^8$      | $4.13 \times 10^{-9}$                 | 0.31                                     | $\beta^-$ , $\gamma$             |
|           | Co     | "  | 60     | 640                     | $1.078 \times 10^{-3}$                | 1.3                                      | $\beta^-$ , $\gamma$             |
| Nickel    | Ni     | 28 | 57     | $1.3 \times 10^5$       | $5.342 \times 10^{-6}$                | 0.67                                     | $\beta^+$                        |
|           | Ni     | "  | 59     | $4.72 \times 10^8$      | $1.48 \times 10^{-9}$                 | 0.05                                     | $\beta^+$                        |
|           | Ni     | "  | 63     | 9350                    | $7.379 \times 10^{-5}$                | 1.9                                      | $\beta^-$ , $\gamma$             |
| Copper    | Cu     | 29 | 58, 60 | 81                      | $8.51 \times 10^{-3}$                 | ...                                      | $\beta^+$                        |
|           | Cu     | "  | 61     | $1.3 \times 10^4$       | $5.342 \times 10^{-5}$                | 0.9                                      | $\beta^+$                        |
|           | Cu     | "  | 61     | $1.3 \times 10^4$       | $5.342 \times 10^{-5}$                | ...                                      | K                                |
|           | Cu     | "  | 62     | 63                      | $1.064 \times 10^{-3}$                | 2.6                                      | $\beta^+$                        |
|           | Cu     | "  | 64     | $4.6 \times 10^4$       | $1.501 \times 10^{-5}$                | 0.58( $\beta^-$ ) ;<br>0.66( $\beta^+$ ) | $\beta^+$ or $\beta^-$<br>or K   |
|           | Cu     | "  | 66     | 300                     | $2.301 \times 10^{-3}$                | 2.9                                      | $\beta^-$                        |
| Zinc      | Zn     | 30 | 63     | 2280                    | $3.0026 \times 10^{-4}$               | 2.32                                     | $\beta^+$                        |
|           | Zn     | "  | 65     | $2.16 \times 10^7$      | $3.29 \times 10^{-8}$                 | 0.4( $\beta^+$ )                         | $\beta^+$ , K, $\gamma$ , $e^-$  |
|           | Zn     | "  | 69     | 3420                    | $2.018 \times 10^{-4}$                | 1.0                                      | $\beta^-$                        |
|           | Zn     | "  | 70     | ...                     | ...                                   | ...                                      | ...                              |
| Gallium   | Ga     | 31 | 64     | 2880                    | $2.39 \times 10^{-4}$                 | ...                                      | $\beta^+$                        |
|           | Ga     | "  | 65     | 900                     | $7.66 \times 10^{-3}$                 | ...                                      | K, $e^-$                         |
|           | Ga     | "  | 67     | $2.99 \times 10^5$      | $2.31 \times 10^{-6}$                 | ...                                      | K, $\gamma$ , $e^-$              |
|           | Ga     | "  | 66     | $3.4 \times 10^4$       | $2.29 \times 10^{-5}$                 | 3.1                                      | $\beta^+$                        |
|           | Ga     | "  | 68     | 4080                    | $1.691 \times 10^{-4}$                | 1.9                                      | $\beta^+$                        |
|           | Ga     | "  | 70     | 1140                    | $6.054 \times 10^{-4}$                | 1.70                                     | $\beta^-$ , $\gamma$             |
|           | Ga     | "  | 72     | $5 \times 10^4$         | $1.38 \times 10^{-5}$                 | 2.6                                      | $\beta^-$ , $\gamma$             |
| Germanium | Ge     | 32 | 71     | $1.1 \times 10^5$       | $6.275 \times 10^{-6}$                | 1.2                                      | $\beta^+$                        |
|           | Ge     | "  | 75     | 4860                    | $1.428 \times 10^{-4}$                | 1.1                                      | $\beta^-$                        |
|           | Ge     | "  | 77     | $2.9 \times 10^4$       | $2.380 \times 10^{-5}$                | 1.9                                      | $\beta^-$                        |
| Arsenic   | As     | 33 | 73     | $1.8 \times 10^5$       | $3.835 \times 10^{-6}$                | 0.6                                      | $\beta^+$                        |
|           | As     | "  | 74     | $1.4 \times 10^6$       | $4.928 \times 10^{-7}$                | 1.3( $\beta^-$ ) ; 0.9( $\beta^+$ )      | $\beta^-$ , $\beta^+$ , $\gamma$ |
|           | As     | "  | 78     | 3780                    | $1.825 \times 10^{-4}$                | 1.4                                      | $\beta^-$ , $\gamma$             |
| Selenium  | Se     | 34 | 81     | 1140                    | $6.054 \times 10^{-4}$                | 1.5                                      | $\beta^-$                        |
| Bromium   | Br     | 35 | 78     | 384                     | $1.796 \times 10^{-3}$                | 2.3                                      | $\beta^+$ , $\gamma$             |
|           | Br     | "  | 80     | 1080                    | $6.398 \times 10^{-4}$                | 2.0                                      | $\beta^-$ , ?                    |
|           | Br     | "  | 82     | $1.2 \times 10^6$       | $5.747 \times 10^{-6}$                | 0.465                                    | $\beta^-$ , $\gamma$             |
|           | Br     | "  | 83     | 8400                    | $8.214 \times 10^{-4}$                | 1.05                                     | $\beta^-$                        |
| Krypton   | Kr     | 36 | 79     | $1.2 \times 10^5$       | $5.751 \times 10^{-6}$                | ...                                      | $\beta^+$                        |
|           | Kr     | "  | ...    | ...                     | ...                                   | ...                                      | ...                              |
|           | Kr     | "  | 88     | $1.1 \times 10^4$       | $6.275 \times 10^{-5}$                | 2.5                                      | $\beta^-$                        |
| Rubidium  | Rb     | 37 | 86     | $1.7 \times 10^6$       | $4.588 \times 10^{-7}$                | 1.56                                     | $\beta^-$                        |
|           | Rb     | "  | 87     | $6 \times 10^{18}$      | $1.150 \times 10^{-19}$               | 0.132 or 0.25                            | $\beta^-$                        |
|           | Rb     | "  | 88     | $1.1 \times 10^4$       | $6.275 \times 10^{-5}$                | 5.1                                      | $\beta^-$                        |
|           | Rb     | "  | 89     | 900                     | $7.666 \times 10^{-4}$                | 3.8                                      | $\beta^-$ , $\gamma$             |
| Strontium | Sr     | 38 | 89     | $4.8 \times 10^6$       | $1.437 \times 10^{-7}$                | 1.50 or 1.32                             | $\beta^-$                        |
| Yttrium   | Y      | 39 | 87     | $5.04 \times 10^4$      | $1.37 \times 10^{-5}$                 | 0.5                                      | $e^-$ , $\gamma$                 |
|           | Y      | "  | 88     | 7200                    | $9.581 \times 10^{-5}$                | 1.2                                      | $\beta^+$                        |
|           | Y      | "  | 90     | $2.2 \times 10^5$       | $3.137 \times 10^{-6}$                | 2.6                                      | $\beta^-$                        |
|           | Y      | "  | 91     | $4.92 \times 10^6$      | $1.406 \times 10^{-7}$                | 1.6                                      | $\beta^-$ , $\gamma$             |
| Zirconium | Zr     | 40 | 89     | $2.8 \times 10^5$       | $2.465 \times 10^{-6}$                | 1.0                                      | $\beta^+$                        |
|           | Zr     | "  | 89     | 27                      | $2.55 \times 10^{-2}$                 | ...                                      | $e^-$ , $\gamma$                 |
|           | Zr     | "  | 93     | $5.44 \times 10^6$      | $1.256 \times 10^{-7}$                | 0.25 or 0.57 or 0.29                     | $\beta^-$ , $\gamma$             |
|           | Zr     | "  | 95     | $6.12 \times 10^4$      | $1.126 \times 10^{-5}$                | 1.0                                      | $\beta^-$                        |
|           | Zr     | "  | 97     | $3.6 \times 10^3$       | $1.917 \times 10^{-3}$                | 1.9                                      | $\beta^-$                        |
|           | Zr     | "  | 97     | $5.4 \times 10^8$       | $1.277 \times 10^{-4}$                | 1.5                                      | $\beta^-$                        |
|           | Zr     | "  | 97     | $2.52 \times 10^5$      | $2.62 \times 10^{-6}$                 | 1.17                                     | $\beta^-$                        |

APPENDIX I — BETA-DECAYING ISOTOPES — *continued*

| ISOTOPE      | SYMBOL | Z  | A      | HALF-LIFE<br>(Sec.)     | DECAY CONST.<br>(Sec. <sup>-1</sup> )                  | ENERGY<br>MEV.        | RADIATION                            |
|--------------|--------|----|--------|-------------------------|--------------------------------------------------------|-----------------------|--------------------------------------|
| Columbium    | Cb     | 41 | 92     | 9.5 × 10 <sup>5</sup>   | 7.28 × 10 <sup>-6</sup>                                | 1.38 or 0.59          | β <sup>-</sup> , γ                   |
|              | Cb     | „  | 94     | 396                     | 1.743 × 10 <sup>-4</sup>                               | 1.4                   | β <sup>-</sup> , γ                   |
|              | Cb     | „  | 95     | 4500                    | 1.533 × 10 <sup>-4</sup>                               | 1.0                   | β <sup>-</sup>                       |
| Molybdenum   | Mo     | 42 | 91     | 1020                    | 6.764 × 10 <sup>-4</sup>                               | ...                   | β <sup>+</sup>                       |
|              | Mo     | „  | 99     | 2.4 × 10 <sup>5</sup>   | 2.874 × 10 <sup>-6</sup>                               | 1.5                   | β <sup>-</sup> , γ                   |
|              | Mo     | „  | 101    | 876                     | 7.88 × 10 <sup>-3</sup>                                | 1.8, 1.0, 2.2         | β <sup>-</sup> , γ                   |
| Masurium     | Ma     | 43 | 101    | 8.4 × 10 <sup>3</sup>   | 8.21 × 10 <sup>-4</sup>                                | 1.1, 1.3              | β <sup>-</sup> , γ                   |
|              | Ma     | „  | 101    | 3.30 × 10 <sup>3</sup>  | 2.089 × 10 <sup>-4</sup>                               | 2.5                   | β <sup>-</sup> , γ                   |
|              | Ma     | „  | 96     | 9.72 × 10 <sup>3</sup>  | 7.18 × 10 <sup>-4</sup>                                | ...                   | β <sup>+</sup> (?)                   |
|              | Ma     | „  | 101?   | 18                      | 3.83 × 10 <sup>2</sup>                                 | ...                   | β <sup>-</sup>                       |
|              | Ma     | „  | 101?   | 1.31 × 10 <sup>5</sup>  | 5.34 × 10 <sup>-6</sup>                                | ...                   | β <sup>-</sup>                       |
|              | Ma     | „  | 99     | 2.38 × 10 <sup>4</sup>  | 2.93 × 10 <sup>-5</sup>                                | ...                   | e <sup>-</sup> , γ                   |
| Ruthenium    | Ru     | 44 | 105    | 1.44 × 10 <sup>4</sup>  | 4.6 × 10 <sup>-5</sup>                                 | 1.5                   | β <sup>-</sup>                       |
|              | Ru     | „  | 105    | 240                     | 2.874 × 10 <sup>-3</sup>                               | 4.0                   | β <sup>-</sup>                       |
| Rhodium      | Rh     | 45 | 102    | 1.81 × 10 <sup>7</sup>  | 3.828 × 10 <sup>-8</sup>                               | 1.1 (β <sup>-</sup> ) | β <sup>-</sup> , β <sup>+</sup> , γ  |
|              | Rh     | „  | 104    | 44                      | 1.568 × 10 <sup>-3</sup>                               | 2.3                   | β <sup>-</sup>                       |
|              | Rh     | „  | 105    | 1.22 × 10 <sup>5</sup>  | 5.70 × 10 <sup>-6</sup>                                | 0.5                   | β <sup>-</sup>                       |
| Palladium    | Pd     | 46 | 107    | 4.7 × 10 <sup>4</sup>   | 1.468 × 10 <sup>-5</sup>                               | 1.03                  | β <sup>-</sup>                       |
|              | Pd     | „  | 111    | 1560                    | 4.424 × 10 <sup>-4</sup>                               | 3.5                   | β <sup>-</sup>                       |
| Silver       | Ag     | 47 | 106    | 1470                    | 4.696 × 10 <sup>-4</sup>                               | 2.04                  | β <sup>+</sup> (?)                   |
|              | Ag     | „  | 108    | 138                     | 5.001 × 10 <sup>-3</sup>                               | 2.8                   | β <sup>-</sup>                       |
|              | Ag     | „  | 110    | 22                      | 3.136 × 10 <sup>-3</sup>                               | 2.8                   | β <sup>-</sup> , γ                   |
|              | Ag     | „  | 112    | 1.2 × 10 <sup>4</sup>   | 5.751 × 10 <sup>-5</sup>                               | 2.2                   | β <sup>-</sup> , γ                   |
|              | Ag     | „  | 111    | 6.48 × 10 <sup>5</sup>  | 1.07 × 10 <sup>-6</sup>                                | 0.8                   | β <sup>-</sup>                       |
| Cadmium      | Cd     | 48 | 115    | 2.2 × 10 <sup>5</sup>   | 3.136 × 10 <sup>-6</sup>                               | 1.11                  | β <sup>-</sup> , γ                   |
|              | Cd     | „  | 115(?) | 3.46 × 10 <sup>4</sup>  | 2.006 × 10 <sup>-7</sup>                               | 0.095                 | β <sup>-</sup> , γ                   |
|              | Cd     | „  | 117    | 1.35 × 10 <sup>4</sup>  | 5.1/ × 10 <sup>-5</sup>                                | ...                   | β <sup>-</sup>                       |
| Indium       | In     | 49 | 110    | 3900                    | 1.781 × 10 <sup>-4</sup>                               | 1.6                   | β <sup>+</sup>                       |
|              | In     | „  | 111    | 1380                    | 5.001 × 10 <sup>-4</sup>                               | ...                   | β <sup>+</sup> , γ                   |
|              | In     | „  | 114    | 72                      | 9.580 × 10 <sup>-3</sup>                               | 1.98                  | β <sup>-</sup>                       |
|              | In     | „  | 116    | 13 or 3240              | 5.342 × 10 <sup>-2</sup> or<br>2.12 × 10 <sup>-4</sup> | 2.8 or .85            | β <sup>-</sup> or β <sup>-</sup> , γ |
|              | In     | „  | 117    | 7020                    | 9.844 × 10 <sup>-5</sup>                               | 1.73                  | β <sup>-</sup> , γ, e <sup>-</sup>   |
| Tin          | Sn     | 50 | 125    | 540                     | 1.28 × 10 <sup>-3</sup>                                | ...                   | β <sup>-</sup>                       |
| Antimony     | Sb     | 51 | 120    | 1020                    | 6.764 × 10 <sup>-4</sup>                               | 1.53                  | β <sup>+</sup>                       |
|              | Sb     | „  | 122    | 2.4 × 10 <sup>5</sup>   | 2.884 × 10 <sup>-6</sup>                               | 0.81                  | β <sup>-</sup> , γ                   |
|              | Sb     | „  | 122    | > 2.4 × 10 <sup>5</sup> | ~ 2.884 × 10 <sup>-6</sup>                             | 1.64                  | β <sup>-</sup> , γ                   |
| Tellurium    | Te     | 52 | 129    | 4320                    | 1.6 × 10 <sup>-4</sup>                                 | ...                   | β <sup>-</sup>                       |
|              | Te     | „  | 131    | 1500                    | 4.60 × 10 <sup>-4</sup>                                | ...                   | β <sup>-</sup>                       |
| Iodine       | I      | 53 | 126    | 1.1 × 10 <sup>8</sup>   | 6.272 × 10 <sup>-7</sup>                               | 1.1                   | β <sup>-</sup> , γ                   |
|              | I      | „  | 128    | 1499.40                 | 4.61 × 10 <sup>-4</sup>                                | 1.85, 1.05, 2.10      | β <sup>-</sup> , γ                   |
|              | I      | „  | 130    | 4.54 × 10 <sup>4</sup>  | 1.516 × 10 <sup>-5</sup>                               | 0.61, 1.03            | β <sup>-</sup> , γ                   |
|              | I      | „  | 131    | 6.9 × 10 <sup>5</sup>   | 1.00 × 10 <sup>-6</sup>                                | ...                   | β <sup>-</sup> , γ                   |
|              | I      | „  | 133    | 7.92 × 10 <sup>4</sup>  | 8.78 × 10 <sup>-5</sup>                                | 1.1                   | β <sup>-</sup>                       |
| Xenon        | Xe     | 54 | 135    | 936                     | 7.371 × 10 <sup>-3</sup>                               | 0.7 or 0.6            | β <sup>-</sup> , γ                   |
|              | Xe     | „  | 137    | 204                     | 3.39 × 10 <sup>-3</sup>                                | 4.0                   | β <sup>-</sup>                       |
|              | Xe     | „  | 135    | 3.38 × 10 <sup>4</sup>  | 2.403 × 10 <sup>-5</sup>                               | 0.95, 0.90            | β <sup>-</sup> , γ                   |
| Cesium       | Cs     | 55 | 134    | 5.35 × 10 <sup>7</sup>  | 1.29 × 10 <sup>-8</sup>                                | 0.9                   | β <sup>-</sup> , γ                   |
|              | Cs     | „  | 134    | 1.1 × 10 <sup>4</sup>   | 6.275 × 10 <sup>-5</sup>                               | 1.0                   | β <sup>-</sup>                       |
|              | Cs     | „  | 138    | 1980                    | 3.48 × 10 <sup>-4</sup>                                | 2.6                   | β <sup>-</sup>                       |
| Barium       | Ba     | 56 | 139    | 5160                    | 1.336 × 10 <sup>-4</sup>                               | 1 or 2.3              | β <sup>-</sup> , γ                   |
|              | Ba     | „  | 140    | 1.08 × 10 <sup>5</sup>  | 6.39 × 10 <sup>-5</sup>                                | 1.2                   | β <sup>-</sup>                       |
| Lanthanum    | La     | 57 | 140    | 1.44 × 10 <sup>5</sup>  | 4.80 × 10 <sup>-6</sup>                                | 1.41                  | β <sup>-</sup> , γ                   |
| Cerium       | Ce     | 58 | 139    | 1.26 × 10 <sup>2</sup>  | 5.322 × 10 <sup>-3</sup>                               | ...                   | β <sup>+</sup>                       |
|              | Ce     | „  | 143    | 1.3 × 10 <sup>5</sup>   | 5.342 × 10 <sup>-6</sup>                               | ...                   | β <sup>-</sup> (?)                   |
|              | Ce     | „  | 141    | 2.59 × 10 <sup>8</sup>  | 2.67 × 10 <sup>-7</sup>                                | 0.65                  | β <sup>-</sup> , γ                   |
| Praseodymium | Pr     | 59 | 140    | 210                     | 3.28 × 10 <sup>-3</sup>                                | 2.4                   | β <sup>+</sup>                       |
|              | Pr     | „  | 142    | 6.95 × 10 <sup>4</sup>  | 1.010 × 10 <sup>-5</sup>                               | 2.14                  | β <sup>-</sup> , γ                   |
|              | Pr     | „  | 143    | 1.17 × 10 <sup>5</sup>  | 6.115 × 10 <sup>-7</sup>                               | 0.95                  | β <sup>-</sup>                       |
|              | Pr     | „  | 143(?) | 1020                    | 6.9 × 10 <sup>-4</sup>                                 | 3.1                   | β <sup>-</sup>                       |



APPENDIX I — BETA-DECAYING ISOTOPES — *continued*

| ISOTOPE      | SYMBOL             | Z  | A        | HALF-LIFE<br>(Sec.)                                 | DECAY CONST.<br>(Sec. <sup>-1</sup> )                   | ENERGY<br>MEV.           | RADIATION                          |
|--------------|--------------------|----|----------|-----------------------------------------------------|---------------------------------------------------------|--------------------------|------------------------------------|
| Neodymium    | Nd                 | 60 | 141      | 9 × 10 <sup>3</sup>                                 | 7.66 × 10 <sup>-5</sup>                                 | 0.78                     | β <sup>+</sup>                     |
| Illinium     | Il                 | 61 | ?        | 4.58 × 10 <sup>5</sup>                              | 1.506 × 10 <sup>-6</sup>                                | 2                        | β <sup>-</sup> , γ                 |
|              | Il                 | „  | ?        | 9.72 × 10 <sup>3</sup>                              | 7.098 × 10 <sup>-6</sup>                                | 2                        | β <sup>-</sup> , γ                 |
|              | Il                 | „  | ?        | 1.38 × 10 <sup>6</sup>                              | 5.000 × 10 <sup>-7</sup>                                | 1.7                      | β <sup>-</sup> , γ                 |
| Samarium     | Sm                 | 62 | 154?     | 1260                                                | 5.321 × 10 <sup>-4</sup>                                | 1.8                      | β <sup>-</sup>                     |
| Europium     | Eu                 | 63 | 150      | 9.72 × 10 <sup>4</sup>                              | 7.098 × 10 <sup>-5</sup>                                | ...                      | β <sup>+</sup>                     |
|              | Eu                 | „  | 154      | 1.57 × 10 <sup>2</sup> to<br>2.52 × 10 <sup>8</sup> | 4.40 × 10 <sup>-9</sup> or<br>2.62 × 10 <sup>-9</sup>   | 0.9                      | β <sup>-</sup> , γ                 |
|              | Eu                 | „  | 152      | 3.31 × 10 <sup>4</sup>                              | 2.091 × 10 <sup>-5</sup>                                | 1.88(β <sup>-</sup> )    | β <sup>-</sup> , γ, e <sup>-</sup> |
| Terbium      | Tb                 | 65 | 160      | 6.22 × 10 <sup>6</sup>                              | 1.193 × 10 <sup>-7</sup>                                | 0.70                     | β <sup>-</sup> , γ                 |
| Dysprosium   | Dy                 | 66 | 165      | 9000                                                | 7.666 × 10 <sup>-5</sup>                                | 1.20                     | β <sup>-</sup>                     |
| Holmium      | Ho                 | 67 | 166      | 1.3 × 10 <sup>5</sup>                               | 5.342 × 10 <sup>-6</sup>                                | 1.6                      | β <sup>-</sup>                     |
| Lutecium     | Lu                 | 71 | 176, 177 | 1.22 × 10 <sup>4</sup>                              | 5.855 × 10 <sup>-5</sup>                                | 1.15                     | β <sup>-</sup>                     |
|              | Lu                 | „  | 176, 177 | 5.70 × 10 <sup>5</sup>                              | 1.22 × 10 <sup>-6</sup>                                 | 0.44                     | β <sup>-</sup>                     |
|              | Lu                 | „  | 176      | 2.3 × 10 <sup>18</sup>                              | 3 × 10 <sup>-19</sup>                                   | 0.215, 0.40              | β <sup>-</sup>                     |
| Tantalum     | Ta                 | 73 | 182      | 8.38 × 10 <sup>6</sup>                              | 8.233 × 10 <sup>-7</sup>                                | 1.0, 0.98, 0.32<br>0.050 | β <sup>-</sup> , γ                 |
| Tungsten     | W                  | 74 | 185      | 6.7 × 10 <sup>6</sup>                               | 1.030 × 10 <sup>-7</sup>                                | 0.55—0.65<br>0.64—0.72   | β <sup>-</sup> , γ                 |
|              | W                  | „  | 187      | 8.3 × 10 <sup>4</sup>                               | 8.915 × 10 <sup>-6</sup>                                | 1.4                      | β <sup>-</sup> , γ                 |
| Rhenium      | Re                 | 75 | 186      | 3.2 × 10 <sup>5</sup>                               | 2.157 × 10 <sup>-6</sup>                                | 1.05                     | β <sup>-</sup>                     |
|              | Re                 | „  | 188      | 6.5 × 10 <sup>4</sup>                               | 1.062 × 10 <sup>-5</sup>                                | 2.5                      | β <sup>-</sup>                     |
| Osmium       | Os                 | 76 | 191      | 1.2 × 10 <sup>5</sup>                               | 5.748 × 10 <sup>-6</sup>                                | 1.5                      | β <sup>-</sup> , γ                 |
|              | Os                 | „  | 193      | 3.9 × 10 <sup>6</sup>                               | 1.770 × 10 <sup>-7</sup>                                | 0.35                     | β <sup>-</sup> , γ                 |
| Iridium      | Ir                 | 77 | 194      | 6.8 × 10 <sup>4</sup>                               | 1.014 × 10 <sup>-5</sup>                                | 2.2                      | β <sup>-</sup> , γ                 |
|              | Ir                 | „  | 192, 194 | 518 × 10 <sup>6</sup>                               | 1.33 × 10 <sup>-7</sup>                                 | ...                      | β <sup>-</sup> , γ                 |
| Platinum     | Pt                 | 78 | 197      | 6.48 × 10 <sup>4</sup>                              | 1.064 × 10 <sup>-5</sup>                                | 0.65, 0.72               | β <sup>-</sup>                     |
|              | Pt                 | „  | 199      | 1860                                                | 3.7 × 10 <sup>-4</sup>                                  | 1.8                      | β <sup>-</sup>                     |
| Gold         | Au                 | 79 | 196      | 4 × 10 <sup>5</sup>                                 | 1.726 × 10 <sup>-6</sup>                                | 0.36                     | β <sup>-</sup> , γ                 |
|              | Au                 | „  | 198      | 2.3 × 10 <sup>5</sup>                               | 3 × 10 <sup>-6</sup>                                    | 0.8                      | β <sup>-</sup> , γ                 |
|              | Au                 | „  | 199      | 2.85 × 10 <sup>5</sup>                              | 2.429 × 10 <sup>-6</sup>                                | 1.01                     | β <sup>-</sup> , γ                 |
| Mercury      | Hg                 | 80 | 203, 205 | 4.45 × 10 <sup>6</sup>                              | 1.550 × 10 <sup>-7</sup>                                | 0.46                     | β <sup>-</sup> , γ                 |
|              | Hg                 | „  | 205      | 330                                                 | 2.9 × 10 <sup>-3</sup>                                  | 1.62                     | β <sup>-</sup>                     |
| Thallium     | Tl                 | 81 | 204      | 253                                                 | 2.727 × 10 <sup>-3</sup>                                | 1.6                      | β <sup>-</sup>                     |
|              | Tl                 | „  | 206      | 1.1 × 10 <sup>8</sup>                               | 6.27 × 10 <sup>-9</sup>                                 | 0.87                     | β <sup>-</sup>                     |
|              | AcC'               | „  | 207      | 291                                                 | 2.371 × 10 <sup>-3</sup>                                | 1.47                     | β <sup>-</sup> , γ                 |
|              | ThC'               | „  | 208      | 186                                                 | 5.480 × 10 <sup>-3</sup>                                | 1.82                     | β <sup>-</sup> , γ                 |
|              | RaC'               | „  | 210      | 79.2                                                | 1.209 × 10 <sup>-3</sup>                                | 1.80                     | β <sup>-</sup>                     |
|              | Lead               | Pb | 82       | 203                                                 | 615.00                                                  | 1.122 × 10 <sup>-3</sup> | 1.66                               |
| Lead         | Pb                 | „  | 209      | 10.8 × 10 <sup>3</sup>                              | 6.389 × 10 <sup>-4</sup>                                | 0.7 ; 0.75               | β <sup>-</sup>                     |
|              | RaD                | „  | 210      | 6.93 × 10 <sup>6</sup>                              | 1.00 × 10 <sup>-9</sup>                                 | 0.255                    | β <sup>-</sup> , γ                 |
|              | AcB                | „  | 211      | 2100                                                | 3.287 × 10 <sup>-4</sup>                                | 0.5 ; 1.4                | β <sup>-</sup> , γ                 |
|              | ThB                | „  | 212      | 3.8 × 10 <sup>4</sup>                               | 1.817 × 10 <sup>-5</sup>                                | 0.36                     | β <sup>-</sup> , γ                 |
|              | RaB                | „  | 214      | 7608                                                | 9.069 × 10 <sup>-4</sup>                                | 0.65                     | β <sup>-</sup> , γ                 |
|              | RaE                | 83 | 210      | 4.3 × 10 <sup>5</sup>                               | 1.604 × 10 <sup>-6</sup>                                | ...                      | β <sup>-</sup>                     |
|              | 87 (Ack)           | 87 | 223      | 1260                                                | 5.33 × 10 <sup>-4</sup>                                 | ...                      | β <sup>-</sup> , γ                 |
|              | Ms Th              | 88 | 228      | 2.1 × 10 <sup>8</sup>                               | 3.193 × 10 <sup>-9</sup>                                | 0.053                    | β <sup>-</sup>                     |
|              | Ms Th <sub>2</sub> | 89 | 228      | 2.2 × 10 <sup>4</sup>                               | 3.137 × 10 <sup>-5</sup>                                | 1.55, 4.5                | β <sup>-</sup> , γ                 |
|              | Uy                 | 90 | 231      | 8.86 × 10 <sup>4</sup> or<br>8.64 × 10 <sup>4</sup> | 7.985 × 10 <sup>-6</sup> or<br>7.787 × 10 <sup>-6</sup> | 0.2                      | β <sup>-</sup>                     |
| Thorium      | Th                 | 90 | 233      | 1380                                                | 5.000 × 10 <sup>-4</sup>                                | ...                      | β <sup>-</sup>                     |
|              | Ux <sub>1</sub>    | „  | 234      | 8.68 × 10 <sup>4</sup> or<br>8.82 × 10 <sup>4</sup> | 7.823 × 10 <sup>-6</sup> or<br>7.77 × 10 <sup>-6</sup>  | 0.11, 0.13, 0.20         | β <sup>-</sup> , γ                 |
| Protactinium | Pa                 | 91 | 233      | 2.37 × 10 <sup>6</sup>                              | 2.911 × 10 <sup>-7</sup>                                | 0.4 or 0.23              | β <sup>-</sup> , γ, e <sup>-</sup> |
|              | Uz                 | „  | 234      | 2.41 × 10 <sup>4</sup>                              | 2.862 × 10 <sup>-5</sup>                                | 0.56, 1.55               | β <sup>-</sup> , γ                 |
|              | Ux <sub>2</sub>    | „  | 234      | 68                                                  | 1.014 × 10 <sup>-2</sup>                                | 2.32                     | β <sup>-</sup> , γ                 |
| Uranium      | U                  | 92 | 237      | 6 × 10 <sup>5</sup>                                 | 1.150 × 10 <sup>-6</sup>                                | 0.26                     | β <sup>-</sup> , γ                 |
|              | U                  | „  | 239      | 1410                                                | 4.894 × 10 <sup>-4</sup>                                | 0.56, 1.2                | β <sup>-</sup> , γ                 |
| Neptunium    | Np                 | 93 | 235      | > 3.15 × 10 <sup>7</sup>                            | < 2.188 × 10 <sup>-8</sup>                              | ...                      | K, γ                               |
|              | Np                 | „  | 238      | 1.728 × 10 <sup>5</sup>                             | 3.993 × 10 <sup>-6</sup>                                | 1.35                     | β <sup>-</sup> , γ                 |
|              | Np                 | „  | 239      | ...                                                 | 3.427 × 10 <sup>-6</sup>                                | 0.14 ; 0.78              | β <sup>-</sup> , γ                 |

# REVIEWS

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**History of Dyes and Dyeing in the Bombay Presidency**, by Mr. B. N. Phadke (Dastane Bros.; Home Service Ltd., 456 Raviwar Peth, Poona 2), 1947, pp. 152. Price Rs. 12.

INDIA IS NOW ONE OF THE LEADING COTTON textile manufacturing countries of the world and Bombay province is the home of the cotton textile industry in India. A survey of the dyeing industry in that province is tantamount to giving a correct picture of the industry in the whole of India. Mr. Phadke's book under review is an important contribution to the history of dyes and dyeing—particularly when there is in evidence a keen desire for the establishment of a dyestuff industry in India.

The book is divided into 5 chapters and an appendix containing 11 tables is included at the end.

Chapter I is a historical introduction. The second chapter deals with indigenous dyestuffs. The process of dyeing of cotton, silk, wool, ivory, wood and leather adopted in various parts of the Bombay Presidency are discussed in detail and recent work on vegetable dyes and efforts to revive the indigenous dyestuff industry are also given.

Various methods of application of mineral pigments on textile fibre for producing shades of khaki are fully treated in the third chapter.

The fourth chapter is the most important in that it is concerned with synthetic dyestuffs and methods of application to different types of fibres like cotton, silk, rayon and acetate silk. This chapter is full of useful information regarding the trade names under which the various classes of dyes are sold in the market by different manufacturers, and their extent of popularity in the trade. The classification of the dyes and their application for miscellaneous purposes, for example, (a) yarn printing; (b) writing inks and stamping paste; (c) food colours; (d) lakes and pigments; and (e) paper, wood and leather colouring are very useful.

That the indigenous dyes, with very few exceptions, cannot compete with coal-tar

colours has been well brought out in the last chapter. The circumstances which have led to the decline of the vegetable dyestuff industry, and the reasons therefor, as well as the possibility of revival of a few indigenous dyes like natural indigo and cutch have also been well brought out.

The appendix contains interesting information on the chemical aspects of some of the dyes, the communities engaged in the industry, the variety of textile goods manufactured in various parts of the Presidency and different classes of dyestuffs used therein, list of manufacturers, importers or sellers and trade names, statistics of export-import data and other relevant details.

The get-up of the book is satisfactory. Some more care might have been exercised about the botanical names of the plants listed in table No. 1 of the appendix where there are innumerable mistakes in the names of the plants: *Butea frondosa*, Rox; *Adanathera payonina*, W; *Hyctanthes arbartristis*; *Sympocos racemosa*, Rox; *Acacia catechu* Will; *Permelia chamchadalis*, etc. It is understandable why the author should have used the out-of-date name of *Calysaccion longifolium* when *Ochrocarpos longifolius*, Benth & Hook, is found in Cooks *Flora of the Bombay Presidency* which has been listed in the bibliography. It would have been better if the author had referred to up-to-date books on chemistry of natural colouring matters before giving the structural formulae of some of the natural dyes. For example, Brazilin has been given the formula ascribed to it by Pfeiffer whereas it has been admitted that Perkins formula is preferable. Similarly, for catechin Freudenberg's formula is to be preferred to Nierenstein formula given by the author. It is difficult to understand how the accepted Robertson and Brockmann formulae have been discarded by the author for rottlerin. The colouring matter of Henna has been identified to be 2-hydroxy 1-4 naphthaquinone whereas the author has given it as  $C_{10}H_6O_3$ .

The book contains valuable information and supplies a long-felt want.

A. CHARI

**Industrial Profits in India**, by M. C. Munshi (Research Department, Federation of Indian Chambers of Commerce & Industry), 1949, pp. iii+326. Price Rs. 15.

A SCIENTIFIC STUDY OF INDUSTRIAL PROFITS is a valuable aid to the framing of business policy. It gives better insight into business activity in general, apart from indicating trends of corporate earnings. Further, a proper analysis of profit trends is indispensable for deciding questions such as a fair return on capital or the influence of taxation on the production incentive. The author of the publication under review has attempted an analysis of the industrial profits in India during the years 1936-44 and has based his study on a painstaking collection of statistical data. By a close study of the balance-sheets and profit and loss statements of a large number of joint-stock companies, the author has arrived at many interesting findings relating to the progress of some of the major industries and their comparative success or otherwise. For instance, the author shows the high profits in cotton, paper and jute industries followed by sugar and iron and steel, and contrasts how the coal industry, because of transport difficulties and internal weaknesses, could benefit little out of the war except towards the end of the period. "An important feature of our industrial development during the War", says the author, "— in marked contrast with those of some other belligerent countries — was that our industries could earn high profits even when the quantum of their production remained either stationary or showed a marked decline." Again, "Another unmistakable trend that stands out in bold relief from our tables is that all these industries during the War have collected from the pockets of the consumer what they filled the coffers of the Government with."

The enquiry covers the seven major industries and relates to 71 cotton mill companies, 60 jute mill companies, 54 coal mining companies, 48 sugar mill companies, 8 paper mill companies and 2 each in the case of iron and steel and cement industries. The book abounds in numerous tables and graphs revealing trends of net profits, dividends, provision for taxation, contributions to reserves, the ratio of net profits to sales and numerous other facts and figures pertaining to the enquiry. The work will, no doubt, be regarded as a significant contri-

bution to the field of statistics relating to industrial profits in India. It is hoped that the study which ends with 1944 will be brought up to date.

R. B. P.

**Schimmel 1946 Report on Essential Oils, Aromatic Chemicals and Related Materials** (published by *Schimmel & Co., Inc.*, New York), pp. 137. Price \$3.25.

THE 1946 ANNUAL SCHIMMEL REPORT ON *Essential Oils, Aromatic Chemicals and Related Materials* has just become available and appears to be a volume of more than usual interest.

An interesting section is devoted to the pharmacology of essential oils, and contains discussions of their skin-irritating properties and of the anti-histaminic action of citral. The biogenesis of perfume materials comprises another interesting section.

Among the developments in the field of essential oils and perfume chemicals during the year, some of the following may prove of particular interest to readers:

The essential oil distilled from wild cloves collected in the Moluccas was discovered to be quite different in composition from commercial clove oil which is produced from clove buds from cultivated trees. The wild clove oil contained no eugenol and deposited crystalline substances not found in ordinary clove oil.

The properties of the volatile oil distilled from the blossoms of the Indian mango tree were reported and the essential oils of several varieties of wild thyme found in Portugal were examined. Myristicin was found to be one of the main constituents of the volatile oil of harvest fennel (*Ridolfia segetum*). A sulphur compound which had not previously been described in literature was found in the oil distilled from the leaves of the sea-buchu, a South African shrub. The compound was identified as butyl-pentenyl disulphide.

Two paraffin hydrocarbons, heptane and undecane, were identified in the essential oil from the oleoresin of Coulter pine. Phellandrene and terpene hydrocarbons in the same oleoresin is a rarity.

Several investigations relative to the composition of the animal fixatives were reported. A pyridine derivative was isolated from natural musk and several additional phenols and acids were identified in castoreum. Progress was made on the problem of the

structure of ambrein, a crystalline material found in ambergris.

In the field of organic synthesis, a new group of perfume materials having intense floral odours was discovered in the ethers derived from dicyclopentadiene. Various epoxides of the ionones were prepared by treating them with hydrogen peroxide or perphthalic acid. Thio ethers having marked insecticidal properties were obtained by the reaction of safrole and isosafrole with mercaptans.

A study of the isomeric forms of the Grignard reagent derived from pinene hydrochloride provided an explanation for the fact that reactions involving this compound often give unexpected products. A simple method was devised for converting the reagent entirely to the form giving the desired product.

The statistical section of the report is also of considerable interest for this year as data not accessible for 1945 or the war years generally was now available. Figures appear on Italian exports, the patchouly trade in Malaya, and Taiwan camphor production, to mention only a few.

**Practical Applications of Recent Lac Research** (Revised & Enlarged Edition), Edited by H. K. Sen & M. Venugopalan (Orient Longmans Ltd.), pp. 123. Price Rs. 6/8.

INDIA PRACTICALLY HOLDS A WORLD MONOPOLY for the most versatile natural resin, lac, the cultivation and refinement of which is an important cottage industry in the country; but it is left to the advanced countries of the West to put it into use in several important industries. When the position of lac was, therefore, seriously threatened by the development of innumerable synthetic plastics, the Institute at Ranchi was established to safeguard the industry by means of research tending to make lac not only more fit for existing industries but also to find new uses for it by suitable modifications of its properties. What now remains to be done is to reduce the dependence of this natural product on foreign markets by starting such consuming industries in this country as have been made possible by the valuable researches of the Institute in this direction.

In the revised edition of "Practical Applications" under review, new chapters

have been added on ethers and ether-esters of shellac, dewaxed lac and varnish, water varnishes, instantaneous sound-recording discs and the manufacture of oil-cloth. Of the other chapters, that on hard lac resin has been expanded to include new ways of preparing the resin. Chapter VI on shellac plastics now includes a section on the possibility of manufacturing fillers in this country and chapter VI is expanded to include shellac cements, waterproof abrasive cloth and paper and possible uses of shellac in the manufacture of ply-wood, laminated boards and abrasive wheels, to mention only a few instances of enlargement of the scope of the book. Undoubtedly, therefore, the publication is of great practical importance to the industrial chemist.

The costing data, however, based on 4 annas per pound of shellac, Re. 1 per gallon of spirit and Rs. 2 per gallon of linseed oil, to take a few instances, tend to make the industrial possibilities appear too optimistic and unreal to a hard-boiled industrialist. The editors, no doubt, say in the preface that production costs are only pointers to normal conditions, but when those "normal" conditions return and whether they will return at all are highly problematical. The Institute would have done well to show in this new edition that with the prevailing prices of raw materials, the commercialization of these researches was a practical proposition.

A few slips in the book as, for example, not mentioning filtration of lac solution after treatment with activated carbon (page 11), omitting to mention the daily or monthly turn-over on which overhead is based (pages 2, 10 and 19), the unusual basis of ten working hours per day (page 28), and 55° barometer for perhaps 55° Bé. (page 82), may be corrected in the next edition. The printers, also, might have taken more care to see to the proper alignment of types on certain pages. These are, however, very minor points which do not in any way detract the value of the publication.

The book should be recommended as a very valuable technical handbook, and if only the industrialists of this country seriously consider the possibilities of establishing even a few of the industries recommended in the book, no doubt the Institute would feel highly rewarded for its efforts.

M. R.

**Centrifugal and Other Rotodynamic Pumps**, by Herbert Addison (Chapman & Hall Ltd.), 1948, pp. x+492. Price 36s.

THE BOOK UNDER REVIEW DEALS WITH CENTRIFUGAL and other rotary types of pumps which the author calls by the name of rotodynamic pumps. It is a valuable addition to the library of books dealing with centrifugal pumps. Though there are many books dealing with the design and construction of centrifugal pumps, yet Mr. Addison's book is the first of its kind in which the other types of rotary pumps like the semi-axial, axial, bore-hole and propeller types of pumps are fully described. His nomenclature of rotodynamic pumps seems to be very apt in that all the above-mentioned pumps have to be directly coupled with a rotary motive unit.

The division of the book into Principles, Design and Construction, Performance and Installations, and Operation is very logical. Though there have been repetitions of the subject-matter in more than one chapter, such repetitions have enabled the understanding of the particular problem of a pump under design, construction and maintenance better.

The chapter dealing with the design and construction is very illuminating for a student of engineering and a practising engineer. The various types of centrifugal and other rotodynamic pumps are classified and their particular merit for specific purposes are fully explained. This chapter recommends the book for inclusion as a handbook on pumps for the use of a practising engineer. The enumeration of certain practical difficulties that might arise in the handling of hot, impure and corrosive liquids is very useful. Some details of construction in handling these types of liquids, like the types of sealing rings in article 84, water-seal glands in article 87 and details of casings in article 89 add greater practical utility to the book.

In the chapter dealing with mixed flow and axial flow pumps, the reference to airfoil theory has brought the subject-matter in level with modern development. The chapter on special-duty pumps is a valuable collection of special types of pumps which a practising engineer needs most in a reference book of this type. The articles on bore-well pumps of different types with the accompanying diagrams are very well written.

Under the heading of pumps for hot and volatile liquids, the particular problems that might arise due to expansion, leakage, extreme heat resulting in the needful protection for the stuffing boxes, protection to packing suggesting their solution are useful additions to the knowledge of the working of the pumps of this type.

In the chapter on definitions and terminology, the author has clearly defined the manometric, hydraulic and actual efficiencies under different conditions of pumping. In the other two chapters following this, the author has dealt with the various causes that contribute to the realization of lower efficiencies than the designed one in actual practice and how best the gap between the two could be minimized.

The chapter on "Installations and Operations" does not form part of the theory of centrifugal and rotodynamic pumps but the enumeration of the allied machinery and auxiliary appliances has added to the usefulness of the book to the student as well as the engineer. It gives an idea of how and when a particular type of motive unit should be selected for a particular type of pump.

In the final survey of complete pumping plants, the author has selected typical examples of a simple layout, a low-lift plant, a land drainage unit, a screw type of plant, a propeller type of installation, a semi-axial and axial pump unit, vertical pumps of high-lift and low-lift varieties as in land drainage and sewage pumping and bore-hole pumping plants. The operation of pumping units in series and in parallel have been very well described with adequate sketches. The principles of boosting pumps and arrangement of boosting stations are also valuable additions. The problems of surge and slum pressures in pumping installations has been particularly dealt with in detail and solutions indicated. A few articles on mobile pumping units such as suction dredger and pontoon pumps are usefully added in the chapter on "Particular Installations".

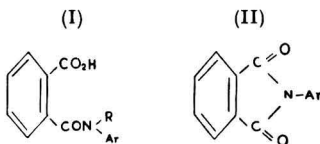
The last chapter of the book deals with certain worked examples of design, selection of pumps and auxiliary units which guide the student and the practising engineer in solving their problems.

On the whole, the book is highly commended as a text-book as well as a book of reference on the subject of centrifugal and rotodynamic pumps.

# NOTES & NEWS

## Plant Growth Regulators

A NEW GROUP OF PLANT GROWTH regulating chemicals, the *n*-aryl phthalamic acids, are described (*Science*, 1949, 109, 588). These compounds, which are readily prepared from phthalic anhydride and aromatic amines, can be represented by the general formula (I). The esters, amides and soluble salts of these acids as well as the *n*-aryl phthalimides (II) possess similar activity.



Tomato plants having 1 to 3 unset blossom clusters were sprayed with a solution (100 c.c.) containing 100 mg. of the chemical and a wetting agent. A general pattern of response was evident. At low concentrations, the fruit set was stimulated and seedless tomatoes were produced. A morphological response involving a broadening of the leaves was also evident. As the concentration of the chemical increased, the formative effect became more pronounced. At higher concentrations (2,000 p.p.m.), the plants were definitely injured.

*N*-1 naphthyl phthalamic acid, at a concentration of 0.1 p.p.m., caused leaf roll, at 0.31 p.p.m., epinasty, and at 20 p.p.m., stem swelling. Above this concentration fruit set was completely inhibited.

Maleic hydrazide is another interesting compound which has been found to produce a pronounced, but temporary, inhibiting effect on plant growth. The effect is secured with little visible harm to the plants.

Maleic hydrazide, its cupric salt and its zinc salt, dissolved or suspended in water at a concentration of 2,000 p.p.m. when sprayed on 6" tomato plants, the plants failed to grow for a period of about 2 months. Root growth was also inhibited. After about 1 month slight chlorosis was observed. After the quiescent period, growth resumed mainly from

lateral buds. A 1,000 p.p.m. solution produced a formative effect when regrowth started from the terminal bud. Similar results were obtained using triethanolammonium and diethanolammonium salts of maleic hydrazide. These salts seemed to be more effective than the free compound. Turf was treated with 8, 4, 2 and 1 lb. per acre of maleic hydrazide. The chemical with a dispersing agent was applied as a spray in 200 gal. of water per acre. The 1 lb. rate had a slight retarding effect; the 2 lb. rate inhibited growth for 1 to 2 weeks; the 4 lb. rate for about 2 weeks, and the 8 lb. rate for over 2 months. After the quiescent period, growth appeared normal.

The chemicals have no effect on the extent or time of germination, but the seedlings suffered a marked retardation of growth.

## Synthesis of Radioactive Silk

THE POSSIBILITY OF BIOLOGICAL synthesis of a stable, homogeneous, radioactive silk protein has been discussed (*Science*, 1949, 109, 625).

This is suggested by the unusual properties of fibroin, the protein of silk, containing  $C^{14}$ -labelled glycine and alanine in exceptionally small concentrations.

To test the ability of the silk worm *Platysamia cecropia* to incorporate radioactive glycine and alanine into the silk it synthesizes, 0.05 c.c. of a mixture of the two was injected into the body cavity of the mature silkworm (animal A). The injection contained 0.013 mg. of  $C^{14}$ -carboxyl-labelled glycine with an activity of 18,000 cpm. and 0.02 mg. of  $C^{14}$ -carboxyl-labelled *dl*-alanine with an activity of 9,000 cpm. (Geiger Muller).

A thin piece of cocoon fabric spun by the worm after 24 hr. of normal feeding exhibited radioactivity. A similar result was obtained with a silk-worm (animal B) injected with 0.05 c.c. of a solution containing 0.04 mg. of  $C^{14}$ -carboxyl-labelled *dl*-alanine alone, with an activity of 18,000 cpm. Fragments of cocoon from each insect placed under an unexposed film and later developed, invariably gave radioautographs

revealing details of the pattern of the silk fibres.

To test whether radioactivity of the silk was actually present in the component amino acids, the protein hydrolysate was subjected to ninhydrin procedure. Duplicate counts of the barium carbonate precipitate revealed 160 and 150 cpm. per 0.1 mm. barium carbonate, thus showing the radioactivity to be present in the  $\alpha$ -carboxyl group of some amino acid associated with the silk fibres.

To make sure that the labelled amino acids were actually incorporated into the peptide chain structure of the protein itself and that they were not adsorbed into the silk fibres, reprecipitated protein was tested for radioactivity. The hydrolysate from 13 gm. of this protein, on ninhydrin determination, gave 12.7 cpm. per 0.1 mm. barium carbonate, or 127 cpm. per 0.1 mm. of hydrolysate of labelled silk fibroin as corrected to standard conditions.

The above results led to experiments on protein synthesis in the isolated silk gland of the insect. Four silk glands from two mature insects were incubated at 37°C. in an atmosphere of oxygen. The proteins from the glands, gave on assay for radioactivity, a total of 74 cpm. in the 0.067 mm. sample of barium carbonate or 93 cpm. per 0.1 mm. of barium carbonate as corrected to standard conditions, implying thereby that the amino acids were incorporated into protein in the living silk-worm gland *in vitro*. Since the fibroin was not isolated, there is no certainty, however, that silk rather than some other protein was synthesized under the conditions of the experiment.

There is the possibility that the labelled amino acids may have entered preformed fibroin molecules by "exchange" rather than by participation in a true synthesis of the fibroin molecule *de novo*. There may also be the formation of side-chain peptide bonds between the labelled amino acids and either amino or carboxyl groups not in the  $\alpha$ -position.

## Storing Seed Potatoes

STORING OF SEED POTATOES IN artificially lighted cellars to check their sprouting and consequent loss of nutrient under ordinary storage conditions is discussed (*Philips Tech. Rev.*, 1949, 10, 318).

Sprouting is checked at low temperatures (2° to 4°C.), and with a little light at 5° to 9°C. While

the former method is neither practicable nor economically justifiable, the latter involves tedious temperature regulations in the frosted glass-sheds where the potatoes have to be stored.

The effects of artificial light on sprouting were, therefore, investigated. One part of an underground test cellar with potatoes so stored in shallow boxes as to allow light to penetrate between them was illuminated with ordinary incandescent lamps and another part with fluorescent lamps of the "daylight" colour. The lighting was on continuously for a period of 3 months after which period the potatoes were taken out for planting.

In the first part lighted with 6 incandescent lamps installed in each section of 7 × 8 m., placed at intervals of 3 m., potatoes were found in a better condition than those which had been kept in clamps, but sprouting was not well checked. Firstly, the potatoes in the immediate vicinity of the lamps sprouted strongly notwithstanding the fact that they were receiving a fair amount of light. This is explained by heat radiations from the lamps which helped sprouting. Secondly, light being radiated from a central point in the incandescent lamp, there was little uniformity in the radiation. Large spaces were, therefore, left without any light.

The second part of the cellar illuminated with 5 TL fluorescent lamps of 40 w. installed in each section of 7 × 8 m. gave better results. In this case radiation of light was uniform all through and there were no shadow points, the lamps being mounted vertically against the wall. Moreover, there was scarcely any heat radiation to promote sprouting.

Investigations were then made on the kind of light which checks sprouting most and to ascertain what amount of light—using light of a certain wavelength—was just capable of sufficiently checking sprouting.

One lot of potatoes was irradiated with various intensities of blue and another lot with different intensities of red light. For the first lot a TL lamp was used with magnesium tungstate as luminophore and the addition of a blue filter; for the second lot a lamp with cadmium borate was used, with a red filter. The irradiation took place at 14°C. It was found that blue-violet light strongly checked the growth of sprouts, while red rays had but little effect.

Very little research having been done into the mechanism of sprouting of potatoes, it was presumed that the blue-violet light destroys the auxin—a hormone which is responsible for growth of sprouts.

Further irradiation tests were so arranged that the light fell continuously upon the potatoes from one side (as would be the case in the storing of potatoes).

Under blue light of an intensity up to about 30 erg./sq. cm. sec., the sprouts were perceptibly checked in growth. The sprout that had received about 2 erg./sq. cm. sec. was still somewhat phototropically directed.

Potatoes under red light, however, showed an unexpected phenomenon. Even a much lower luminous intensity of 1 erg./sq. cm. sec. (than with blue light) produced noticeable results. Moreover, the checked sprouts did not grow towards the light, not even under much stronger radiations.

From these experiments it appears that the checking of sprouting—at least under the influence of red light—is not to be accounted for by reason of a growth-stimulating substance being rendered inactive by the light. Rather it is to be supposed that, under red light, a growth-checking substance in the potatoes is activated. The question whether this hypothesis of activation of a growth-checking substance is correct is to be further investigated experimentally.

### Continuous Superphosphate Process

A NEW SUPERPHOSPHATE PROCESS with centralized control known as the "Socket Super Flo-process" is described (*C.T.J.*, 1949, 125, 103).

The raw materials, Florida pebble rock and 66° Bé. sulphuric acid are treated in an acidulating tower into which reactants are introduced in a finely divided state: the ground rock as a dust stream and the acid as an atomized spray. The reaction is very rapid. The superphosphate has a hard porous grain structure and possesses good physical properties for ammoniation, handling and application.

The rock, ground to 100 mesh in an air-swept Raymond pulverizer, is conveyed to continuous weighing machine by screw conveyor and bucket elevator. An air seal between the pulverized rock-feeding spout and the machine makes continuous weighing

possible by checking the fluid flow of the rock. For average operation, the scales are set to weigh out 25 tons of ground rock per hr. which, on reaction with approx. 20 tons of acid per hr., constitute a 45 ton per hr. operation.

The weighed rock dust is introduced through a horizontal steel air duct tangentially into the upper side of the cylindrical rubber-lined steel acidulating tower. Acid at 115° to 135°F. is delivered to the tower at the rate of 20 tons per hr. by a stainless steel atomizing nozzle in the centre of the tower.

The tower discharges directly into a puddler over which it is situated. The puddler has a rubber-lined housing and is equipped with propelling flights which pass the viscous material along to solidifier. The solidifier, a channel-like pallet conveyor made up of steel plates formed in a U-shape, fulfils the function of the den in the order process, the material leaving it being solid superphosphate ready for storage and curing.

The centralized control of the process makes it possible for a 3-man crew to operate a 45-ton per hr. plant as against 10 to 12 crew required with a den plant of about equal capacity. Power costs also compare favourably, the Super Flo-process using 1.12 kW.-hr. per shrunk ton as compared to 1.75 kW.-hr. in the den installation.

### Beneficiation of Lead-zinc Ore from Zawar Mine

THE LEAD-ZINC MINE OF ZAWAR IS situated about 25 miles by road from Udaipur, Rajputana. Two picked samples of ore from the mine have been investigated and the results of the tests have been recently reported (*Trans. Geo. Met. Inst. of India*, 1949, 45, No. 1).

The first ore sample (—100 mesh) analysed to the following:

|                                | %     |
|--------------------------------|-------|
| Pb                             | 14.10 |
| Zn                             | 6.97  |
| Fe                             | 2.67  |
| Insol.                         | 41.52 |
| CaO                            | 9.60  |
| MgO                            | 5.74  |
| S                              | 6.23  |
| Sb                             | 0.064 |
| Cd                             | 0.059 |
| Al <sub>2</sub> O <sub>3</sub> | 0.21  |

A second ore sample investigated had an assay value of 0.20 per cent lead and 5.3 per cent zinc.

Concentration of the ore by gravity methods—jigging and tabling—and by flotation, was tried. In the case of the second ore sample, the flotation tests indicated that the lead, although present in low amounts, could be successfully recovered as a lead concentrate of reasonably good grade.

On the basis of the experimental work on ore samples containing 0.2 to 2.0 per cent lead and 4.0 to 10.0 per cent zinc, a flow sheet for a pilot mill to treat 6 tons of ore per hour has been drawn up and machinery for such a plant has been specified. Recoveries from this plant could be expected to exceed 90 per cent of the lead in a concentrate assaying lead, 66 per cent; zinc, 6 per cent; and 85 per cent of the zinc in a concentrate assaying zinc, about 63 per cent and lead, less than 0.5 per cent.

The silver content of the lead concentrates is so low (approx. 6.5 oz./ton) that recovery of silver would not be justified.

The cadmium-content of the zinc concentrates is such that if electrolytic methods of zinc production were adopted, a valuable cadmium by-product could be obtained.

### De-enamelling

THE SALVAGING OF PORCELAIN enamel parts, not suitable for repair, by de-enamelling is described (*Amer. Cer. Soc. Bull.*, 1949, 28, 183). The process can be applied if it is possible to salvage the formed-metal shapes involved at a cost less than, or equal to, the metal shapes involved, fabricating cost, and if the salvaged metal is suitable for re-enamelling. Naturally, in times when metal shortages are involved, the salvaging of parts to maintain production operations will considerably outweigh many of the cost considerations. Additional factors involved are concerned with the cause of final rejection of the part in question, that is whether the fault is due to a flaw or failure in the part itself which is not repairable, or whether it is due to failure in the enamelling operation. There is no economic advantage to de-enamel parts which cannot be salvaged.

Two methods are employed: (1) chemical solution; and (2) mechanical removal. In many cases, de-enamelling procedures combine both methods. For the chemical dissolution of enamels,

the following procedures are employed: (1) acid de-enamelling employing hydrochloric, sulphuric, acetic and hydrofluoric acids, and combinations thereof; (2) the use of caustic alkalies, either molten or in boiling water solutions. In some cases combinations of acid and alkali treatments have been employed.

Of the mechanical means employed, the most satisfactory has been the use of sand-blast.

In one method employing sulphuric acid, the ware was placed in a tank containing acid at a temperature of 140° to 150°F., held at this temperature for 1 hr. and then allowed to cool. De-enamelling was reported to be complete after 12 hr. A recent process consists in immersing the ware to be de-enamelled in a 10 per cent sulphuric acid solution with 0.03 per cent of a commercial inhibitor. The acid is held at 180°F. and the ware remains in the tank for 30 to 50 min. Following this operation, the ware is rinsed in a hot-water spray, neutralized in a second spray, and dried and sand-blasted to remove sludge and any enamel which has not been taken off. This procedure appears to hold considerable promise for de-enamelling non-acid-resistant coating.

In the process employing molten caustic soda (*U.S. Patent*, 528,156) the ware is de-enamelled in a large heavy-walled steel tank, electrically heated and filled with molten caustic soda. The process is rapid during the first hour or two, de-enamelling being complete in a few minutes; but after 5 or 6 hr., the time required increases. So, it is customary to stop the operation after 8 hr. and allow the sludge to settle. Improvements incorporated in the procedure are: (1) the caustic bath is heated by electrical resistance, the current being passed through the bath; and (2) caustic soda is mixed with other ingredients which help to control the flow current and prolong the life of the bath.

The method is expensive and the removal of adhering caustic soda is difficult. No lead enamels can be used in this process.

A boiling solution of caustic soda (5 to 15 lb. per gal. of water) has found the most general use in de-enamelling processes. The ware is immersed in the boiling solution for 10 to 12 hr. during which period either the ware is agitated or the solution is circulated. Following de-enamelling, the ware is normally cleaned by high-

pressure spray, in some cases followed by wire brushing or light sand-blasting. This method has not found much favour as it is often difficult to produce first-quality ware on re-operation.

Sand-blasting usually employed to remove porcelain enamel from cast-iron surfaces has several advantages. It provides a very high-quality surface for the re-enamelling operation, eliminating the possibility of nickel-flaking, caustic carry-over and blistering which have been the major defects encountered with chemically de-enamelled ware. It has been found that by controlling the orifice size, air pressure, and the angle of incidence of the blast, it is possible to sand-blast ware of 24 gauge thickness without undue difficulty. When the blast is directed at an angle of 15° to 20° from the horizontal, warping of the ware is eliminated. Distortion is further minimized by working across the piece from front to back so that there is always a coating of enamel ahead of the sand stream.

### Properties of Some Indian China Clays

INVESTIGATION OF THE MINEROLOGICAL and colloidal nature of 4 important Indian China clays, viz. Rajmahal clay, Kasimbazar clay, Travancore clay and Banda clay, by chemical analysis, electro-dialysis, viscosity measurements, differential thermal analysis and examination by X-ray and the petrographic microscope, has been recently completed (*Amer. Cer. Soc. Bull.*, 1949, 28, 187). The results of this investigation show that these clays are essentially kaolinite and had largely the properties of typical China clay.

Tables I and II give the chemical analyses of the untreated and electro-dialysed clays.

The theoretical mineral constitution of these clays, as calculated from the chemical analyses of the untreated clay, is given in Table III.

The morin dye test was used to detect the presence of free aluminium ions in the clear water extract of the clays and it can be used as an indirect measure of stability. The clay is leached by soaking in dilute hydrochloric acid overnight and then taking the supernatant liquid for test. The liquid is neutralized with caustic soda and then made slightly acidic. On addition of a few drops of morin dye, a greenish-yellow fluorescence is observed under



ultra-violet light if aluminium ions are present. The greater the intensity of the fluorescence, the greater the number of free aluminium ions in the test solution. The dye test shows that Kasimbazar clay is the least stable, Banda and Travancore clays are slightly more stable and Rajmahal clay is still more stable in so far as it loses its aluminium ions with difficulty.

Thermal analysis of the clays showed that these clays consist mostly of kaolinite. The Banda and Travancore clays contain larger percentages of kaolinite as their intensity in reaction is greatest. The Rajmahal clay comes next and the Kasimbazar clay shows the least intensity, thereby showing the presence of considerable amounts of other minerals.

Both untreated and electro-dialysed clays were examined by the powder method in a General Electric XRD X-ray unit. The  $d$  values of these clays were compared with the  $d$  values of the Langley clay from North Carolina

which was taken as the standard kaolinite. Except Kasimbazar clay, the others gave peaks at the same angles and almost the same intensity as the standard. Quartz, feldspar, mica, etc., usually associated with the clays, especially with kaolinite, were not detected in the three clays. Kasimbazar clay, in addition to kaolinite peaks, gave peaks for other minerals, particularly muscovite.

Petrographic examination was conducted on crushed clays (100 mesh). The results showed that Rajmahal clay was fine grained, and very few hexagonal kaolinite crystals were seen at high magnification. Grains of quartz were present. The heavy residue consisted of clear, rounded quartz, with a few flakes of muscovite and very few grains of magnetite. Banda clay was coarser in texture. Kaolinite "shrimps" and flakes were common. Rutile, titanite, tourmaline, magnetite, zircon and quartz were found in the residues. Kasimbazar clay showed crystals of kaolinite, quartz and muscovite. The residues contained mica,

quartz and feldspar. Travancore clay was very fine grained, and it was difficult to observe single crystals although occasional typical kaolinite "shrimps" were found. The residues contained very small grains of quartz, both round and angular, and few flakes of mica.

Particle-size measurements indicate that the Travancore clay has the least material in the range above 10 microns, the Banda clay is only slightly coarser, and that the Kasimbazar clay is the coarsest of the group. Rajmahal clay has an appreciable content of relatively large grains and, at the same time, the largest amount of fine material below 1 micron. These conclusions have been confirmed by electron-microscope studies.

A study of the colloidal behaviour of the clays by viscosity and  $pH$  measurements on samples of electro-dialysed clays shows that they show little base-exchange capacity, which is characteristic of kaolinite.

### Impervious Crucibles

CRUCIBLES WITH IMPROVED STRUCTURE and highly impervious lining have been developed at the U.S. National Bureau of Standards (*Chem. Age*, 1949, 61, 13).

The new crucibles can withstand attack by highly corrosive melts on account of minute interconnecting passages between their pores and as such can meet the exacting requirements peculiar to the production of optical glass and the smelting of highly corrosive high-lead glazes and enamels.

In the method employed, the clay casting slip—the mixture of dry ingredients with the proper amounts of deflocculants and water (to give good flow characteristics)—is poured into plaster-of-Paris moulds consisting of two parts, a shell and a core, locked in position before the pot is cast. During the initial stage of casting, the mould is slightly tilted to lessen the tendency to entrap air bubbles between the lining and the pot. After the bottom is filled, the mould is levelled and rocked lightly until the pot is completely cast.

Lining of the pots is applied before the pots are cast. In other words, the lining slip is first poured around the core of the mould, which is removed and transferred to the shell. The pot is then cast in the same manner as for unlined pots. Double and triple layers of lining, up to a

TABLE I—CHEMICAL ANALYSIS OF THE UNTREATED CLAYS

|                                                  | RAJMAHAL<br>CLAY<br>% | KASIMBAZAR<br>CLAY<br>% | BANDA<br>CLAY<br>% | TRAVANCORE<br>CLAY<br>% |
|--------------------------------------------------|-----------------------|-------------------------|--------------------|-------------------------|
| SiO <sub>2</sub>                                 | 54.07                 | 47.71                   | 43.92              | 44.84                   |
| Al <sub>2</sub> O <sub>3</sub>                   | 32.10                 | 34.93                   | 41.12              | 40.70                   |
| Fe <sub>2</sub> O <sub>3</sub>                   | 0.30                  | 0.92                    | 0.72               | 0.21                    |
| CaO                                              | 0.69                  | 1.62                    | Trace              | 0.66                    |
| MgO                                              | 0.12                  | 1.92                    | Trace              | Trace                   |
| Alk.                                             | 0.35                  | 1.10                    | 0.53               | 0.20                    |
| Ignition loss                                    | 12.37                 | 11.80                   | 12.71              | 13.39                   |
| Total                                            | 100.00                | 100.00                  | 100.00             | 100.00                  |
| SiO <sub>2</sub> ·Al <sub>2</sub> O <sub>3</sub> | 3.00                  | 2.30                    | 1.80               | 1.80                    |

TABLE II—CHEMICAL ANALYSIS OF THE ELECTRO-DIALYSED CLAYS

|                                | RAJMAHAL<br>CLAY<br>% | KASIMBAZAR<br>CLAY<br>% | BANDA<br>CLAY<br>% | TRAVANCORE<br>CLAY<br>% |
|--------------------------------|-----------------------|-------------------------|--------------------|-------------------------|
| SiO <sub>2</sub>               | 55.44                 | 50.21                   | 45.36              | 45.61                   |
| Al <sub>2</sub> O <sub>3</sub> | 30.67                 | 35.60                   | 41.98              | 40.40                   |
| Fe <sub>2</sub> O <sub>3</sub> | 0.14                  | 0.21                    | 0.09               | 0.01                    |
| CaO                            | Nil                   | Trace                   | Nil                | Trace                   |
| MgO                            | Nil                   | Trace                   | Nil                | Trace                   |
| Alk.                           | Nil                   | Trace                   | Nil                | Trace                   |
| Ignition loss                  | 13.75                 | 13.92                   | 13.01              | 13.86                   |
| Total                          | 100.00                | 100.00                  | 100.00             | 100.00                  |

TABLE III

| MINERAL           | RAJMAHAL<br>CLAY<br>% | KASIMBAZAR<br>CLAY<br>% | BANDA<br>CLAY<br>% | TRAVANCORE<br>CLAY<br>% |
|-------------------|-----------------------|-------------------------|--------------------|-------------------------|
| Orthoclase        | 1.7                   | 6.1                     | 2.72               | 1.1                     |
| Anorthite         | 3.6                   | 7.3                     | ...                | 3.3                     |
| Haematite         | 0.3                   | 0.8                     | 0.6                | 0.2                     |
| MgCO <sub>3</sub> | 0.3                   | 3.6                     | ...                | ...                     |
| Kaolinite         | 78.0                  | 76.3                    | 89.2               | 92.0                    |
| Quartz            | 15.8                  | 5.9                     | ...                | ...                     |
| Alumina           | ...                   | ...                     | 7.5                | 3.3                     |

total thickness of  $\frac{1}{4}$ " or more, are sometimes applied.

The additional step that has been introduced for lining crucibles consist in spraying a coat or coats of clay slip over the lining to seal incipient cracks. Such faults often open during use and permit a fluid glass or glaze to penetrate into the walls or, in some instances, to leak through the crucible.

The cores are removed as soon as practicable after casting in order to prevent cracking of the lining—timing of this operation is critical—and the shells are removed when the pots have set sufficiently to retain their shape. The pots are then air-dried and finally, immediately before use, are heated to approximately the temperature of the melt they are to contain. A carefully regulated heating schedule has to be followed and the rise in temperature controlled in accordance with the relative speed with which the pot can be heated through certain ranges without damage.

In addition to its unusual pore structure, the new crucible has low gas permeability (even though it shows a porosity of 22 per cent after firing at 1435°C.). The crucible has a somewhat low resistance to thermal shock.

## High-temperature

### Laboratory Kiln

AN INEXPENSIVE ALUNDUM-LINED kiln that can be fired to 1950°C. in about 40 min. with uniform temperature control is described (*Amer. Cer. Soc. Bull.*, 1949, **28**, 192). The kiln is a small up-draft laboratory kiln, unique in both ease of firing and replacement of lining. Standard gas-welding equipment was found suitable as it provided high flame temperatures with a minimum of combustion space.

The interior of the kiln (9" x 9" x 7") is lined with standard 9" straight brick of high-purity (99 per cent), electrically fused alumina. These had been fired to cone 35 (1785°C.) in the course of manufacture. The lining was insulated with 2900°F. insulating brick. The use of high-purity alumina refractories not only allows high temperatures to be reached but permits unusual kiln atmospheres without the danger of flashing and staining caused by volatilization of impurities in furnace lining. Temperatures as high as 1950°C. were reached without lining injury. For temperatures above 1900°C., elec-

trically fused magnesia and electrically fused stabilized zirconia are suitable. Under strongly oxidizing conditions, magnesia is satisfactory. Under reducing conditions, however, there was a tendency for magnesia to volatilize at high temperatures.

Replacement of refractories in the kiln is easily accomplished as the lining is not cemented and all the parts can be readily lifted out and new posts set in. This was found to be a distinct advantage as it permits free expansion and contraction of the lining without development of cracks.

In 40 min. a temperature of 1950°C. was reached with either oxygen-propane torches equipped with multi-flame heating tips or oxygen-acetylene torches equipped with standard welding tips. The former permits slower heating schedule and more uniform temperatures. For temperatures below 1,000°C., small gas-air burners can be used.

The advantages of using propane in lieu of acetylene are numerous. With propane multi-flame tips operate more successfully. Because propane is stored as a liquid in tanks, changes are less frequent, and it is safer to store and use than acetylene. The main advantage with acetylene is its higher flame temperature.

Torches should be so placed that the flame does not impinge on the ware. The burner ports are cut in the brick and should not be more than  $\frac{1}{2}$ " larger than the burner-tip diameter. It is advisable to set the ware on refractory pedestals or trays about  $\frac{1}{4}$ " above the hearth to permit free circulation. The burner tips are placed flush with the outside surface of the inner refractory lining and concentric with the burner ports. The tips are water cooled. These precautions prolong their useful life. The tips have 2 or 3 turns of copper tubing which are silver soldered. With acetylene, welding tips having an orifice 0.067" to 0.052" are required. With propane, No. 4 multi-flame heating tips were used. The kiln has only 2 burners firing from opposite sides.

### Electric Gauge

AN ELECTRIC GAUGE HAS BEEN developed for measuring the bores of small guns or tubes of about  $\frac{1}{8}$ " inside diameter (*J. of Res.*, 1949, **42**, 461).

The gauge operates on the principle of mutual inductance of two solenoids mounted on each

prong of a fork. A constant voltage is maintained in the primary coil. The current induced in the secondary is a measure of the distance between the two coils. The gauge is calibrated with rings of known diameter and the diameter of a tube or gun bore is read directly on the dial of the instrument.

The gauges so far available, though quite satisfactory for production control, consume much time, are unwieldy and are not quite accurate for research work. The electric gauge described is relatively inexpensive, quick and accurate, gives continuous reading and has a sensitivity of 0.002" which holds over the entire range of 0.02" without recalibration. A wider range than 0.02" can be obtained without recalibration by exchanging the gauge points for another pair and taking the difference in height of the points into consideration. The nearer the gauge points are situated to the Y of the fork, the greater will be the mechanical magnification of their motion by the solenoids. A variation in the use of the gauge is to connect the output to a recording potentiometer instead of to the micro-ammeter. In this way a continuous record of the profile of the bore of a tube can be obtained. This feature of the instrument has, however, not been fully developed.

### New Synthetic Fibre

FIBRES FROM COTTON-SEED PROTEIN and chemically modified cellulose produced experimentally at the U.S. Bureau of Agr. & Ind. Chemistry's Res. Lab., are described (*Chem. Age*, 1949, **61**, 64).

Cotton-seed fibre is obtained by a special acid treatment of the proteins from solvent-extracted cotton-seed meal. This changes the structure of the protein, corrects its tendency to gel or lump in untreated dispersions, and makes it suitable for spinning.

The fibres from sodium carboxymethyl cellulose (a soluble compound made from wood or cotton cellulose) are derived by extruding its solution in water through a spinnerette into a bath containing the salt of one or more of heavy metals. The fibres being soluble in soapy water or other weak alkali solutions (similar to alginate fibres from alginic acid, a constituent of sea weed), are considered suitable for use as a spacing agent—to provide the "missing threads"—in specially woven fabrics.

### Drying Oil Improver

A SYNTHETIC ORGANIC CHEMICAL, "dryene", for improving naturally occurring drying oils, has been developed by *Carbide & Carbon Chemicals Corp.*, New York (*Chem. Eng.*, 1949, **56**, 172).

A low molecular weight polyester, "dryene" is a viscous liquid of low volatility. Under conditions of a varnish or alkyd cook, it undergoes ester interchange and dehydration to yield a product structurally similar to that obtained from a 6-carbon dienolic acid. For purposes of determining the balance between hydroxyl and acid groups in an alkyd formulation, the up-grader may be assumed to be an acid with a combining weight of 112.

The action of "dryene" is based on the introduction of increased conjugated unsaturation into a finish formulated from a soft-drying oil, permitting a high degree of cross-linking by the formation of carbon-to-carbon linkages rather than ester linkages, the former being more stable than carbon to oxygen linkage, typical of phthalic anhydride-glycerol alkyls.

The improved drying characteristics of the oil reacted with the up-grader make it of great interest in treating oils to produce alkyd resin varnishes with improved alkali and water resistance, more rapid set, improved mar resistance and increased toughness and durability. The same results are obtained by incorporating "dryene" in varnishes and alkyd resins.

### Modified Drying Oils

MODIFIED OILS HAVING SPECIAL setting properties have been prepared by reacting soya-bean and linseed oils with dicyclopentadiene (*Chem. Eng.*, 1949, **56**, 186).

Dicyclopentadiene is depolymerized by heat and the monomer reacts with the double bonds of the oil in accordance with the Diels-Alder reaction. Linseed and soya-bean bases have been called "Cykelin" and "Cykelsoy" respectively.

Both the products have practically the same properties and are used for similar purposes except that the linseed base has lighter colour and sets quicker than the soya-bean base. Both set in a few hours, dry hard overnight, give films resistant to water and alkali, and are miscible with bodied or unbodied oils. Their use is specially recommended in varnishes and enamel vehicles.

### Foam Inhibitors

THE TENDENCY OF LUBRICATING oils to foam in use, which reduces lubricating efficiency and impedes oil circulation in I.C. engines has been studied. Recycling of the oil through a column of activated charcoal at 50°C. or lower, reduced foam to  $\frac{1}{15}$ th the volume of the foam from untreated oil. Activated manganese dioxide, silica gel, and "porocell", when packed in columns for recycling, also defoam the oil to a lesser extent (*Chem. Age*, 1949, **61**, 47).

### Insect-proofing of Cotton Flour Bags

DISCOVERY OF A NEW TREATMENT to make cotton flour bags insect-proof without impairing the quality of the flour is claimed by *U.S. Dept. of Agriculture*. The most effective treatment is stated to consist in spraying 10 mg. of pyrethrins alone or mixed with 100 mg. of piperonyl butoxide per sq. ft. of cloth. Bags so treated admitted no insects during seven months of exposure to *Cadelle*, the Mediterranean flour moth (*USIS*).

### Wood Preservative

A NEW CHEMICAL WOOD PRESERVATIVE, copperized chromated-zinc chloride, developed by *Koppers & Du Pont*, is described (*Chem. Eng.*, 1949, **56**, 186).

Copperized CZC containing 73 per cent zinc chloride, 20 per cent sodium bichromate, and 7 per cent cupric chloride possesses greater permanence of its toxic constituents than CZC. The metal corrosion factor, the glow characteristics, the handling and control, however, of the two preservatives are reported to be about the same.

### Electronic Filing Machine

AN ELECTRONIC MACHINE THAT can file away documents on 10,000,000 different subjects, then automatically select documents relating to one subject and make a picture record of them in a few minutes, was recently demonstrated at *U.S. Dept. of Agriculture*, Washington.

The new machine, called the "Microfilin Rapid Selectro", combines the actions of a motion-picture project photo-electric eye, and a high-speed camera.

Documents to be filed in the machine are first micro-filmed on conventional 35 mm. film. The

contents of 50,000 ordinary library cards can be put on a single reel of film.

While a document is being micro-filmed, a code pattern, consisting of black and white squares, is filmed beside it to indicate the subject to which the document relates.

To operate the machines, a master code card is inserted into a slot in the machine. On the master card is a code pattern identical with that appearing besides all micro-filmed documents relating to a subject, say penicillin. As a reel of micro-filmed documents passes rapidly through the machine, a photo-electric eye scans them, and when a document having a code pattern similar to that of the master code card for penicillin appears, it breaks an electronic beam, tripping the shutter of a super-fast camera. The camera instantaneously takes a picture of the document. (*USIS*).

### New Colour-computing Device

A NEW DEVICE FOR MEASURING and analysing a large number of colours with precision and rapidity known as General Aniline-Librascope Tristimulus Integrator is described (*J. Franklin Inst.*, 1949, **247**, 541).

The integrator, an electronically controlled ball and disc mechanism, integrates the three components of colour. First the light, which is energy radiating from the sun or the lamp, secondly the dyed or coloured object under observation which reflects varying percentages of light of different wavelengths, and finally the eye itself. Internationally accepted data on light and the eye are available and reflectance of the object is measured by a spectrophotometer. The mathematical combination of these factors, hitherto involving multiplication at scores of wavelengths and addition of the products, is achieved in 2.5 min.

### Improved Plough

THE RECENTLY INVENTED DOUBLE plough consisting of double bottoms of two standard *desi* ploughs (suitably coupled by means of an iron framework and pulled by a single central beam) has been further modified by the *Indian Agricultural Research Institute*. The new plough can tackle with ease the most difficult fields infested with weeds or left with stubbles of previous crops.

### Ciba Science Centre

*Ciba Ltd.*, of BASLE, SWITZERLAND, have established a unique institution in London, the Ciba Foundation for the Promotion of International Co-operation in Medical and Chemical Research. The Foundation will hold informal conferences from time to time to which scientists will be invited, and lecturers from abroad will be asked to address. Besides, grants will be made to younger men of outstanding merit and enable them to study methods of research. The trustees will provide accommodation for other scientific bodies and universities in the buildings, for purposes within the objects of the Foundation (*Chem. Age*, 1949, 60, 915).

### Indian Standard for Plywood Tea-chests

INDIAN STANDARD SPECIFICATION for Plywood Tea-chests lays down the definitions used in the plywood industry and trade, standard specifications for plywood and dimensional standards for tea-chests and components, and test methods for plywood for tea-chests.

Of the seven appendices included in this standard, the first one catalogues the various timbers that are to be used for plywood and the treatment to be given to them in the manufacture of plywood for tea-chests. The rest of the appendices detail the various methods of test connected with this standard and the form of report of results of tests. A useful table, laying down the dimensions of component parts for tea-chests of various sizes, is included.

The publication is priced at Rs. 1/12. Copies can be had from the Secretary (Administration), *Indian Standards Institution*, Old Secretariat, Delhi 2.

### Wool Grading

STANDARDS FOR GRADING OF Indian wool have been drawn up by the Textile Division Council of the *Indian Standards Institution*. The step will enable the Indian wool-exporting industry to solve problems arising from the vari-

ability and the uncontrolled quality of wool that is being sent out. The standard apply to wool produced in India and specify grades according to colour and quality. Specifications for packing materials are also included.

The publication is priced at As. 8 and can be had from the Secretary (Administration), *Indian Standards Institution*, Old Secretariat, Delhi 2.

### Announcements

*Australian Fellowships for Indian Students* — Australia has offered 5 scholarships for the year 1950 to Indian students for higher training in industry and technology and postgraduate studies in Australia.

*UNESCO Fellowships* — India has received during 1947-48, 18 fellowships — 15 awarded by U.N. and 3 by UNESCO — for studies in social welfare, health work, and physical and biological sciences.

*An International Institute for Research in Sound* has been established in Paris to co-ordinate world research in acoustics. For the time being, the Institute will concentrate on problems such as design of concert halls, distribution of orchestral instruments and fight against noise in large cities.

### REPORTS FROM STATES & PROVINCES

(Continued from page 425)

heavy rains would preclude composting operations) is expected to benefit an area of 10,000 acres each year.

#### Thermal Station at Kurnool

A SCHEME FOR THE INSTALLATION of a central thermal station at Kurnool at a cost of Rs. 145.27 lakhs has been sanctioned. The station will have two 5,000 kW. generating sets with sub-stations at Dhane, Gooty, Pamidi and Anantapur and will cater to power requirements of important industries being planned in and around Kurnool and also provide scope for large-scale rural development.

### NEW DELHI

#### Wheat Rust Control

A COMPREHENSIVE SCHEME FOR research on an all-India basis aiming at evolving a type of wheat which is simultaneously resistant to all the various kinds of rust — keeping in view the botanical, mycological and agronomical aspects of the problem — has been sanctioned by the Central Ministry of Agriculture. A sum of Rs. 8,58,000 has been sanctioned for the purpose for the first 5 years.

So far, research has been concentrated mostly in finding out the physiology and etiology of the 3 kinds of rust — black, brown and yellow. The Wheat Rust Control Committee, appointed in 1947, recommended trial, as a short-term scheme, of the theory that while infection is killed in the plains by intense summer heat, it is carried from season to season by the summer sowings of wheat and barley mainly in the Nilgiris and Palaney Hills. On this basis, it is held that if sowings of wheat and barley in summer are completely stopped in Peninsular India, the source of infection will disappear and the menace of rust will be mitigated if not completely eradicated. This theory is being given a trial in consultation with provincial and state governments concerned.

Seed-mixture trial is the second short-term scheme recommended by the Committee and aims at mixture of those varieties of seeds which are claimed to be tolerably resistant to the 3 kinds of rust individually. This scheme is now a part of the Grow More Food Campaign.

Under the long-term scheme now sanctioned, research would be conducted on the following lines: (a) finding out physiological races of the 3 kinds of rust and maintaining their live culture; (b) breeding of wheat varieties resistant to all the 3 types of rust; and (c) ascertaining whether the effects of rusts can be minimized by changing cultural and manurial practices, e.g. cropping, spacing and so forth.

# Reports from States & Provinces

BOMBAY

## Potato Cultivation

TO AUGMENT THE PRODUCTION OF potatoes in the province, the Government is supplying cultivators 131,000 bags of seed potatoes, 5,000 tons of powdered groundnut oil cake and 87,000 bags of special manure mixture during the *rabi* season and 35,000 bags of seed potatoes and 4,300 tons of powdered groundnut oil cake during the 1950 *kharif* season.

A committee of 8 non-official representatives will arrange procurement and despatch of seed potatoes from Simla Hills during the season.

## MYSORE

### Plans for Development of Technical Education

A CONFERENCE PRESIDED OVER BY the Dewan of Mysore and attended by the heads of technical institutions and other state authorities was recently held in Bangalore to discuss ways and means of developing technical education in the State.

The conference first considered the question of co-ordinating all technical education in the State and steps to be taken for avoiding duplication of instruction in various institutions. The conference emphasized the need for practical training in the various courses of study and the need for further applied training for students in industrial establishments before the diploma is given.

The conference recommended the establishment of an advisory body, the Board of Technical Education, with the following functions:

(i) To review from time to time the courses of studies and practical instructions given in various technical institutes;

(ii) to suggest alterations or modifications in such instruction;

(iii) to propose courses of study and instructions in the new technical institutes that may be established;

(iv) to advise on the opening of new technical institutes at any centre in the State; and

(v) generally to advise on all matters connected with technical education through such institutes.

The appointment of a Director of Technical Education to supervise the working of the various institutes and to advise the Board and the Government on the co-ordination of the activities of these institutes was recommended by the conference.

The need for establishing a school of arts and crafts for imparting instruction in fine arts was confirmed by the conference. It was suggested that the Chamarajendra Technical Institute at Mysore may form the nucleus of such an institute.

The conference also recommended that when all the technical institutes are functioning, a Central Technical Examination Board may be formed.

## RAJASTHAN UNION

### Tapping Sub-soil Water

POSSIBILITY OF FINDING SUB-SOIL water in Samdari, a small town in Marwar, 50 miles west of Jodhpur, is being explored by irrigation engineers of the Government of India.

Samdari, situated at the apex of the Luni river is in its depression area. From investigations already carried out, the sponsors of the scheme hope that in a 40-mile stretch of land, beginning from Samdari to Tilwara, fresh water exists which, at a conservative estimate, would be able to irrigate immediately at least 8,000 acres of land besides providing sweet water to thousands of people in this area.

Explorations made at a spot 3 miles from Samdari railway station resulted in a find of water (6 ft. from the surface) whose level was unaffected even after pumping out for several hours at 40,000 gal. per hr. indicating thereby that the rate of infiltration from the sub-soil was equivalent to the rate of pumping.

Samples of water from this area have been found sweet. 20 miles down-stream from Samdari, water becomes slightly brackish although it is considered good enough for cultivation. Besides the saline content of water is considerably reduced during and after the rains as rain water dilutes the salts, making cultivation of *rabi* crops possible.

On the question of water supply depends the development of this fertile area for agricultural purposes. Water will also make afforestation possible so that passing monsoon from the Arabian Sea is attracted, thus increasing the annual rainfall. The investigations now proceeding are, therefore, of vital importance to Marwar.

## EAST PUNJAB

### Improved Types of Wheat & Barley

THE EAST PUNJAB AGRICULTURE Department has evolved 2 improved types of wheat and 1 of barley which are fairly resistant to rust and are high yielders. One variety of wheat, No. 3970, yields 1.8 md. to 2.2 md. more than the standard varieties, No. C.591 and C.250. This variety is also fairly resistant to all the 3 types of rust. The other high-yielding type yields 28.4 md. of grain per acre as compared with 23.15 md. by C.591.

The new variety of barley evolved possesses bright and plump grain and gives higher yields than the common barley varieties, T.4 and T.5. This variety, which is resistant to loose-must disease, is of a good malting quality. Its yield per acre in irrigated areas is 41.3 md. as against 38.6 md. of the T.4 variety.

## MADRAS

### Compost Scheme

A 2-YEAR SCHEME FOR PRODUCTION OF 88,000 tons of compost in rural areas of Madras at a cost of Rs. 99,000 has been announced by the Director of Agriculture.

The peasants themselves will excavate the pits required and a subsidy of Re. 1 per ton will be paid to them. The ripe compost will be made available to the peasant on his own land at a uniform rate of Rs. 2 per ton. Plans for efficient transport of compost are also being drawn up.

The scheme which is to be worked in all the districts except South Kanara and Nilgiris (where

(Continued on page 424)

# INDIAN PATENTS

The following is a list of a few of the Patent Applications accepted in the *Gazette of India*, Part II, Section 1, for August 1949. Patents from the *Council of Scientific & Industrial Research* are indicated by an asterisk\*.

## Organic Chemicals

- \*39426. DEY, MALLER, PAI & UDUPA: An improved method for the production of 2:4-diaminophenol: *Reduction of m-nitraniline in sulphuric acid at a copper cathode in the presence of copper sulphate.*
- \*39427. DEY, MALLER & PAI: An improved method for the production of 2:4-diaminophenol: *Electrolysing 2:4-dinitrophenol in 30 per cent sulphuric acid at copper cathode.*
- \*39428. DEY, MALLER & PAI: An improved method for the production of 4-amino meta-cresol: *Electrolytic reduction of ortho-nitro-toluene in dilute sulphuric acid in presence of amalgamated monel cathode and catalyst.*
- \*39429. DEY, MALLER & PAI: An improved method for the production of 4-amino-ortho-cresol: *Electrolytic reduction of meta-nitro-toluene in dilute sulphuric acid in the presence of amalgamated monel cathode and catalyst.*
- \*39441. DAMODARAN, RANGACHARI & RAMAKRISHNAN: An enzymic method for the production of calcium gluconate: *Biologically oxidizing glucose in the presence of calcium carbonate by means of glucose oxidase obtained from Aspergillus niger.*
- \*39442. DAMODARAN & SUBRAMANYAN: An improved method for the manufacture of sorbose from sorbitol: *Bacterially oxidizing sorbitol with acetic bacteria in which the medium contains extract of bran or of germinated seeds.*
- \*40261. DEY, MALLER & PAI: Production of 6,6'-dichloro-3',3'-diethoxy-benzidine (Dichloro-o-diphenetidine): *Electrolytically reducing 2-nitro-4-chloro phenetole in aqueous caustic soda removing the hydrazo compound formed by a solvent and treating the hydrazo compound with an acid.*

## Instruments (Professional, Scientific & Controlling)

40169. THAPAR & Co. LTD.: Mechanism for manufacture of sheets, block or like built up mica products: *Sprinkling upon conveyor belt thin-risted mica flakes, supplying adhesive solution to conveyor, when the product of desired thickness is made, it is cut into lengths.*
40355. KSHIRSAGAR: Improved *bidi*-making machine: *Comprises a leaf feeder, a carrier guided by a lever, a set of rollers fitted to a toothed wheel.*

## Metal & Metal Products

40803. STANDARD OIL DEV. Co.: Method of treating iron ores: *Ore in subdivided form is brought to a high temperature and melted in dilute suspension in hot gases.*

## Paper & Allied Products

39172. ISOLERINGSAKTIEBOLAGET WMB: Method and apparatus for the manufacture of impregnated paper of heat-insulation of multi-layer type: *The paper sheets are dipped in asphalt or bitumen dissolved in a volatile solvent, which is expelled by a current of hot air.*

## Printing, Publishing & Allied Industries

- \*40257. S. SIDDIQUI, J. S. AGGARWAL & MATHUR: Improvements in or relating to manufacture of inks for stamping, printing, stencilling or like purposes: *Resinol or its derivatives, used as sterilizer.*

## Rubber & Rubber Products

41453. DUNLOP RUBBER LTD.: Apparatus for the vulcanization of rubber articles in transitu: *A chamber connected to two inlet and outlet tubular passages, maintained at a constant vulcanizing temperature through which articles pass over an endless conveyor.*
39472. I.C.I. LTD.: Rubber Antioxidants: *Comprising a mixture of bis-(2-hydroxy 3:5-dimethyl-phenyl) alky methanes and corresponding bis-(4-hydroxy-2:5-dimethyl-phenyl) alky methanes.*
- \*39443. UMA SHANKAR: Creaming of rubber latex: *Hot aqueous extract of tamarind seed powder is mixed with latex and allowed to stand until rubber creams up.*

## Textile & Textile Products

41071. THE BRADFORD DYERS' ASSOC. LTD.: Process for reducing the feltability of wool: *Progressive passing of the material through a chamber, continuously feeding halogen gas into the chamber and continuously withdrawing water vapour therefrom.*

## Miscellaneous

40816. CARTER: Separation and extraction of stalk from dry tea: *comprises a pair of parallel co-operating toothed rollers with means for rotating the rollers in opposite directions at different speeds.*
40817. CARTER: Separation and extraction of stalks from vegetable tissue such as dry tea: *Comprises a tabular casing with a roller having one or more helical V-shaped peripheral grooves rotating with clearance within the casing.*
40671. ROY: An improved process for manufacture of pencils: *Casing for the lead rod prepared out of a pliable mass composed of saw-dust or the like made into a dough-like mass by mixing with a binding agent and fed into moulds.*

# Journal of Scientific & Industrial Research

Vol. VIII B

OCTOBER 1949

No. 10

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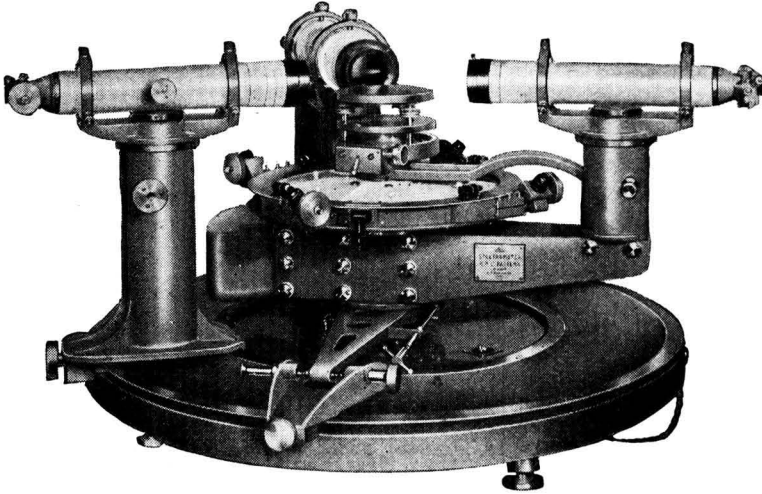
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# Chemical Examination of *Picrorhiza kurrooa* Benth.—Part I

RAM PRAKASH RASTOGI, VISHWA NATH SHARMA & SALIMUZZAMAN SIDDIQUI

Chemical Laboratories, C.S.I.R., Delhi

**P**ICRORHIZA *kurrooa* Benth. (*Scrophulariaceae*; Hindi: *kutki* or *katki*) grows from Kashmir to Sikkim at an altitude of 5,000 to 10,000 feet. The rhizome and the roots are extremely bitter to taste and have been recommended as a substitute for the imported gentian roots. They are extensively used in the indigenous system of medicine as a tonic, a febrifuge and an anti-periodic. In spite of the considerable importance attached to the plant, its chemical investigation has been limited to a preliminary examination by Dymock and Hooper<sup>1</sup> who reported the presence of a "brown, resinoid glucoside", picrorhizin (yield, 15 per cent), which, on hydrolysis with 1 per cent hydrochloric acid, yielded a reducing sugar, and an aglucone, picrorhizetin, a reddish-brown, brittle, resinous, tasteless mass. They further obtained the free aglucone, picrorhizetin, from the plant along with a wax melting at 61°C. Chopra and Ghosh<sup>2</sup> also noted the presence of the bitter glucoside in the drug but were unable to isolate any crystalline product.

As a result of the present investigations, the following products have been isolated from the roots in a crystalline form:

1. A glucosidal bitter principle, kutkin,  $C_{23}H_{28}O_{12}(C_{23}H_{24}O_{10}.2H_2O)$ ; m.p. 211°C.; yield 3.4 per cent on the weight of the dry roots; bitter in a dilution of 1 in 7,500.
2. A non-bitter product, kurrin,  $C_9H_{18}O_9$ ; m.p. 166°C.; yield, 0.5 per cent on the weight of the dry roots.
3. Vanillic acid,  $C_6H_3(OH)(OCH_3)(COOH)$ ; yield, 0.1 per cent on the weight of the dry roots.

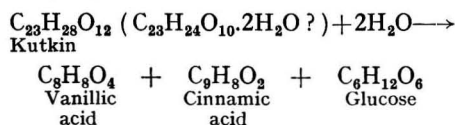
4. Kutkiol,  $C_{40}H_{82}O$ ; m.p. 118°C.; occurring as the acetate,  $C_{40}H_{81}OOC.CH_3$ , m.p. 74°C.; yield, 0.06 per cent on the weight of the dry roots.

5. Kutki-sterol,  $C_{24}H_{40}O$ ; m.p. 124°C.; yield, 0.18 per cent on the weight of the dry roots;  $[\alpha]_D^{25} = -39.0^\circ$  in 1 per cent chloroform solution.

As described in detail in the experimental, the method employed for the isolation of kutkin and kurrin was based on the difference in their solubilities in alcohol. The isolation of kurrin was comparatively easier, but kutkin could be obtained in a pure crystalline form after a lengthy process of purification employing mixed solvents.

*Constitution of Kutkin*,  $C_{23}H_{28}O_{12}$ —Kutkin is soluble in water, methanol or alcohol in the hot, but insoluble in other organic solvents. It contains one methoxy group, but no phenolic group, as it does not give any colouration with ferric chloride in alcoholic solution. It dissolves in dilute alkali or ammonia with a yellow colouration but is not precipitated out on acidification. In conformity with its glucosidal character, it reduces Fehling solution after hydrolysis with dilute mineral acids. Kutkin is, moreover, a  $\beta$ -glucoside, as it is also hydrolysed by emulsin under the usual conditions. No crystalline aglucone could be obtained from the products of its hydrolysis with mineral acids due to considerable resinification occurring during hydrolysis. On treatment, however, with dilute aqueous alkali at the ordinary temperature or with saturated baryta solution on the water bath, it yielded 2 crystalline substances melting at 202°C. and 133°C. respectively, along with glucose

which was identified through its osazone. The yields of the two crystalline products were very much better when the hydrolysis was carried out with baryta solution. The substance melting at 202°C. could also be obtained in a small yield through hydrolysis with 1 per cent acetic acid in a sealed tube at 125°C. The analytical data and the chemical characteristics of the substances melting at 202°C. and 133°C. as well as of their derivatives showed them to be vanillic and cinnamic acids respectively. This finding was confirmed through the mixed melting point determinations of the two products and their derivatives with authentic samples of vanillic and cinnamic acids and their corresponding derivatives. The course of hydrolysis may be represented by the following equation :



Accordingly, kutkin as a glucose ester of cinnamoyl-vanillic acid should correspond to the molecular formula  $\text{C}_{23}\text{H}_{24}\text{O}_{10}$  (requires C, 60.0; H, 5.2). The C and H values for the substance dried at 100°C. *in vacuo* over phosphorus pentoxide (C, 55.0; H, 5.3) are in agreement with the molecular formula  $\text{C}_{23}\text{H}_{28}\text{O}_{12}$  (requires C, 55.6; H, 5.6). This could, however, be accounted for by the presence of 2 molecules of water of crystallization in kutkin, which are not removable at 100°C. Such an explanation should find support from a similar behaviour in the case of Gentiopikrin<sup>3</sup>, a glucoside from *Gentiana lutea* L., which splits off its 1 molecule of water of crystallization only on hydrolysis and not under the usual conditions of drying. Further studies to elucidate this point are in progress but the following tentative structure of kutkin may be put forward at this stage :



As it was of interest to find out whether vanillic and cinnamic acids occur in the drug in a free state, a sample of the powdered drug was Soxhleted with ether after defatting it through extraction with petrol ether. The

etheral extract, on working up in the usual manner, yielded vanillic acid (yield 0.1 per cent), but no cinnamic acid could be obtained from it.

*Kurrin*,  $\text{C}_9\text{H}_{18}\text{O}_9$  — Kurrin is readily soluble in water, sparingly so in methanol and insoluble in the other organic solvents. It does not give a reaction for a free carboxylic group, contains one methoxy group, and 5 (or 6) active hydrogens as determined after Zerewitinoff. It is optically inactive. Kurrin does not reduce Fehling solution either before or after hydrolysis with dilute sulphuric acid. On benzylation, both after Schotten-Baumann and with benzoyl chloride in pyridine solution, it yields only a penta-benzoyl derivative. The analytical data of the acetyl derivative, however, obtained with acetic anhydride and fused sodium acetate indicate the formation of a tetra acetyl derivative with the intramolecular loss of two molecules of water. Action of phenyl hydrazine on kurrin also results in the formation of a triphenyl hydrazone with the intramolecular elimination of two molecules of water. It could not be methylated with either methyl iodide and silver oxide or with dimethyl sulphate and alkali. With formaldehyde and formic acid, no methylation could be effected, but a crystalline product was obtained by this reaction, which conforms to the molecular formula  $\text{C}_9\text{H}_{14}\text{O}_6$  and does not contain the methoxy group present in the original substance. It is recovered unchanged on fusion with 50 per cent potash. Kurrin yields a crystalline semi-carbazone but its micro-N-values are too high for any conceivable formula. Further investigations to clarify these points and elucidate the chemical constitution of kurrin are in progress.

*Fatty Portion*: *Kutkiol*,  $\text{C}_{40}\text{H}_{81}\text{OH}$  — The petrol ether extract of the drug yielded a fatty portion which, on keeping in benzoyl solution, gave kutkiol acetate,  $\text{C}_{42}\text{H}_{84}\text{O}_2$ , m.p. 74°C. On hydrolysis with alcoholic potash, this product gave acetic acid and an aliphatic alcohol,  $\text{C}_{40}\text{H}_{81}\text{OH}$ , melting at 118°C., which has been provisionally named as kutkiol.

On saponification of the fatty residue left on removal of the solvent from the mother liquors of kutkiol acetate, a sterol,  $\text{C}_{24}\text{H}_{40}\text{O}$ , was obtained from the unsaponifiable portion, which melts at 124°C. and appears to be different from the known sterols. It has been provisionally named as kutki-sterol.

**Experimental**

*Isolation*—1 kg. of the finely powdered roots was extracted with petroleum ether and the defatted material was repeatedly percolated with alcohol till the roots were no longer bitter in taste. The total alcoholic percolates were concentrated *in vacuo* below 50°C., and the dark-brown, viscid concentrate was freed of the residual fat by digestion with petroleum ether. The combined petroleum ether extracts, on removal of the solvent, gave 9.6 gm. of fatty matter.

The petroleum ether insoluble residue was taken up in alcohol and the alcoholic solution was kept in the cold, when it gave a crystallizate of crude kurrin which was filtered and washed with alcohol. It melted at 158°C. (decomp.), yield 0.5 per cent.

The combined filtrate and washings from kurrin were treated with ether and petroleum ether which precipitated out darkish non-bitter matter. The supernatant reddish-brown solution was freed of the solvent first on the water bath at the ordinary pressure and finally *in vacuo*. The residue was taken up in alcohol and subjected to a similar procedure of purification through ether and petroleum ether. The alcohol-ether-petroleum ether solution obtained at this stage was kept in the cold, when a crystalline deposit of crude kutkin (m.p. 196°C. decomp.) slowly formed, which was filtered and washed with a mixture of alcohol and ether. The combined filtrate and washings were evaporated off and the residue was again dissolved in alcohol and treated with ether-petroleum ether, when a second crop of the crude kutkin was obtained (total yield 3.4 per cent). The final mother liquors were bitter in taste but failed to give any further crystalline product either from any of the organic solvents or on repetition of the process of purification through alcohol, ether and petroleum ether.

*Kutkin*,  $C_{23}H_{28}O_{12}$ —Crude kutkin (m.p. 196°C. decomp.) was repeatedly crystallized from alcohol and methanol when it was finally obtained in the form of colourless clusters of needles melting at 211°C. Kutkin is soluble in water, methanol and alcohol in the hot, and insoluble in acetone, ether and ethyl acetate. In 1 per cent alcoholic solution, it showed  $[\alpha]_D^{25} = -165.0^\circ$ . Kutkin readily reduces Fehling solution

after hydrolysis with 0.1 per cent sulphuric acid. It does not give any effervescence with sodium bicarbonate solution nor any colouration with ferric chloride. It dissolves in dilute alkali and ammonia with a yellow colouration which turns pink on keeping overnight, but the substance cannot be precipitated out on acidification of the alkaline or the ammoniacal solution. It dissolves in concentrated sulphuric acid with a deep violet colouration and in concentrated nitric acid with a light-yellow colour. It did not suffer any loss in weight on drying at 100°C. *in vacuo* over phosphorus pentoxide and the dried sample, on analysis, gave C, 55.0; H, 5.3;  $OCH_3$  (after Zeisel), 2.0.  $C_{23}H_{28}O_{12}$  requires C, 55.6; H, 5.6;  $OCH_3$  (for one), 3.0 per cent.

*Acetyl Kutkin*,  $C_{31}H_{36}O_{16}$ —0.2 gm. of kutkin, 5 c.c. of acetic anhydride and a few drops of pyridine were refluxed at 135°C. for 2 hr. After working up the reaction product in the usual manner, the acetyl derivative was crystallized from dilute methanol (yield 0.26 gm.). It crystallizes in clusters of needles melting at 170°C. and soluble in methanol, alcohol, acetone, ether and benzene, insoluble in water, and petroleum ether. After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, it gave C, 54.9; H, 5.2; M.W. (Rast), 621. Tetra-acetyl kutkin,  $C_{31}H_{36}O_{16}$ , requires: C, 56.0; H, 5.4; M.W., 664. Penta-acetyl kutkin,  $C_{33}H_{38}O_{17}$ , requires C, 56.0; H, 5.3, M.W., 706.

*Hydrolysis of Kutkin (Isolation of Vanillic & Cinnamic Acids from the Hydrolysate*—

*Solution*—A solution of 0.5 gm. of kutkin in 15 c.c. of saturated baryta solution was heated on the water bath for half an hour. The resulting pink solution was saturated with carbon dioxide and the precipitated barium carbonate filtered off. The clear filtrate was acidified with dilute sulphuric acid and extracted with ethyl acetate. The ethyl acetate solution was washed with water, dried over anhydrous sodium sulphate and freed of the solvent. The residue (0.24 gm.), on crystallization from chloroform, yielded two crystalline products, one (0.15 gm.) sparingly soluble in chloroform in the cold and melting at 200°C., and the other (0.07 gm.) fairly soluble in chloroform in the cold and melting at 126°C.

The aqueous acidic layer from the above working was treated with excess of barium

carbonate, filtered and tested for sugars, as described later.

The crystalline product melting at 200°C. was repeatedly crystallized from chloroform when it finally yielded colourless needles melting at 202°C., soluble in methanol, alcohol, acetone, ether and ethyl acetate, and insoluble in water, petrol ether and benzene. It did not give any colouration with ferric chloride, but dissolved in sodium carbonate solution with effervescence. It is soluble in dilute alkali with a yellow colouration, and is precipitated out unchanged on acidification of the solution. After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, it gave C, 56.8; H, 4.7; M.W. (Rast), 151;  $\text{OCH}_3$  (after Zeisel), 8.0;  $\text{C}_8\text{H}_8\text{O}_4$  requires C, 57.1; H, 4.8; M.W., 168;  $\text{OCH}_3$  (for one), 8.9 per cent. On admixture with an authentic sample of vanillic acid, it did not show any depression in the melting point.

The acetyl derivative (m.p. 141°C.) of the product was prepared in the usual manner with acetic anhydride and fused sodium acetate. On analysis, it gave C, 56.9; H, 5.2; M.W. (Rast), 218;  $\text{C}_{10}\text{H}_{10}\text{O}_5$  requires C, 57.1; H, 4.8; M.W., 210. On admixture with acetyl vanillic acid, it did not give any depression in the melting point.

Demethylation of the product melting at 202°C. with hydriodic acid gave a phenolic acid (m.p. 195°C. decomp.), identical with protocatechuic acid.

The second crystalline product melting at 126°C., obtained from the hydrolysate, after a few crystallizations from hot water finally melted at 133°C. It was soluble in water and most of the organic solvents. It did not give any colouration with ferric chloride, and dissolved in bicarbonate solution with effervescence. It absorbed bromine in the cold in chloroform solution. After drying to constant weight at 62°C. *in vacuo* over phosphorus pentoxide, it gave C, 72.7; H, 5.6; M.W. (Rast), 154;  $\text{C}_9\text{H}_8\text{O}_2$  requires C, 72.9; H, 5.4; M.W. 148.

On oxidation with potassium permanganate in alkaline solution, it gave a smell of benzaldehyde, and from the reaction mixture a crystalline acid was obtained which was identified as benzoic acid. On admixture with an authentic sample of cinnamic acid, the crystalline product melting at 133°C. did not show any depression in the melting point.

*The Sugar Component*—The aqueous solution, obtained after extraction of vanillic and cinnamic acids and neutralization with barium carbonate in the manner described earlier, yielded a residue on evaporation, which gave an osazone of golden-yellow needles melting at 203°C. The osazone did not show any depression in the melting point on admixture with glucosazone.

For a quantitative estimation of the sugar, kutkin was hydrolysed with 5 per cent aqueous sodium hydroxide by keeping for 48 hr. at the room temperature. The pink-coloured reaction mixture was heated with Fehling solution and the precipitated cuprous oxide was filtered in a Gooch crucible, washed, dried and weighed. The weight of the cuprous oxide corresponded to 31.7 per cent of glucose.  $\text{C}_{23}\text{H}_{28}\text{O}_{12}$  requires (for one molecule of glucose) 36.2 per cent.

A 2 per cent aqueous solution of kutkin, on keeping with emulsin at 40°C. for 30 min., turned yellow and reduced Fehling's solution.

*Action of Dilute Acids & Alkali on Kutkin*—Hydrolysis of kutkin with 5 per cent aqueous sodium hydroxide on keeping for 48 hr. at room temperature also yielded the two crystalline products obtained through hydrolysis with baryta solution, but the total yield of the two products was only 30 per cent on the weight of kutkin as against 63 per cent of the theoretical and 48 per cent by the baryta hydrolysis.

Hydrolysis of kutkin with 0.2 per cent of sulphuric acid on the water bath led to considerable resinification and blackening of the solution, and no crystalline product could be obtained from the reaction mixture.

Dilute acetic acid (1 to 3 per cent), in a sealed tube at 125°C., hydrolysed kutkin, and from the products of hydrolysis vanillic acid could be isolated in a yield of 20 per cent. No cinnamic acid could, however, be obtained from this hydrolysate.

*Isolation of Free Vanillic Acid from the Roots*—The ether extract of a sample of the defatted drug was concentrated on the water bath and shaken with dilute sodium bicarbonate solution. The bicarbonate solution was acidified, extracted with ether, the ethereal layer was washed with water and dried over anhydrous sodium sulphate, and freed of the solvent. The light brown residue gave, through chloroform, colourless

needles of vanillic acid melting at 202°C. (yield 0.1 per cent on the weight of the dried powdered roots).

*Kurrin*,  $C_9H_{18}O_9$ —Crude kurrin, melting at 158°C. (decomp.), was repeatedly crystallized from hot dilute methanol, when it finally yielded the pure product in the form of colourless, silky needles melting at 166°C. Kurrin is soluble in cold water, sparingly soluble in hot methanol and nearly insoluble in other organic solvents. It does not reduce Fehling solution either before or after hydrolysis with dilute sulphuric acid and does not give any effervescence with sodium carbonate solution. It readily dissolves in concentrated sulphuric acid and concentrated nitric acid giving colourless solutions. It is optically inactive. Attempts to methylate kurrin with dimethyl sulphate and alkali or with methyl iodide and silver oxide after the method of Irvine and Purdie were not successful. Kurrin was recovered unchanged on heating with dilute sulphuric acid or fusion with 50 per cent potassium hydroxide at 190°C. After drying to constant weight *in vacuo* over phosphorus pentoxide, it gave C, 39.7; H, 7.3;  $OCH_3$  (after Zeisel), 5.5; active H (Zerewitinoff), 1.7, 2.1; mean, 1.9.  $C_9H_{18}O_9$  requires C, 40.0; H, 6.6;  $OCH_3$  (for 1), 5.5; active H, (for 5) 1.8; (for 6 H), 2.2 per cent.

*Penta-benzoyl-kurrin*,  $C_{44}H_{38}O_{14}$ —0.1 gm. of Kurrin was dissolved in 10 per cent sodium hydroxide and 1 c.c. of benzoyl chloride was slowly added to the solution with vigorous shaking and cooling. After completion of the reaction, the resulting product was extracted with ether and the ethereal solution was washed with dilute alkali and then water, dried over anhydrous sodium sulphate and freed of the solvent on the water bath. The residue, on crystallization from methanol, yielded the benzoyl derivative in the form of colourless clusters of needles, melting at 142°C.; yield 0.29 gm. After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, it gave C, 66.6; H, 4.5;  $C_{44}H_{38}O_{14}$  requires C, 66.7; H, 4.8 per cent.

0.1 gm. of the substance on benzoilation in dry pyridine solution with 2 c.c. of benzoyl chloride also gave the benzoyl derivative obtained above in a yield of 0.26 gm.

*Acetylation of Kurrin (Anhydro-tetra-acetyl Kurrin)*—A mixture of 0.1 gm.

of substance, 1 gm. of fused sodium acetate and 2 c.c. of acetic anhydride was heated on a water bath for 2 hr. The reaction mixture was diluted with water and the acetyl derivative, obtained as a whitish powder, was filtered, washed with water and crystallized from alcohol. It melts at 120°C. (yield 0.12 gm.), is soluble in methanol, alcohol, benzene, ether and ethyl acetate, and insoluble in water, chloroform and petrol ether. After drying to constant weight at 61°C. *in vacuo* over phosphorus pentoxide, it gave C, 49.6; H, 6.2; M.W. (cryoscopic in benzol), 398.  $C_{17}H_{22}O_{11}$  (tetra-acetyl kurrin with intramolecular elimination of two molecules of water) requires C, 50.7; H, 5.5; M.W., 402.

0.1 gm. of the substance, on acetylation with 2 c.c. of acetyl chloride in dry pyridine solution, also gave an identical acetyl derivative (yield 0.11 gm.).

*Action of Phenyl Hydrazine on Kurrin*—An aqueous solution of 0.1 gm. of the substance was heated on the water bath for 2 hr. with 1 gm. of phenyl hydrazine hydrochloride and 1 gm. of sodium acetate. The reaction mixture was acidified with dilute acetic acid and extracted with ether. The ethereal layer was washed with water, dried over anhydrous sodium sulphate and freed of the solvent. The residue, on crystallization from ligroin, yielded clusters of yellowish-orange needles melting at 128°C.; yield 0.07 gm. After drying to constant weight at 61°C. *in vacuo* over phosphorus pentoxide, it gave C, 64.5; H, 6.5; N, 17.0 per cent.  $C_{27}H_{32}O_6N_6$  (triphenyl hydrazone of kurrin with intramolecular elimination of two molecules of water) requires C, 64.2; H, 6.3; N, 16.6 per cent.

#### *The Fatty Portion*—

*Kutkiol Acetate*—The petroleum ether extract of the powdered roots, after removal of the solvent under vacuo, yielded a greenish-brown oil (9.6 gm.) with a characteristic smell which, on keeping in benzol solution, gave a crystalline deposit of the crude kutkiol acetate. After a few crystallizations from hot ethyl acetate, it was finally obtained as clusters of needles melting at 74°C. (yield 0.06 per cent). Kutkiol acetate is soluble in petroleum ether, benzene, ether and ethyl acetate in the hot, and insoluble in methanol and alcohol. After drying to constant weight *in vacuo* over phosphorus pentoxide, it gave C, 81.4; H, 13.7; M.W. (Rast), 535.  $C_{42}H_{84}O_2$  requires C, 81.3; H, 13.5; M.W., 620.

On hydrolysis with alcoholic potassium hydroxide, it yielded an alcohol, kutkiol, melting at 118°C. and acetic acid. Kutkiol, on analysis after drying to constant weight *in vacuo* over phosphorus pentoxide, gave C, 82.6; H, 14.7; M.W. (Rast), 542.  $C_{40}H_{82}O$  requires C, 83.0; H, 14.1; M.W., 578.

*Kutki-sterol* — 10 gm. of the residue left on removal of the solvent from the mother liquors of kutkiol acetate was saponified with 15 per cent alcoholic potash by heating on the water bath for 3 hr. The alkaline solution was diluted with water and repeatedly extracted with ether and the ethereal layer was washed free of alkali, dried over anhydrous sodium sulphate and freed of the solvent. The orange-coloured residue (3.9 gm.), on repeated crystallizations from alcohol, gave colourless needles of the sterol melting at 124°C.

The sterol gave the characteristic Liebermann-Burachard colour reactions for a phytosterol. It is soluble in petroleum ether, ether, ethyl acetate and chloroform, sparingly soluble in alcohol, and insoluble in methanol and water. In 1 per cent chloroform solution, it gave  $[\alpha]_D^{25} = -39.0^\circ$ . It gave a digitonide melting at 245°C. (decomp.). After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, the sterol gave C, 83.6; H, 11.8; M.W. (Rast), 384.  $C_{24}H_{40}O$  requires C, 83.7; H, 11.6; M.W., 344.

The acetyl derivative prepared by heating the sterol with acetic anhydride and fused sodium acetate on the water bath melted at 110°C.

The percentage of sterol in the fat was determined by the digitonide method of Windaus. 25 c.c. of 1 per cent solution of digitonin in 95 per cent alcohol were added to the unsaponifiable matter obtained from 5 c.c. of the fat, dissolved in the same solvent. After standing overnight the precipitate was filtered on a weighed Gooch crucible, washed with alcohol, dried at 110°C. and weighed (0.88 gm.; equivalent to 0.18 per cent on the weight of the dry roots).

#### Acknowledgement

The authors take this opportunity to express their grateful thanks to the Conservator of Forests, Government of Jammu and Kashmir, Srinagar, for sending a supply of the material. They are thankful to Mr. R. C. Tewari and Drs. Weiler and Straus, Oxford, for some of the analytical data embodied in the present communication.

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## Chemical Composition of the Flowers of *Butea superba*

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THE flowers, seeds and root bark of *Butea frondosa* have been used in Indian medicine and have already been examined chemically. The closely related plant, *Butea superba*, has also been used in medicine; the flowers and bark have been claimed to be useful in scorpion stings and snake bites. The roots, which are considered in Siam to have rejuvenating

properties, were examined and the presence of two glucosides noted<sup>1</sup>. Preliminary physiological experiments indicated the presence of an oestrogenic substance and also a poisonous substance<sup>2</sup>. In the present note, the results of the examination of the flowers of *Butea superba* obtained from the forests of the Godavari Agency are presented. They were studied on the same lines as were

adopted for *Butea frondosa* flowers<sup>3,4</sup> From the alcoholic extract, on concentration, was obtained a heavy precipitate of a glycoside which was insoluble in ether, chloroform and acetone. When this was purified and crystallized from aqueous alcohol, it was colourless, melted at 193°-194°C. (decomp.), and agreed in all its properties with butrin. The identity was established by hydrolysis with dilute acid whereby a mixture of butin and butein were obtained. Confirmation was provided by methylation using dimethyl sulphate and anhydrous potassium carbonate in dry acetone medium and final hydrolysis of the methylated product yielding 2:4-dimethylbutein.

In certain experiments it was found possible to isolate mono-methyl butrin which was identical with a similar product obtained by the action of diazomethane on butrin<sup>5</sup>. This, on hydrolysis, yielded a mixture of 4'-methyl-butin and 4'-methyl butein. This type of partial methylation yielding a methyl ether of a flavanone without a free hydroxyl group in the 5-position is ordinarily unusual<sup>6</sup> but in the present case, probably owing to the sparing solubility of butrin and its mono-methyl-ether in acetone, its formation and isolation are possible. Earlier, Lal<sup>7</sup> reported the formation of mono-methyl butrin by the action of methyl iodide and potassium carbonate in absolute methyl alcoholic medium. A typical methylation experiment yielding this result is described here. Finely powdered butrin (1.0 gm.), anhydrous potassium carbonate (3.0 gm.), dimethyl sulphate (1 c.c.) and pure dry acetone (40 c.c.) were refluxed together for 30 hr. The acetone was removed and the residue was dissolved in 25 c.c. of water and set aside. A colourless, crystalline mass slowly separated out. This was filtered and crystallized from hot water. The substance melted at 222°C., did not give any colour with ferric chloride, but gave a bright-violet colour with magnesium powder and hydrochloric acid. It was found to be identical with mono-methyl butrin obtained by methylating butrin with diazomethane.

The yield of butrin from *B. superba* flowers is almost the same as from *B. frondosa* flowers. From the mother liquor left after the separation of butrin could be isolated butein and butin. Thus there is close resemblance in every respect between the two flowers. The *superba* flowers have a more marked orange-red colour but

this seems to be due to the presence of carotenoids.

With reference to the medicinal properties of these flowers, it may be appropriate to mention here the recent finding<sup>8</sup> that butein and similar chalkones have marked bacteriostatic properties which are also possessed by related flavanones. Consequently, we have now examined the toxic properties of butein and butin on fish. It is found that in a concentration of 100 mg. per litre (a dilution of 1 in 10,000), butein exhibits toxic effects (fish turning upside down) in the course of 2 hr. and 50 per cent of the fish employed do not recover but die even after their removal to fresh water. On the other hand, butin is considerably less toxic since, even at a concentration of 200 mg. per litre, it exhibits no toxic effect on fish during 24 hr. Thus, it would appear that hydroxy-flavanones, in virtue of their lower toxicity, may serve as better anti-bacterials. A number of related chalkones, flavanones and their methyl ethers were examined recently<sup>9</sup> with respect to their toxic properties and the results reported. With chalkones, the toxic symptoms set in more slowly but they are more persistent; the methoxy-chalkones are less toxic than the isomeric flavanones whereas the reverse is the case when a number of hydroxyl groups are present. As soon as all the hydroxyl groups of the chalkones are methylated, the toxicity increases considerably. The present study of butin and butein agree in general with these conclusions.

### Summary

The flowers of *Butea superba* yield the same crystalline components as those of *Butea frondosa*, the predominant product being butrin. Butin is far less toxic to fish than butein.

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# Some Polymerized Products from Castor Oil

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WHILE carrying out investigations on the dehydration of castor oil with various catalysts, it was found that selenium did not catalyse dehydration of the oil to any appreciable extent. An interesting phenomenon, however, was observed when the treated oil was allowed to cool. The oil, on cooling, was found to have thickened and become almost solid like wax.

Action of catalysts on polymerization of oils has been widely studied but these studies mainly refer to the production of drying oils. Sulphur and sulphur-containing compounds have been employed by Gardner<sup>1</sup> in the bodying of tung oil to prevent gelation. Harfer<sup>2</sup> used selenium for the same purpose. Parkin<sup>3</sup> has patented the use of dairy and dialkyl disulphides. The action of liquid sulphur monochloride on oils like castor oil and rape-seed oil has been studied by Kaufman, Baltés and Mardner<sup>4</sup>. Recently, drying oil polymers have been developed having the properties of natural rubber such as Norepol, Agripol<sup>5</sup>, Vulprene<sup>6</sup>, etc., from soya-bean or linseed oil.

The thickened product obtained by us is different from the products referred to above and does not appear to have been studied so far. No reference in literature could be traced in respect of the use of selenium as a polymerization catalyst.

A study of the polymerized products formed under varying experimental conditions

has been, therefore, made and the results are given in this communication.

## Experimental

The oil was thoroughly mixed with 1 per cent metal powder and heated at different temperatures for varying periods. The products obtained were examined for their softening points, acid, iodine, saponification and acetyl values, and solubility in ethyl alcohol. The results are shown in Table I.

Increase in the acid value indicates increased splitting and decomposition of the glycerides with increase in temperature. The iodine value is stationary, indicating that there is no change in the unsaturation of the glycerides. Fall in the acetyl value indicates some reduction in the hydroxyl groups. Solubility in alcohol at all stages shows that the hydroxyl groups are not appreciably affected.

The above experiments show that: (1) products of varying consistencies are obtained; (2) heating at 200°C. appears to produce maximum hardening; and (3) the products have rather high acidity, a feature which is undesirable for most of the uses to which these products are likely to be put.

Experiments were then carried out to obtain products of low acidity by heating the oil in an atmosphere of carbon dioxide for different intervals of time and testing the resultant products.

TABLE I—CATALYST 1 PER CENT

| No.* | TEMPERATURE OF REACTION °C. | TIME OF HEATING hr. | NATURE OF PRODUCT     |                               | ACID VALUE | IODINE VALUE (Wijs') | SAPONIFICATION VALUE | ACETYL VALUE | SOLUBILITY IN 95% ETHYL ALCOHOL (1:1) | SOFTENING POINT °C. |
|------|-----------------------------|---------------------|-----------------------|-------------------------------|------------|----------------------|----------------------|--------------|---------------------------------------|---------------------|
|      |                             |                     | on cooling            | after 24 hr.                  |            |                      |                      |              |                                       |                     |
| 1.   | 150-160                     | 1                   | Liquid, greenish      | Solid, greenish               | 16.4       | 85.7                 | 182.4                | 138.7        | Completely soluble                    | 45                  |
| 2.   | 150-160                     | 2                   | do                    | do                            | 17.6       | 84.8                 | 183.9                | 138.1        | do                                    | 54                  |
| 3.   | 150-160                     | 3                   | do                    | do                            | 19.2       | 84.9                 | 184.9                | 137.2        | do                                    | 52                  |
| 4.   | 200-210                     | 1                   | do                    | Solid, light green            | 21.2       | 84.4                 | 183.4                | 134.3        | do                                    | 55                  |
| 5.   | 200-210                     | 2                   | do                    | do                            | 22.1       | 84.3                 | 184.6                | 132.1        | do                                    | 66                  |
| 6.   | 200-210                     | 3                   | do                    | do                            | 22.9       | 84.2                 | 185.7                | 130.2        | do                                    | 64                  |
| 7.   | 250-260                     | 1                   | Thick liquid brownish | Solid, slightly reddish brown | 31.0       | 84.6                 | 185.2                | 129.5        | do                                    | 50                  |
| 8.   | 250-260                     | 2                   | do                    | do                            | 34.5       | 84.3                 | 186.6                | 126.0        | do                                    | 43                  |

\* In experiments 1, 2, and 3, the catalyst was collected at the bottom in powder form at the end of the reaction, while in experiments 4 to 8, it was in a molten state and formed a lump. On cooling, the mass was found to be porous and could be easily powdered.



TABLE II

Temperature 200°C.; carbon dioxide atmosphere;  
catalyst selenium (a) 1%; (b) 2%

| No. | INTERVALS AT WHICH SAMPLES WERE DRAWN, min. | CONSISTENCY                   | ACID VALUE | SOFTENING POINT (CAPILLARY TUBE METHOD) °C. | REMARKS                                                     |
|-----|---------------------------------------------|-------------------------------|------------|---------------------------------------------|-------------------------------------------------------------|
|     | Untreated oil                               | Liquid                        | 2.3        | ...                                         |                                                             |
| 1.  | (a) 30                                      | Solid after keeping overnight | 2.2        | 40.6                                        | } Suitable for use as petroleum jelly and vaseline          |
|     | (b) 30                                      | do                            | 2.0        | 43.3                                        |                                                             |
| 2.  | (a) 60                                      | do                            | 2.2        | 49.51                                       | } Suitable for use as wax substitute and lubricating grease |
|     | (b) 60                                      | do                            | 2.1        | 50                                          |                                                             |
| 3.  | (a) 90                                      | do                            | 2.4        | 54.5-57                                     |                                                             |
|     | (b) 90                                      | do                            | 2.2        | 55.6                                        |                                                             |
| 4.  | (a) 120                                     | do                            | 2.5        | 68.70                                       |                                                             |
|     | (b) 120                                     | do                            | 2.2        | 60                                          |                                                             |
| 5.  | (a) 150                                     | do                            | 2.7        | 60.62                                       |                                                             |
|     | (b) 150                                     | do                            | 2.4        | 57.2                                        |                                                             |
| 6.  | (a) 180                                     | do                            | 2.8        | 57.60                                       |                                                             |
|     | (b) 180                                     | do                            | 2.5        | 53.9                                        |                                                             |

The results of these experiments are given in Table II.

From the above results, the following points emerge:

1. In an atmosphere of carbon dioxide, the acidity of the final product is low.

2. In conformity with results of the previous findings, after a certain period of heating, the melting point of the product begins to fall.

3. The maximum hardening is reached after about 2 hr.; the product obtained has a somewhat higher softening point; and

4. there is no advantage in using higher percentages of the catalyst.

Larger samples of hardened castor oil of the consistency of petroleum jelly or vaseline were prepared using 1 per cent selenium catalyst in a carbon dioxide atmosphere. The product was faintly greenish yellow and had an acid value of 2.9.

Samples of the product were sent to the Ordnance Laboratories, Kanpur, for being tested whether it conforms to the Indian Stores Department specification for petroleum jelly, and to the Principal, Medical College, Agra, for being tested as a substitute for vaseline. The report from the Ordnance Laboratories was that the product conformed in all respects to the Indian Stores Department Specification G/O-101/86 for mineral jelly except for the lower softening point and higher organic acidity.

The report of the Principal, Medical College, Agra, shows that the first sample sent to him was found to cause irritation when applied to the eyes and had an acrid

smell. The product was refined to remove the acidity by caustic soda treatment and subsequent washing and drying. The moisture-free neutral material was then treated with 3 per cent bleaching earth and 1 per cent of "Decol" for half an hour at 80°C., and filtered hot. The filtered oil was then treated for an hour with superheated steam (at 250°C.) under vacuum and at a temperature of 100° to 125°C. The improvement resulting from the treatment is shown in Table III.

The refined sample was found satisfactory as a base for medicinal ointments. It did not promote bacterial growth, caused no irritation and possessed no objectionable odour.

As selenium is rather costly, it was considered advisable to study whether the catalyst can be used continuously with a view to lengthen its useful life and obtain a maximum yield of the polymerized product. To ascertain whether the reaction could be carried out continuously, fresh quantities of the raw oil were run into the flasks containing the catalyst mass left over from previous trials. Samples were drawn out, as before, at intervals of 1 hr. The results obtained are given in Table IV.

The results show that the action of the catalyst used for a second time was not quite satisfactory. It was mainly due to the lack of thorough intermixing of the catalyst and the oil. Accordingly, the residual catalyst was thoroughly mixed with the oil by grinding before the heating operation.

TABLE III

|            | PRODUCT BEFORE TREATMENT | PRODUCT AFTER TREATMENT |
|------------|--------------------------|-------------------------|
| Colour     | Faint green              | No change               |
| Acid value | 2.9                      | 0.8                     |
| Smell      | Acrid                    | Odourless               |

TABLE IV

Temperature, 200°C.; castor oil, 500 gm.; catalyst, 5 gm. selenium;  
carbon dioxide atmosphere

| No. | INTERVAL AT WHICH SAMPLES WERE DRAWN | CATALYST LEFT OVER FROM 1ST SERIES OF EXPERIMENTS USED | CATALYST LEFT OVER FROM 2ND SERIES OF EXPERIMENTS USED |
|-----|--------------------------------------|--------------------------------------------------------|--------------------------------------------------------|
| 1.  | 1 hr.                                | Product: liquid after 24 hr. cooling                   | Product: liquid after 24 hr. cooling                   |
| 2.  | 2                                    | do                                                     | Product: viscous liquid after 24 hr. cooling           |
| 3.  | 3                                    | Product: viscous liquid after 24 hr. cooling           | Product: very thin paste after 24 hr. cooling          |
| 4.  | 4                                    | do                                                     | Product: thin paste after 24 hr. cooling               |

TABLE V

Temperature 200°C.; castor oil, 500 gm.; catalyst, 5 gm. selenium; carbon dioxide atmosphere

| No. | INTERVALS AFTER WHICH SAMPLES WERE DRAWN min. | SOFTENING POINTS OF THE PRODUCT IN °C. WHEN CATALYST USED FOR: |          |          |          |          |          | REMARKS                       |
|-----|-----------------------------------------------|----------------------------------------------------------------|----------|----------|----------|----------|----------|-------------------------------|
|     |                                               | 1st time                                                       | 2nd time | 3rd time | 4th time | 5th time | 6th time |                               |
| 1.  | 30                                            | 39                                                             | 36       | ...      | ...      | ...      | ...      | The products set after 24 hr. |
| 2.  | 45                                            | ...                                                            | ...      | 38       | ...      | ...      | ...      |                               |
| 3.  | 60                                            | 48                                                             | 42       | ...      | 39       | 35       | 28       |                               |
| 4.  | 90                                            | 53                                                             | 48       | 46       | 46       | 42       | 29       |                               |
| 5.  | 120                                           | 67                                                             | 52       | ...      | 49       | 45       | 31       |                               |
| 6.  | 135                                           | ...                                                            | ...      | 53       | ...      | ...      | ...      |                               |
| 7.  | 150                                           | 63                                                             | 58       | 56       | 55       | 47       | 33       |                               |
| 8.  | 180                                           | 58                                                             | 59       | ...      | 54       | 49       | 35       |                               |

TABLE VI

| No. | INTERVALS AFTER WHICH SAMPLES WERE DRAWN min. | SOFTENING POINT OF THE PRODUCT IN °C. WHEN CATALYST USED FOR: |          |          |          |          |          |                       |                               | REMARKS |
|-----|-----------------------------------------------|---------------------------------------------------------------|----------|----------|----------|----------|----------|-----------------------|-------------------------------|---------|
|     |                                               | REACTION TEMP. 175°C.                                         |          |          |          |          |          | REACTION TEMP. 250°C. |                               |         |
|     |                                               | 1st time                                                      | 2nd time | 3rd time | 4th time | 5th time | 1st time | 2nd time              |                               |         |
| 1.  | 30                                            | 36                                                            | 32       | ...      | ...      | ...      | 56       | 50                    | The products set after 24 hr. |         |
| 2.  | 45                                            | ...                                                           | ...      | 29       | ...      | ...      | 56       | 48                    |                               |         |
| 3.  | 60                                            | 44                                                            | 35       | ...      | 29       | 23       | 48       | 43                    |                               |         |
| 4.  | 90                                            | 52                                                            | 41       | 37       | 33       | 28       | 42       | 38                    |                               |         |
| 5.  | 120                                           | 59                                                            | 46       | ...      | 35       | 30       | 36       | 34                    |                               |         |
| 6.  | 135                                           | ...                                                           | ...      | 45       | ...      | ...      | 29       | ...                   |                               |         |
| 7.  | 150                                           | 58                                                            | 50       | 48       | 39       | 33       | ...      | ...                   |                               |         |
| 8.  | 180                                           | 56                                                            | 52       | ...      | 44       | 37       | ...      | ...                   |                               |         |

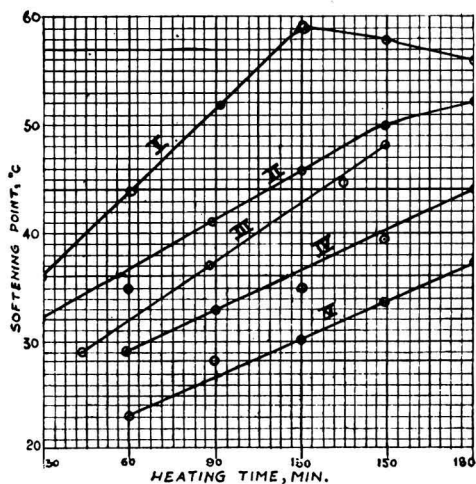


FIG. 1 — RELATION BETWEEN DURATION OF HEATING AT 175°C. AND SOFTENING POINT OF POLYMERIZED PRODUCTS.

The results obtained are given in Table V. The effect of the used catalyst at two different temperatures (175°C. and 250°C.)

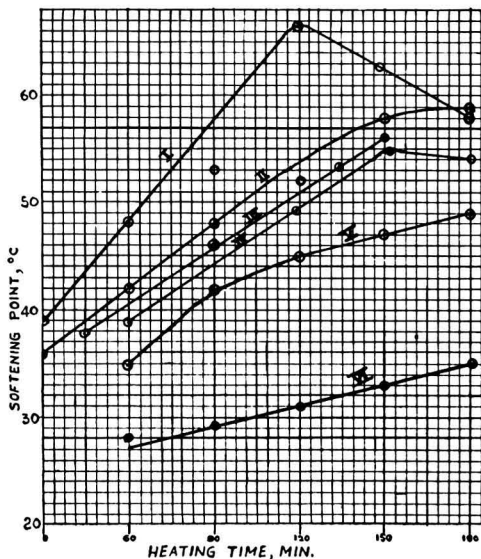


FIG. 2 — RELATION BETWEEN DURATION OF HEATING AT 200°C. AND SOFTENING POINT OF POLYMERIZED PRODUCTS.

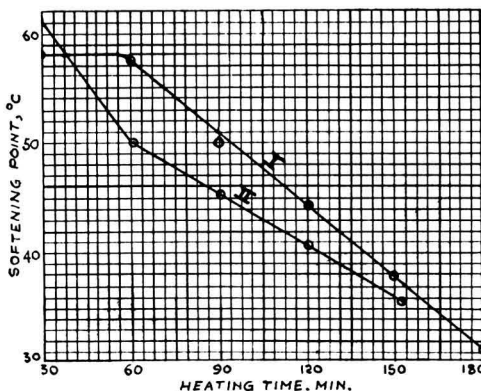


FIG. 3 — RELATION BETWEEN DURATION OF HEATING AT 250°C. AND SOFTENING POINT OF POLYMERIZED PRODUCTS.

was studied, all other conditions being kept constant. The results obtained are given in Table VI.

The results contained in Tables V and VI have been shown graphically in Figs. 1 to 2 and 3.

Summary

Polymerized products of varying consistencies and melting points have been prepared from castor oil using selenium as

catalyst, and their properties studied. The optimum conditions for obtaining the products are: (1) heating at a temperature of 200°C.; and (2) use of 1 per cent selenium on the weight of oil as catalyst.

The catalyst can be collected and reused 4-6 times for treating fresh lots of castor oil.

The products can be refined to produce neutral, colourless and odourless products, if necessary. The various polymerized products can be used as substitutes for petroleum jelly for the protection of steel struc-

tures, for vaseline in pharmacy, as a wax in leather dressing and sizing, and as a lubricating grease.

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## Sebacic Acid & Secondary Octyl Alcohol from Castor Oil

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SEBACIC acid and other long-chain dicarboxylic acids have found many useful applications in the manufacture of plastics, plasticizers, fungicidal sprays, and as a starting material for the synthesis of muscone. No detailed description for the preparation of sebacic acid is available in literature<sup>1-3</sup>. Gilman<sup>4</sup> describes a method for the preparation of octyl alcohol from castor oil soap, but does not describe the isolation of sebacic acid which remains in the residue.

It was considered desirable to work out the conditions for obtaining optimum yields of sebacic acid and secondary octyl alcohol from castor oil.

Previous investigators have generally prepared sebacic acid by the pyrolysis of castor oil soap in the presence of caustic soda. The effect of the addition of various alkaline materials to the castor oil soap on the yield of sebacic acid and secondary octyl alcohol has been studied and the results obtained are given in Table I.

Castor oil soap was heated in the presence of 30 per cent caustic soda under steam pressure (c. 700 lb./sq. in.) at 260°C. It was found that heating the soap for 8 hr.

| BASE USED        | TABLE I       |                      |                                    |
|------------------|---------------|----------------------|------------------------------------|
|                  | QUANTITY<br>% | SEBACIC<br>ACID<br>% | SECONDARY<br>OCTYL<br>ALCOHOL<br>% |
| Blank            | ...           | 3.2                  | 5.5                                |
| Caustic soda     | 10            | 17.5                 | 30.0                               |
| "                | 20            | 20.5                 | 30.0                               |
| "                | 30            | 22.4                 | 29.0                               |
| Sodium carbonate | 10            | 11.0                 | 33.0                               |
| "                | 20            | 14.0                 | 35.0                               |
| "                | 30            | 15.8                 | 37.2                               |
| "                | 40            | 15.0                 | 36.0                               |
| Lime             | 10            | 5.8                  | 25.2                               |
| "                | 20            | 5.5                  | 24.5                               |
| "                | 30            | 4.8                  | 22.1                               |
| Barium oxide     | 10            | 6.0                  | 18.0                               |
| "                | 20            | 6.5                  | 18.5                               |
| "                | 30            | 7.0                  | 17.6                               |
| Zinc oxide       | 10            | 3.5                  | 17.0                               |
| "                | 20            | 4.0                  | 17.4                               |
| "                | 30            | 4.5                  | 18.0                               |
| "                | 40            | 3.5                  | 14.2                               |
| Sodium silicate  | 10            | 3.5                  | 7.5                                |
| "                | 20            | 3.2                  | 8.0                                |
| "                | 30            | 3.0                  | 8.5                                |
| "                | 40            | 2.7                  | 6.2                                |

gives the maximum yield of sebacic acid, i.e. 53 per cent.

After the removal of sebacic acid from the resultant solid mass, a residue of brown, thick, liquid acids remained (c. 20 per cent). The sodium soap of these acids gave a thick lather with water indicating the possibility of their use as a component of scouring agents for laundering purposes.

### Experimental

Castor oil (5 kg.) was saponified by slight warming with 1 kg. of caustic soda dissolved in 2 litres of water. The resulting pale-white soap contained 20 per cent moisture and about 1 per cent excess alkali. 200 gm. lots of this soap were mixed thoroughly with weighed amounts of different basic materials, transferred to round-bottom flasks with separating funnels and distilling heads, and heated in an air bath. The temperature was raised gradually. Much hydrogen gas was evolved in the beginning and care was taken not to lose the octyl alcohol which distilled over along with some water. The water was separated from the alcohol and poured back into the flask through the separating funnel at the rate of 2 or 3 drops a minute. The temperature was gradually raised to 200°C. till no more octyl alcohol distilled over. This process takes 50-70 hr. continuous heating. The crude octyl alcohol thus collected was fractionally distilled and the fraction between 175°-180°C. was pure secondary octyl alcohol. The solid product which remained in the flask was suspended in water, acidified with dilute hydrochloric acid, boiled and filtered hot. The filtrate, on cooling, deposited crystals of sebacic acid (m.p. 133°-134°C.). They were filtered and dried at 40°-50°C. The percentage of octyl alcohol and sebacic acid obtained are given in Table I.

#### TABLE II

| PERIOD OF HEATING<br>hr. | YIELD OF<br>SEBACIC ACID<br>% |
|--------------------------|-------------------------------|
| 4                        | 25.5                          |
| 8                        | 53.0                          |
| 12                       | 53.8                          |

The results show that a maximum yield of sebacic acid is obtained when castor oil soap is heated in the presence of 20-30 per cent of caustic soda. The yield of secondary octyl alcohol, however, is maximum when castor oil soap is heated with 30 per cent sodium carbonate. Besides a higher yield, the alcohol is purer.

In order to find out the effect of heating under pressure, 200 gm. of the soap were mixed with 60 gm. of caustic soda and the mixture heated in an autoclave at 260°C. at a steam pressure of 700 lb./sq. in. for different lengths of time. The yields of sebacic acid are given in Table II.

Heating for 8 hr. under pressure will give a higher yield of sebacic acid than pyrolysis under ordinary pressure.

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## Extraction of Potash from Felspar

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**F**ELSPAR has been located in Hyderabad State in abundant quantity in Raichur, Mahboobnagar, Gulberga and Nalgonda districts<sup>1</sup>. Potash, a valuable fertilizer, is locked up in felspar. No commercial process is as yet available for its recovery from this mineral. The dry processes for the extraction of potash from felspar require high temperature and special equipment. The wet processes

consisting of digestion of felspar with a calcium compound under pressure are comparatively simpler. Besides, in the latter case, the residual material after the recovery of potash is a valuable by-product which can be used in the preparation of cement and bricks.

Ross<sup>2</sup> obtained a 90 per cent yield of potash when a mixture of felspar (5 gm.) and lime (8 gm.) is digested in a nickel-

steel bomb of 250 c.c. capacity, under a pressure of 10-15 atm. The purpose of this investigation is to study the above process using commercial lime and larger quantities of the materials and also to study the effect of using basic calcium nitrate in the place of lime.

### Experimental

• The Golconda felspar contains 11.3 per cent  $K_2O$  and 5.0 per cent  $Na_2O$ , while Mahboobnagar variety consists of 9.86 per cent  $K_2O$  and 4.67 per cent  $Na_2O$ . The Golconda felspar which is richer in potash content was used in all the experiments.

The felspar was heated in an oven to about  $700^{\circ}$ - $800^{\circ}C.$ , quenched in cold water, crushed to pass through 120 mesh and analysed. The results of the analysis was as follows :

|                 |     |     | %     |
|-----------------|-----|-----|-------|
| Silica          | ... | ... | 64.58 |
| Alumina         | ... | ... | 17.71 |
| Iron oxide      | ... | ... | 0.50  |
| Calcium oxide   | ... | ... | 0.80  |
| Magnesium oxide | ... | ... | —     |
| $K_2O$          | ... | ... | 11.30 |
| $Na_2O$         | ... | ... | 5.00  |

Felspar (120 mesh powder), commercial quicklime (120 mesh powder) and enough water were added into an autoclave and thoroughly mixed. The steel autoclave

(4 litres capacity) was enclosed in an oil bath, and provided with a safety valve, a pressure gauge and a thermometer and heated with a stove. The digestions were carried out at 100, 150, 200 and 250 lb. per sq. in. pressure. After digestion, the resulting slime was filtered through a Buchner funnel, the residue was washed 4 times with hot water and the potash in the filtrate estimated by the cobalt-nitrite method<sup>3</sup>. The results of experiments with different samples of commercial lime, containing 54.6, 70 and 90 per cent of  $CaO$ , at different pressures and intervals of time have been given in Table I.

It will be seen from the results that the yield of potash increases with increase in pressure, 90.8 per cent of the potash contained in felspar being recovered when a mixture of 225 gm. of lime (containing 200 gm. of calcium oxide) and 100 gm. of felspar powder is digested at 250 lb. per sq. in. pressure for 6 hr. (expt. 10). Increase in the quantity of the felspar in the digestion mixture results in a lower yield under similar conditions (cf. expts. 6, 7 and 8). The results of experiments 10, 11 and 12 show that the yield of potash is proportional to the calcium oxide present in the mixture. Calcium carbonate present in commercial samples of lime does not act on felspar. The yield is enhanced when calcium carbonate

TABLE I

| EXPT. No. | WT. OF FELSPAR gm. | WT. OF QUICKLIME gm. | PURITY OF LIME % | CALCIUM PRESENT AS $CaO$ gm. | REACTION PRESSURE lb./sq. in. | TIME hr. | YIELD OF POTASH % |            |
|-----------|--------------------|----------------------|------------------|------------------------------|-------------------------------|----------|-------------------|------------|
|           |                    |                      |                  |                              |                               |          | Observed          | Calculated |
| 1         | 100                | 200                  | 54.6             | 78                           | 100                           | 4        | 10.4              |            |
| 2         | 100                | 200                  | 54.6             | 78                           | 100                           | 6        | 23.4              |            |
| 3         | 50                 | 143                  | 70               | 71                           | 150                           | 4        | 61.0              |            |
| 4         | 50                 | 143                  | 70               | 71                           | 150                           | 6        | 66.0              |            |
| 5         | 50                 | 143                  | 70               | 71                           | 200                           | 6        | 78.0              |            |
| 6         | 50                 | 143                  | 70               | 71                           | 250                           | 6        | 88.0              |            |
| 7         | 100                | 286                  | 70               | 142                          | 250                           | 6        | 71.8              |            |
| 8         | 200                | 570                  | 70               | 284                          | 250                           | 6        | 63.3              |            |
| 9         | 200                | 570                  | 70               | 284                          | 250                           | 10       | 70.0              |            |
| 10        | 100                | 225                  | 90               | 144                          | 250                           | 6        | 90.8              |            |
| 11        | 100                | 112                  | 90               | 72                           | 250                           | 6        | 46.3              |            |
| 12        | 100                | 56                   | 90               | 36                           | 250                           | 6        | 23.3              |            |

TABLE II

| EXPT. No. | WT. OF FELSPAR gm. | WT. OF QUICKLIME gm. | PURITY OF LIME % | NITRIC ACID (66%) C.C. | TOTAL CALCIUM IN THE MIXTURE gm. | REACTION PRESSURE lb./sq. in. | TIME hr. | YIELD OF POTASH |            |
|-----------|--------------------|----------------------|------------------|------------------------|----------------------------------|-------------------------------|----------|-----------------|------------|
|           |                    |                      |                  |                        |                                  |                               |          | Observed        | Calculated |
| 13        | 100                | 185                  | 54.6             | 120                    | 104                              | 150                           | 4        | 45.9            | ...        |
| 14        | 100                | 185                  | 54.6             | 120                    | 104                              | 150                           | 6        | 55.5            | ...        |
| 15        | 100                | 185                  | 54.6             | 120                    | 104                              | 150                           | 8        | 62.8            | ...        |
| 16        | 100                | 92                   | 54.6             | 60                     | 52                               | 150                           | 4        | 21.35           | 22.9 (13)  |
| 17        | 100                | 92                   | 54.6             | 60                     | 52                               | 150                           | 6        | 28.7            | 27.8 (14)  |
| 18        | 100                | 92                   | 54.6             | 60                     | 52                               | 150                           | 8        | 36.4            | 31.4 (15)  |
| 19        | 50                 | 70                   | 70               | 60                     | 46                               | 150                           | 4        | 36.0            | 39.52 (3)  |
| 20        | 50                 | 70                   | 70               | 60                     | 46                               | 200                           | 6        | 53.6            | 50.54 (5)  |
| 21        | 50                 | 70                   | 70               | 60                     | 46                               | 250                           | 6        | 58.3            | 57.02 (6)  |
| 22        | 100                | 107                  | 90               | 120                    | 73                               | 250                           | 6        | 49.3            | 46.01 (10) |

is converted into a reactive calcium compound such as calcium nitrate.

Enough nitric acid was added to the mixture of felspar, lime and water so as to form a mixture ultimately consisting of felspar, calcium nitrate and calcium oxide. The quantities of felspar, lime, nitric acid employed, the conditions of digestion and yield of potash are given in Table II.

When felspar is digested with a mixture of calcium oxide and calcium nitrate, the yield of potash is proportional to the total calcium content. The observed results show good agreement with the calculated values. Further work is in progress to find out the part played by calcium oxide and calcium nitrate in the reaction.

#### Utilization of Sludge

Bricks made from the sludge remaining over after the extraction of potash, under a pressure of 2 tons per sq. in., and steamed for 8 hr. at a pressure of 100 lb./sq. in. possess a crushing strength of 2,200 lb. per sq. in.

Superior quality sand-lime bricks can be made from the sludge when mixed with suitable amount of sand and water. Bricks were prepared with 55 gm. of sludge, 365 gm. of sand and 25 c.c. of water and steamed as described above. The crushing strengths of the bricks compare well with those of first-grade sand-lime bricks (TABLE III).

#### Summary

1. The yield of potash is 90.8 per cent when felspar and lime (90 per cent CaO)

TABLE III

| PURITY OF LIME USED FOR DIGESTION % | CRUSHING STRENGTH lb./sq. in. | CRUSHING STRENGTH ; A.S.T.M. STANDARDS FOR SUPERIOR GRADE (SW) S.L. BRICK lb./sq. in. |
|-------------------------------------|-------------------------------|---------------------------------------------------------------------------------------|
| 90                                  | 5130 }                        | 3500-4500                                                                             |
| 70                                  | 3829 }                        |                                                                                       |

mixture containing felspar : calcium oxide as 1 : 2 by weight is digested at 250 lb./sq. in. pressure.

2. The yield of potash is proportional to calcium content of lime. Calcium carbonate present in the commercial samples of lime is inert. The yield is improved by converting calcium carbonate to calcium nitrate by the addition of nitric acid. The yield is then proportional to the total calcium content.

3. The sludge obtained after recovery of potash can be used in the preparation of sand-lime bricks.

#### Acknowledgement

I wish to express my sincere thanks to Dr. M. Qureshi for suggesting the problem and for his guidance. My thanks are also due to Dr. D. S. Datar for constant help and supervision.

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## Letters to the Editor

### ANALYTICAL USES OF BENZOHYDROXAMIC ACID

THE ANALYTICAL APPLICATIONS OF BENZOHYDROXAMIC ACID in qualitative and quantitative inorganic analysis are described in this note. The reagent gives deep-coloured, stable, water-soluble complexes with a number of cations. It promises to be of

great use in the detection of traces of metals like uranium, vanadium and molybdenum.

The potassium salt of the reagent is easily obtained by reacting a methyl alcohol solution of ethyl benzoate with hydroxylamine in the presence of potassium hydroxide. The reagent is neutralized with dilute hydrochloric acid before use. Best results are obtained in nearly neutral media as the

development and intensity of colour are adversely affected by low or high  $pH$ .

The sensitivity and the limit of concentration for uranium and vanadium with this reagent are superior to the limits obtained with the other standard "spot" test reagents in use (potassium ferrocyanide and hydrogen peroxide respectively). The results obtained with molybdenum also compare favourably with those obtained with phenylhydrazine reagent.

Tests carried out on a "spot" plate with 3 per cent neutral aqueous solution of the reagent gave the following results :

TABLE I

| CATION     | REAGENT                             | SENSITIVITY | LIMIT OF CONCENTRATION |
|------------|-------------------------------------|-------------|------------------------|
| Uranium    | Benzohydroxamic acid                | 0.5 $\mu$   | 1 : 200,000            |
|            | Pot ferrocyanide                    | 0.9 $\mu$   | 1 : 50,000             |
| Vanadium   | Benzohydroxamic acid                | 0.2 $\mu$   | 1 : 250,000            |
|            | Hydrogen peroxide                   | 2.5 $\mu$   | 1 : 20,000             |
|            | Ferric chloride<br>Dimethylglyoxime | 1.0 $\mu$   | 1 : 50,000             |
| Molybdenum | Benzohydroxamic acid                | 1.0 $\mu$   | 1 : 200,000            |
|            | Phenylhydrazine reagent             | 0.1 $\mu$   | 1 : 300,000            |

**Uranium**—A deep-orange colour is obtained in weak acid medium, while in weak ammoniacal solution, an orange precipitate soluble in excess of ammonia is produced.

**Vanadium**—A deep-purple colour is given by pentavalent vanadium in neutral or slightly alkaline solutions.

**Molybdenum**—A golden-yellow colour is produced in weakly ammoniacal medium.

The test for vanadium is of especial importance as it promises to be a convenient method for colorimetric detection and estimation of vanadium in magnetite ores, specially because the deep colour of the iron complex can be easily masked with hydrofluoric acid.

The conditions for the quantitative determination of the cations are being investigated.

The work was undertaken at the suggestion of Dr. J. Gupta, Asst. Director, National Chemical Laboratory, to whom our thanks are due.

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July 11, 1949

### A NOTE ON THE CHEMICAL EXAMINATION OF NIM (*MELIA AZADIRACHTA*) EXUDATE

THE CHEMICAL EXAMINATION OF NIM EXUDATION was undertaken as a part of the scheme of systematic investigation on nim (*Melia azadirachta*; *Melia indica*)<sup>1-6</sup>. The exudation is a rare, seasonal occurrence in comparatively older trees, and is reputed in the indigenous systems of medicine as an alterative tonic. The sap was collected locally in the months of December-January. It had a strong smell of fermented liquor with the characteristic odour of nim, and was opalescent in character. It was slightly sweetish in taste and acidic to litmus.

The freshly collected exudate had a specific gravity, 1.0525 at 19°C. and a  $pH$ , 5.0. It contained total solids, 12.5 per cent; reducing sugar (calculated as glucose), 0.5 per cent; total acids (calculated as acetic acid), 0.8 per cent. The sap was dextro-rotatory but exact determination of the optical rotation was not possible because of the opalescent character of the liquid which could not be got over by repeated filtration. Analysis of the ash from the total solids, precipitated by the addition of alcohol, showed the presence of phosphate, sulphate, iron and calcium. An essential oil, giving a qualitative test for sulphur, was isolated from the steam distillate of the exudation, in a comparatively poor yield. Acetic acid was identified in the sap distillate.

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August 22, 1949

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### A NOTE ON THE CHEMICAL EXAMINATION OF NIM (*MELIA AZADIRACHTA*) TRUNK-BARK

CHEMICAL EXAMINATION OF THE NIM TRUNK-BARK, reputed as a febrifuge in the indigenous systems of medicine, has been carried out in continuation of the systematic investigation on the various parts of the nim tree (*Melia azadirachta*; *Melia indica*). The crystalline bitter constituents, nimbin and nimbinin, and the amorphous bitter, nimbidin, isolated earlier from the nim oil<sup>1</sup> and the root-bark<sup>2</sup>, have also been obtained from the alcoholic extract of the trunk-bark in an yield of 0.04, 0.001 and 0.4 per cent respectively. The oil-soluble bitter, nimbidol (loc. cit.), was not, however, found to be present in the trunk-bark. An essential oil having similar characteristics to that isolated from the nim blossoms<sup>3</sup> has also been obtained from the petrol-ether soluble fraction of the alcoholic extract in an yield of 0.02 per cent. The unsaponifiable fraction of the fatty portion of the extract yielded nimbosterol (loc. cit.) in an yield of 0.03 per cent. The tannin content of the bark has been found

to be *ca.* 6 per cent on the weight of the dry bark.

The isolation of an amorphous glucoside from the nim trunk-bark has been reported earlier by Sen<sup>4</sup>, but following the procedure adopted by him for the isolation of the substance, it has only been possible to obtain a product consisting of tannin material contaminated with the bitter principles of the nimbidin series.

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*Supplement*

AROMATIC PLANTS OF INDIA



FIG. 35 — *Psoralea Corylifolia* Linn.

( To face page 159 of Part IX, Vol. VIII, No. 2, February 1949 )

# AROMATIC PLANTS OF INDIA

## PART X

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### Family XLI—ROSACEAE

(Rose Family)

THIS is a large family comprising about 100 genera and over 2,000 species of herbs, shrubs, and trees which are spread over the whole world, but most abundant in north temperate regions.

Many plants of this family are cultivated for ornamental purposes; roses, spiraeas, flowering cherries, and plums are some of the prominent examples. A few only, such as *Prunus puddum* Roxb. ex Wall., yield timber of some value. There are, however, a large number of plants which furnish valuable fruits, such as the apricot (*Prunus armeniaca* Linn.), peach [*P. persica* (Linn.) Stokes], almond (*P. amygdalus* Batsch), sweet cherry or gean (*P. avium* Linn.), plums and prunes (*P. domestica* Linn.), alucha (*P. communis* Huds., var. *insititia* Hook. f.), cherries (*P. cerasus* Linn. and others), loquat [*Eriobotrya japonica* (Thunb.) Lindl.], strawberry (*Fragaria vesca* Linn.), raspberry and blackberry (*Rubus* sp.), apple (*Pyrus malus* Linn.), pears (*P. communis* Linn. and *P. sinensis* Lindl.), quince (*Cydonia oblonga* Mill., syn. *Pyrus cydonia* Linn. and *Cydonia vulgaris* Pers.), etc.

Some plants are used for medicinal purposes, and the root, bark, leaves, and flowers of these are said to possess astringent, tonic, and anthelmintic properties. The mucilaginous seeds of *Cydonia oblonga* have been used in indigenous medicine for a long time as a demulcent in the treatment of gastro-intestinal and other conditions.

Many of the plants of this family possess poisonous properties and these are due to the cyanogenetic glycosides, such as amygdalin, prunasin, etc., which occur in the leaves, bark, and seeds. Of these, amygdalin occurring in bitter almonds (*Prunus amygdalus* Batsch, var. *amara* of authors) is well known.

Apart from cyanogenetic glycosides, other glycosides, such as phloridzin, gaultherin, etc., saponins, such as those in the bark of *Quillaja saponaria* Molina (soapbark tree) of Chile, etc., and essential oils are found in plants of this family.

Almost all the fruits mentioned above and many others (such as those of species belonging to *Fragaria*) have a flavour and aroma peculiar to each, and this is due to the presence of some essential oil or the other, possibly as esters or aldehydes. None of these, however, is extracted because of the very small quantity present in expensive fruits. For this reason, primarily, the fruit essences are prepared from synthetic products, simulating the natural flavours.

Oil of roses, which has perhaps been the most persistently popular perfume from antiquity, is derived from flowers of certain species of *Rosa* which are cultivated for the purpose. Many other plants belonging to *Crataegus*, *Geum*, *Prunus*, *Pyrus*, *Rubus*, and *Spiraea* are also known to contain essential oils, and will be described under the accounts of these genera.

The flowers of *Filipendula ulmaria* Maxim. (*Spiraea ulmaria* Linn.) of Europe, N. America, and N. Asia yield 0.2 per cent. of an essential oil, which is heavier than water. The constituents of the oil are salicylaldehyde, methyl salicylate, and traces of heliotropin and vanillin (Wehmer). The salicylaldehyde is not found in the flowers as such, but is formed during distillation. The roots of this species also yield an oil the main constituent of which is methyl salicylate, which is formed from the glycoside gaultherin by the action of the enzyme gaultherase.

The fruit of a number of species of *Pygeum*, e.g., *P. gardneri* Hook. f., a middle-sized tree of W. India and the Nilgiris, and *P. acuminatum* Coleb., a moderate-sized to

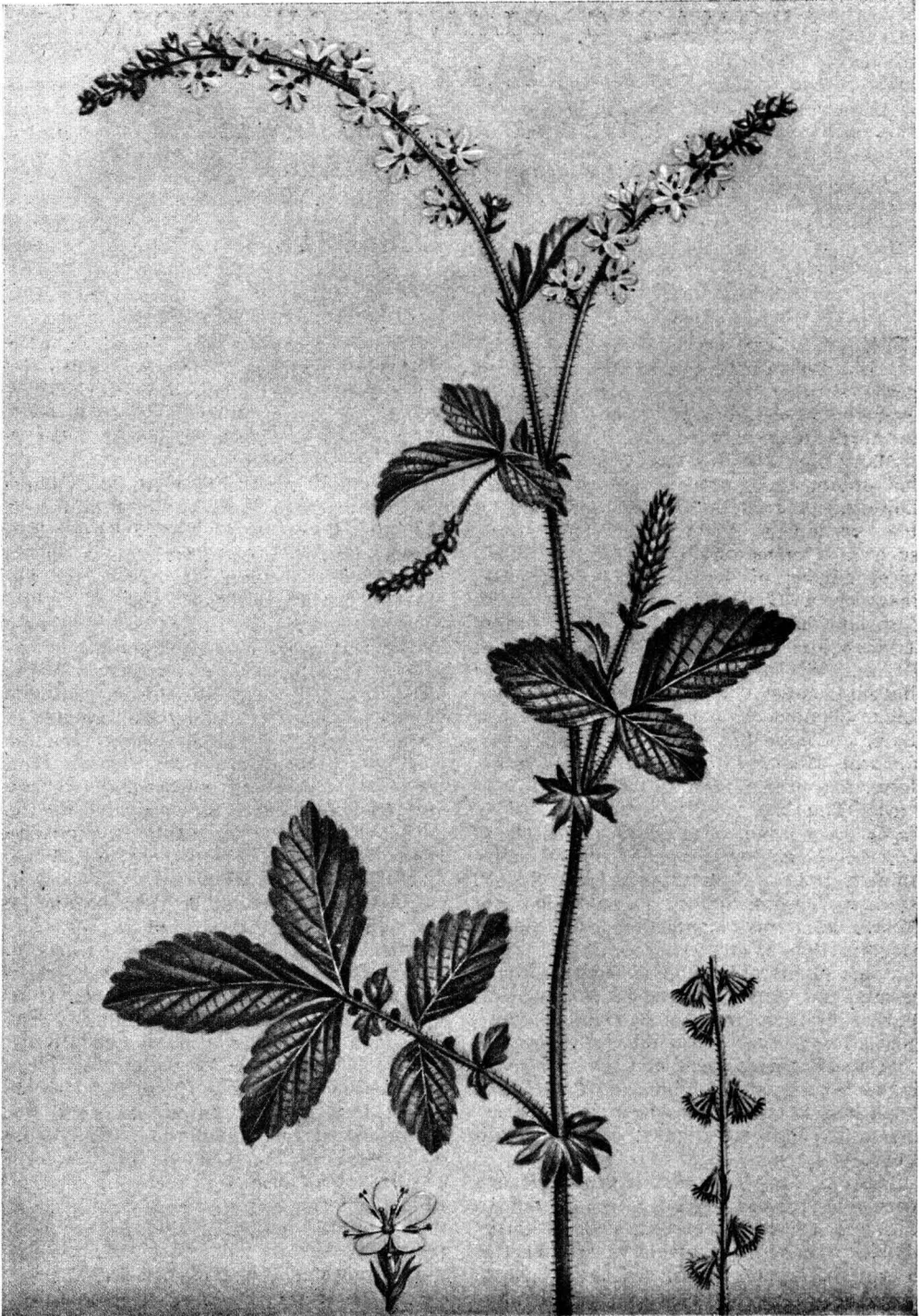


FIG. 36 — *Agrimonia eupatoria* Linn.

large tree of E. Bengal and Khasi Hills, if crushed, emits a strong smell of bitter almonds. Similarly, the blaze (wood) of some species, such as *P. glaberrimum* Hook. f., a large tree of the E. Himalayas and Assam, *P. montanum* Hook. f., a small to medium-sized tree of the E. Himalayas and Assam, and *P. acuminatum*, also smells of bitter almonds. Even the leaves of some species smell of bitter almonds. Although none of the Indian species have so far been investigated, hydrocyanic acid has been reported from the bark of *P. parviflorum* Teijsm. & Binn. of Malaysia. The greenish-white or pale-yellow flowers of *P. montanum*, which are  $\frac{1}{2}$  in. across, are slightly scented.

*Eriobotrya bengalensis* Hook. f., a middle-sized to large tree of the E. Himalayas and Assam, bears white fragrant flowers which are  $\frac{1}{2}$  in. across.

*Potentilla fruticosa* Linn. (*pinjung*), a shrub in the temperate and subalpine Himalayas from Kashmir to Sikkim at altitudes of 8,000 to 12,000 ft., bears numerous, bright-yellow flowers 1 to  $1\frac{1}{2}$  in. in diameter. Its pinnately compound fragrant leaves are said to be used as a substitute for tea by the local people, but no essential oil appears to have been distilled and examined. From the roots of some species of *Potentilla*, such as the W. Himalayan *P. anserina* Linn. and *P. reptans* Linn., a small quantity of an alcohol, tormentol, has been isolated.

*Agrimonia eupatoria* Linn., a herb of the Himalayas, Khasi and Mishmi Hills, is reported to contain an essential oil (Wehmer), the nature of which has not so far been investigated.

From among the constituents of essential oils reported from plants of this family may be mentioned: amyl alcohol, phenyl-ethyl alcohol, rhodinol (citronellol), geraniol, nerol, linalool, eugenol, farnesol, acetaldehyde, salicylaldehyde, nonylic aldehyde, benzaldehyde, furfural, citral, phenylacetic acid, methyl salicylate, coumarin and other lactones, amylamine, trimethylamine, cadinene, triacontane, and stearoptene.

Salicylic acid has been frequently identified in the distilled juices of several fruits, but it is not certain whether it pre-exists, as has been often claimed, in the form of methyl salicylate, in the juice of apple, strawberry, raspberry, cherry, and plum (Naves & Mazuyer, 1947. *Natural Perfume Materials*: p. 182).

## 1. CRATAEGUS Tourn. ex Linn.

(From the Greek *krataigos* — a flowering thorn.)

This genus comprises about 100 species which are usually spiny shrubs or small trees, the hawthorns, having white or pink flowers in terminal corymbs, and small berry-like pomes. In India it is represented by 4 species, all of which are confined to the Himalayas. Some species are cultivated in foreign countries on account of their ornamental flowers.

Although many species of this genus yield an essential oil, the hawthorn perfume of the market is not a natural product but is manufactured synthetically. The base for all perfumes of this type is anisaldehyde, but the odour is rounded off with acetophenone and finished with rose otto or other floral extracts.

Some of the constituents of the essential oils from flowers of some of the species are trimethylamine, amylamine, and sometimes ammonia. Similarly constituted oil has also been obtained from the leaves of some species, such as *C. monogyna* Jacq. of Europe.

### *Crataegus oxyacantha* Linn.

Fl. Brit. Ind., II, 383.

(Hawthorn, Maybloom, *Ban-sangli*)

This is a small tree with shining, often lobed leaves, white or pink fragrant flowers, and small red fruits often called "haws". It is found in the W. Himalayas from the Indus to the Ravi at altitudes of 4,000 to 9,000 ft. It is often cultivated for the sake of its flowers and edible fruit, which is said to be better than that of the European hawthorn.

The hawthorn has been regarded by the Greeks as a tree of fortune, and is said to be used for decorations during the May Day festivities of some European countries.

The flowers have a spicy odour, which recalls that of almonds. They yield 0.157 per cent. of an essential oil which contains trimethylamine (Wehmer). The natural oil is not an article of commerce.

## 2. GEUM Linn.

(The Latin name of herb bennet, *G. urbanum* Linn.; from the Greek *geuo* — to stimulate; referring to the aromatic roots.)

This is a genus of perennial herbs, having white, yellow or purple flowers. It



FIG. 37 — *Crataegus oxyacantha* Linn.

comprises about 40 species, which are natives of temperate and arctic regions, but only 2 are found in the temperate and alpine Himalayas.

**Geum urbanum** Linn.

Fl. Brit. Ind., II, 342.

( *Avens*, Cloverroot, Herb Bennet )

This is an erect perennial herb with highly dissected leaves, yellow flowers  $\frac{1}{2}$  to  $\frac{3}{4}$  in. across, and hairy fruitlets. It is found in the temperate W. Himalayas from Murree to Kumaon at an altitude of 6,000 to 11,000 ft.

The root is aromatic, having a clove-like odour, and is sometimes chewed to sweeten the breath. Owing to its astringent

properties it has been employed in Europe in cases of dysentery and diarrhoea. It was also used to flavour ale in olden times.

Fresh comminuted roots, after standing for 12 hours, yield 0.1 per cent. of an oil on distillation, the bulk of which is eugenol (benzoyl eugenol). The roots contain a glycoside, "gein", which gets hydrolysed by an enzyme, "gease", also present in the root, leading to the formation of eugenol (Bourquelot & Hérissé, 1905. *C.R. Acad. Sci., Paris*, 140 : 870 ; Hérissé & Cheymol, 1925. *Ibid.*, 180 : 384).

The dry root was distilled by Haensel (1903. *Chem. Zbl.*, 1 : 1137), who obtained 0.022 per cent. of a reddish-brown oil with an aromatic odour, bitter taste, and sp. gr.  $13.5^{\circ}$  1.037.

FIG. 38 — *Geum urbanum* Linn.

### 3. PRUNUS (Tourn.) Linn.

(The classical name of the plum tree.)

This genus comprises 85 species of mostly unarmed shrubs or trees of north temperate regions. Of the 15 species found in India, the apricot (*P. armeniaca* Linn.), peach [*P. persica* (Linn.) Stokes], almond (*P. amygdalus* Batsch), sweet cherry or gean (*P. avium* Linn.), plums and prunes (*P. domestica* Linn.), *alucha* (*P. communis* Huds., var. *insititia* Hook. f.), cherries (*P. cerasus* Linn. and others) are well known.

The most important feature of this genus is the occurrence of cyanogenetic or hydro-

cyanic-acid-producing glycosides in the seeds of many of its representatives. These get decomposed by the enzymes (emulsin in almonds), which usually co-exist in the plant. Amygdalin, the glycoside of bitter almonds, is a typical example. It is also found in small amounts in peach kernels, plums, cherries, etc. In some plants the leaves also contain cyanogenetic glycosides, such as prulaurasin in the leaves of cherry laurel (*P. laurocerasus* Linn.), one of the ornamental shrubs of S. Europe.

*P. acuminata* (Wall.) Hook. f., a moderate-sized or small tree of the Central and Eastern Himalayas and Khasi Hills, bears small, white, scented flowers in axillary racemes.

### 1. *Prunus amygdalus* Batsch

*P. amygdalus* Baill., Fl. Brit. Ind., II, 313.

(Almond Tree, *Badam*)

It is a small to medium-sized tree which is cultivated in the cooler parts of the Punjab and Kashmir. It flowers profusely in the spring, before the leaves appear. The flowers are white, tinged with red. The drupes have a velvety, dry pericarp which separates into two valves when ripe. The stone is compressed, with shallow wrinkles and minute holes.

Two varieties of almond, viz., *amara* and *dulcis*, the bitter and the sweet, are well known even though no botanical characters are known to distinguish one from the other.

Apart from the fatty oil, the bitter almonds yield an essential oil, known as "oil of bitter almonds", to the extent of 0.5 to 0.8 per cent. The bitter almond oil containing hydrocyanic acid is a strongly refractive liquid which, when fresh, is colourless, but acquires a yellow colour on standing. It has the predominating smell of hydrocyanic acid which is present and should, therefore, be smelt with care. The oil has sp. gr. 1.045 to 1.070, acid val. 0 when fresh,  $[\alpha]$  0°,  $n$  1.532 to 1.544, and is soluble in 1 to 2 volumes of 70 per cent. alcohol. The content of hydrocyanic acid varies, being as high as 14 per cent. in the crude oil. It goes down on rectification.

Bitter almond oil from which hydrocyanic acid has been removed is a colourless, optically inactive liquid having b.p. 179°, sp. gr. 1.050 to 1.055,  $n$  1.542 to 1.546, and is soluble in 1 to 2 volumes or more of 70 per cent. alcohol. The above characteristics are comparable with those of benzaldehyde which is the main constituent of the oil that has been freed from hydrocyanic acid. Hydrocyanic acid is not present as such in the bitter almonds, but is formed during the hydrolysis of the glycoside amygdalin by the action of the enzyme emulsin which also is present in the bitter almonds.

In order to obtain the oil of bitter almonds, the fatty oil (which is present to the extent of 35 to 60 per cent.) is first extracted by pressing through a hydraulic press. The oil cake is then finely ground and suspended in 6 to 8 parts of boiling water and allowed to stand for a few hours. When it has cooled to about 50°C., about  $\frac{1}{2}$  part of fresh bitter almond paste in 6 to 8 parts

of water is added to start the enzymic action. The mixture is allowed to stand for 12 hours at about 50°C., by which time the fermentation should be complete. This mixture is now ready for distillation with steam. Hydrocyanic acid is highly poisonous and, therefore, fullest precautions should be taken to prevent its escaping into the air in a factory.

As has been stated before, the bitter and sweet varieties of almonds have no characteristic distinguishing features. Further, the sweet variety, which is far more common than the bitter, is devoid of the essential oil. For this reason and the high cost of almonds, the commercial oil of bitter almonds is mainly derived from the bitter kernels of some "varieties" of apricot (*P. armeniaca* Linn.) and peach [*P. persica* (Linn.) Stokes]. The essential oil of apricot and peach kernels is similar to that from bitter almonds. The yield of the essential oil of apricot kernels is 0.6 to 1 per cent. and that of the fatty oil 35 to 40 per cent.

The oil of bitter almonds contains benzaldehyde, hydrocyanic acid, and benzaldehydecyanhydrin (or mandelonitrile). The common adulterant of the oil is, therefore, synthetic benzaldehyde. Benzaldehyde gets readily oxidized to benzoic acid when left exposed to air. Bitter almond oil containing hydrocyanic acid, on the other hand, is much less prone to oxidation, the latter acting as a preservative.

Hydrocyanic acid can be removed from the oil of bitter almonds by mixing with water, red oxide of mercury, slaked lime, and ferrous chloride, out of contact with air, and heating on a water bath. The hydrocyanic acid gets removed and the oil rectified.

The United States Pharmacopoeia XII requires that the oil should not contain less than 80 per cent. of benzaldehyde, and not less than 2 per cent. and not more than 4 per cent. of hydrocyanic acid. This oil is intended for medicinal use and neither it nor its solution should be used for flavouring food. For flavouring purposes hydrocyanic acid is more or less completely removed, and such an oil is known as *oleum amygdalae*, S.A.P. (= *sine acido prussico*).

Even though the synthetic benzaldehyde is equivalent to the oil of bitter almonds, the latter is preferred by manufacturers of flavouring materials since it has a superior flavour due perhaps to small amounts of



FIG. 39 — *Prunus amygdalus* Batsch

other constituents, whose nature has not yet been investigated.

Bitter almond oil is used in pharmacy as a nerve sedative and as a base for soothing skin lotions. It is used in small quantities in such perfumes as heliotrope, and has also a wide application in the soap industry. It is also largely used as a flavouring agent in confectionery, liquors, etc.

## 2. *Prunus armeniaca* Linn.

Fl. Brit. Ind., II, 313.

(Apricot, *Khubani*, *Zardalu*)

The apricot is a medium-sized deciduous tree having pinkish flowers which turn white

after some time. The flowers appear before the leaves, and are either solitary or in bunches. The fruit is downy with a thickened and grooved margin. It is commonly cultivated in N.-W. India, especially in the hills at altitudes of 4,000 to 9,000 ft., and occasionally grows as self-sown.

Apricot is the commonest fruit in the Punjab Himalayas, and is eaten both fresh and dried. The seeds are eaten in the same way as almonds, and in fact form a valuable substitute. The oil expressed from these is largely used as an illuminant, for cooking, etc. As in the case of almonds, there is also a "bitter variety" of the seeds which is undoubtedly poisonous.

Apricot kernel of the bitter variety is the main source of the commercial "oil of bitter almonds". It yields 1.6 per cent. of the essential oil (Rabak, 1908. *U.S. Dept. Agr. Bur. Plant. Ind.*, 133: 23). The method of winning the oil is the same as described under bitter almonds. The uses of the oil are also similar.

### 3. *Prunus avium* Linn.

Fl. Brit. Ind., II, 313.

(Cherry, Sweet Cherry, Gean)

This is a medium-sized tree, with sweet or bitter, nearly black fruit having a smooth stone. It is cultivated in the N.-W. Himalayas up to an altitude of 8,000 ft., especially in Kashmir.

The fruit is well known and much valued as a dessert. The seed-kernel contains amygdalin (Wehmer).

### 4. *Prunus cerasus* Linn.

Fl. Brit. Ind., II, 313.

(Cherry, *Alubalu*, *Gilas*)

This is a deciduous shrub or a small tree bearing globose fruit which is light red or nearly black in colour, and acid or sweet in taste. The stone is smooth. It is cultivated in the Himalayas of the Punjab and N.-W. Frontier Province up to an altitude of 8,000 ft. The fruit is edible, and the kernel is used for flavouring liquors in Europe.

The seed-kernels as well as the leaves contain the cyanogenetic glycoside amygdalin (Wehmer).

### 5. *Prunus mahaleb* Linn.

Fl. Brit. Ind., II, 312.

(*Khwati*, *Mahaliib*)

This is a small, much-branched shrubby tree with white, fragrant flowers in peduncled corymbose racemes. The drupe is  $\frac{1}{4}$  in. in length, ovoid and tipped with a short, blunt point. It is a native of Central Asia and Central Europe, probably also occurring in N.-W. India. It is cultivated in Baluchistan.

The "scented kernels" are sold in the bazars of N.-W. India for use in indigenous

medicine (Watt). The seeds are stated to contain amygdalin (Wehmer), and, when chewed, have a strong odour of hydrocyanic acid (Dymock, Warden & Hooper, 1890. *Pharmacographia Indica*, 1: 567).

### 6. *Prunus padus* Linn.\*

Fl. Brit. Ind., II, 315.

(Bird Cherry, *Jamnoi*)

This is a small to medium-sized deciduous tree, the leaves turning red in autumn. The drupe is  $\frac{1}{2}$  in. in diameter, globose, acid to taste, of reddish colour turning to dark purple or black. It is a native of the temperate Himalayas, occurring at 4,000 to 12,000 ft.

According to earlier workers almost all parts of the plant (leaves, flowers, seeds, and bark) contain the cyanogenetic glycoside amygdalin (Wehmer). Recent work shows that the glycoside found in the twigs is prunasin (mandelonitrile glycoside) which, on hydrolysis, yields hydrocyanic acid, benzaldehyde, and glucose (Hérissey, 1912. *C.R. Acad. Sci., Paris*, 154: 1249).

The heartwood has an unpleasant smell when first exposed.

### 7. *Prunus persica* (Linn.) Stokes

*P. persica* Benth. & Hook. f., Fl. Brit. Ind., II, 313.

(Peach, Nectarine, *Aru*)

This is a large deciduous shrub or a small tree, bearing pink flowers which appear before or sometimes with the leaves. The fruit is succulent and its stone deeply and irregularly furrowed. Peach is a form with downy pericarp, and nectarine with a glabrous one.

Peach is widely cultivated in India, especially in the N.-W. Himalayas where some of the finest varieties of the fruit are grown. It also occurs in a naturalized state in the north-west up to an altitude of 10,000 ft. It is also cultivated extensively in the plains of the Punjab and N.-W. Frontier Province, and in the Nilgiris between altitudes of 5,000 to 7,000 ft.

The fruit pulp, on distillation and solvent extraction of the distillate, yields 0.0008 per cent. of a pale-yellow essential oil of peach odour. The reported constituents

\*Some authors prefer to call it *Prunus cornuata* Wall.

of the oil are acetaldehyde, furfural, cadinene and linalool, and esters of formic, acetic, valeric and caprylic acids (Power & Chesnut, 1921. *J. Amer. chem. Soc.*, **43**: 1725; 1922. *Ibid.*, **44**: 2966).

The seed-kernels contain amygdalin and yield about 0.7 per cent. of an essential oil, which contains chiefly hydrocyanic acid, benzaldehyde, and benzaldehydecyanhydrin (Rabak, 1908. *U.S. Dept. Agr. Bur. Plant. Ind.*, **133**: 23). These are one of the sources of commercial "oil of bitter almonds".

The flower, leaf, and bark have the peculiar odour and taste of bitter almonds, and are stated to contain hydrocyanic acid.

#### 8. *Prunus puddum* Roxb. ex. Wall.\*

Fl. Brit. Ind., **II**, 314.

(Wild Cherry of the Himalayas, *Padam*)

This is a moderate-sized to a large tree of brilliant appearance when in flower. The flowers are  $1\frac{1}{2}$  to  $1\frac{3}{4}$  in. in diameter, pink or white, and appear before the leaves. The drupe is  $\frac{1}{2}$  to  $\frac{3}{4}$  in. in length, oblong or ellipsoid, obtuse at both ends, with scanty, reddish or yellowish, acid flesh, and wrinkled and furrowed stone. It is found wild in the temperate Himalayas from Garhwal at altitudes of 3,000 to 6,000 ft. to Sikkim and Bhutan from 5,000 to 8,000 ft., and is also met with in the hill stations of Kodaikanal and Ootacamund in S. India.

The leaves and kernels are said to contain a substance which yields hydrocyanic acid (Greshoff, 1906. *Arch. Pharm., Berl.*, **244**: 670). The bark is also stated to contain a hydrocyanic-acid-yielding substance, and the smaller branches are used in indigenous medicine as a substitute for hydrocyanic acid (Watt).

The heartwood has an evanescent scent.

#### 9. *Prunus undulata* Buch.-Ham.

Fl. Brit. Ind., **II**, 316.

(*Aruwa*)

This is a medium-sized deciduous tree with rounded crown. It bears white flowers and ovoid drupes. It is found in the temperate Himalayas from Kumaon at altitudes of 6,000 to 8,000 ft. to Sikkim and Bhutan

from 8,000 to 12,000 ft., and also in the Khasi Hills.

The leaves and fruits contain substances which yield hydrocyanic acid (Greshoff, 1906. *Arch. Pharm., Berl.*, **244**: 670).

#### 4. *PYRUS* (Tourn.) Linn.

(The classical name of the pear tree.)

This genus comprises about 65 species of shrubs or trees of north temperate and cold regions. Economically it is an important genus in so far that fruits from many of its species, such as the apples (*P. malus* Linn.) and pears (*P. communis* Linn.), are edible and highly prized.

The essential oil is usually found in the fruit skin, though the flowers also in some cases have a smell. The flowers of *P. aucuparia* Gaertn., for example, have the smell of hawthorn (*Crataegus oxyacantha* Linn.) flowers, and the flowers of a foreign species are stated by Wehmer to smell of ammonia. Hydrocyanic acid is present in many species, particularly in their seeds.

#### 1. *Pyrus aucuparia* Gaertn.\*

Fl. Brit. Ind., **II**, 375.

(Mountain Ash, Rowan Tree, *Battal*)

This is a small tree with pinnate leaves, pink flowers  $\frac{1}{2}$  to  $\frac{1}{2}$  in. in diameter, and red globose fruits. It occurs in the temperate W. Himalayas from Kashmir to Kumaon at altitudes from 9,000 to 13,000 ft.

The flowers have the smell of hawthorn (*Crataegus oxyacantha* Linn.) flowers, and yield trimethylamine (Wehmer).

The leaves, buds, young twigs, and bark are said to contain amygdalin (Wehmer).

The fruit resembles in size and flavour that of the same tree in Europe. In certain parts of Europe it is made into a jelly, and also used in the preparation of a liqueur or cordial.

The defatted seeds were found to contain about 0.073 per cent. of hydrocyanic acid (van Itallie & Nieuwland, 1906. *Arch. Pharm., Berl.*, **244**: 164).

#### 2. *Pyrus malus* Linn.

Fl. Brit. Ind., **II**, 373.

(Apple Tree, *Seb*)

This is a small tree rarely exceeding 30 ft. in height. It is largely cultivated in

\*Some authors prefer to call it *Prunus cerasoides* D. Don.

\*Some authors prefer to call it *Sorbus aucuparia* Linn.

the Himalayas, the Punjab, Sind, N.-W. Frontier Province, Central India, and the Deccan. It is also found wild in the N.-W. Himalayas.

The apple is a well-known fruit, occupying the foremost place among fruits of temperate regions in importance and extent of cultivation. It also forms the basic raw material for the cider and pectin industries.

The fruit skin of apple yields, on distillation with steam, a very small quantity (0.00065 to 0.0035 per cent.) of an essential oil, which soon congeals. This on treatment with ether and subsequent treatment with alcohol can be separated into a solid part and an oily part. The yellow oily portion has the odour of apples.

The nature and the constituents of the essential oil differ with different horticultural varieties. The oil from the variety known as "Ben Davis", for example, contains acetaldehyde, amyl alcohol, furfural, formic, acetic, caproic and caprylic esters, a solid crystalline waxy substance of the nature of triacontane, etc. (Power & Chesnut, 1920. *J. Amer. chem. Soc.*, **42**: 1509). The oil from the "McIntosh" variety contains geraniol, citral, amyl ester, etc. (Wehmer). The proportion of these constituents varies according to the varieties, and this accounts for the differences in odour.

The seed-kernels contain amygdalin (Rosenthaler, 1912. *Arch. Pharm., Ber.*, **250**: 298).

## 5. ROSA Tourn. ex Linn.

(The classical name.)

This is a genus of well-known erect, sarmetose or climbing shrubs, the roses, having odd-pinnate leaves, mostly prickly stems, red, white, pink or yellow (never blue) flowers with many stamens, and urn-shaped calyx tube which becomes fleshy in fruit (rose hip) enclosing the achenes. It comprises about 150 species which are widely distributed in the north temperate regions and on tropical mountains. In India it is represented by eleven species (excluding those cultivated). Over four thousand horticultural varieties, with many petals, are recognized. These varieties, which have been mainly derived by repeated hybridization of about 30 wild species, are divided

according to habit or culture into 43 classes or races of which the following are the most important: climbing tea, polyantha, hybrid perpetual, hybrid tea, multiflora, Noisette, and tea. A number of species with numerous "varieties" have been introduced into India and are cultivated in public and private gardens, mainly for ornamental purposes. Not all of them are highly fragrant and rich in oil content; in fact some of them are more or less inodorous. It is only in a few that the oil content is high and they alone are fit for extraction of the oil. Of the indigenous species the following have fragrant flowers: *R. anserinaefolia* Boiss. of Waziristan and Baluchistan, *R. gigantea* Collett ex Crép. of Manipur, *R. macrophylla* Lindl. of the temperate Himalayas, *R. moschata* Herrm. (musk rose) of the W. Himalayas, *R. longicuspis* Bertol. of the Khasi Hills, and *R. sericea* Lindl. of the temperate Himalayas.

According to Parry, "It is of interest to note that the odorous organs of the rose may be divided into two categories—firstly, the petals of the flower; and secondly, certain green parts of the plant, which include the calyx and sometimes the green leaves. These two sets of organs work in the evolution of the perfume independently of each other, to such an extent that no case is known where the perfume of the two organs are identical in the same plant. For example, the *Rubiginosae*, of which the sweet briar is typical, has odourless flowers, whilst its leaves are delightfully fragrant. And when this rose is hybridised, as it has successfully been by Lord Penzance, the so-called Penzance briars (all hybrids) have sweet-scented foliage."

Only a few species of *Rosa* have been examined for their essential oil, and of these may be mentioned: *R. canina* Linn. (dog rose) of Europe and N. Asia, *R. damascena* Mill. (damask rose) cultivated in S. Europe, India, Arabia, Persia, Bulgaria, Turkey, and N. America, *R. centifolia* Linn. (cabbage rose, garden rose) cultivated in many countries including India, *R. gallica* Linn. (French rose) of middle and S. Europe and also grown in India, *R. moschata* Herrm. (musk rose) of N. Africa and India, *R. indica* Linn. of China and cultivated in India, and *R. alba* Linn. of Bulgaria but also grown in India. The characteristic rose perfume is well developed in *R. damascena*, *R. centifolia*, *R. gallica*, and *R. alba*. The species which, in Asia, is mostly grown for the preparation

of otto or attar\* is *R. damascena* with a very generous yield of flowers; *R. centifolia* is also used for this purpose.

The yield of the oil from different species is different, varying from 0.015 to 0.050 per cent. on fresh petals (or 1 part of oil in 2,000 to 6,000 parts of petals), and the nature of the constituents is also different. In general the constituents are geraniol, rhodinol (citronellol), linalool, eugenol, nerol, farnesol, phenylethyl alcohol, esters, citral, nonylic aldehyde, and stearoptene (Bertram & Gildemeister, 1894. *J. prakt. Chem.*, **49**(2): 185; 1896. *Ibid.*, **63**(2): 225; Schimmel & Co., 1890, Oct. *Rep.*; 1900, Oct. *Ibid.*: p. 53; Soden & Rojahn, 1900. *Ber. dtsh. chem. Ges.*, **33**: 1720; Soden & Treff, 1904. *Ibid.*, **37**: 1094).

Apart from the essential oil the rose flowers contain a bitter principle, red colouring matter, tannin (2.5 per cent.), fatty oil (1.7 to 2.6 per cent.), organic acids (about 3 per cent.), quercitrin, and a wax. The rose petal wax melts at about 60°C., and has been fully described by Prophète (1926. *C.R. Acad. Sci., Paris*, **182**: 1559). Along with the above about 2 per cent. of saccharose and 10 to 15 per cent. of invert sugars are also present. In India the rose petals are converted into a preserve with sugar and the preparation is known as *gulkand*. This preparation is used as a mild purgative and is a very pleasant medicine to take.

The rose hip is one of the richest sources of vitamin C, and for this reason was and still is in demand.

### 1. *Rosa centifolia* Linn.

Fl. Brit. Ind., II, 364.

(Cabbage Rose, Hundred-leaved Rose, *Gulab*)

This rose is often cultivated in Indian gardens. Next to *R. damascena* Mill., it is the most important rose used in perfumery. In India, however, it is seldom used for the extraction of the perfume. For further details, the reader is referred to what has been written under the account of *R. damascena*.

\*Although attar, according to various dictionaries, means otto or essential oil, in India it has come to mean, particularly in reference to rose, that oil which has been distilled in combination with some quantity of sandalwood oil.

### 2. *Rosa damascena* Mill.

Fl. Brit. Ind., II, 364.

(Damask Rose, Persian Rose, Rose, *Gulab*)

This is an erect shrub armed with curved unequal-sized prickles mixed with prickly and glandular bristles. The flowers are rosy pink, borne in bunches of 5 to 10. The sepals are bent backwards after flowering. Flowering occurs in February to April.

This is the commonest Indian garden rose, being cultivated for its beautiful fragrant flowers, and is the main species grown at Ghazipur, Hathras, Patna, Lahore, Amritsar, and several other places in India. This is the rose from which essential oil (otto or attar) and rose water have been manufactured in India for several centuries past.

The charm of the flower and its delightful fragrance have been appreciated since earliest antiquity, and for 5,000 years and more the rose has sweetened and often sanctified the life of man all the world over. From Persia and the Shah's own gardens one may trace its itinerary until, today, there is nowhere outside the frozen wastes where it does not bloom and give infinite joy. It graces the humble cottage and adds more than lustre to the vases in palaces. Rose throughout the ages has decked the bride and garlanded emperors and prelates. Indeed, no other flower or perfume has been so persistently popular as rose.

*History*: The distillation of roses probably originated in Persia even before the start of the Christian Era, but the earliest documentary evidence of rose water dates back to 810 A.D. At this period, in the reign of the Caliph Mamoun, the province of Farsistan was compelled to provide annually a tribute of 30,000 bottles of rose water to the treasury at Baghdad. Considerable quantities also used to be exported from Farsistan to India, China, Egypt, etc. The first authentic description of essential oil occurs in a work written by Geronimo Rossi of Ravenna who in 1574 observed small drops of the oil floating on the surface of rose water. In India the otto of roses is said to have been discovered by Nur Jehan, the wife of the Moghul Emperor Jehangir, in 1612 A.D., when she noticed a film of oily layer in a 'canal' filled with rose petals and water, in a palace in Delhi. She is said to have had this delicate perfume skimmed with a feather, and followed this practice to obtain her supply of rose oil. This discovery led



FIG. 40 — *Rosa damascena* Mill.

to the establishment of rose-oil and rose-water industry in India, particularly in Kanauj and Ghazipur where the industry has existed for at least 300 years, and even today Ghazipur and Hathras produce the bulk of whatever little rose oil or water is manufactured in India.

Rose cultivation in India is said to have been started about the seventeenth century, and in Bulgaria in the year 1710. By

1750 Bulgaria had become the principal source of supply of rose otto (Parry).

*Cultivation*: Rose plantations are raised almost invariably from cuttings. When cultivated for the purpose of oil, those varieties are selected which have a fine aroma, are rich in the yield of oil, produce larger number of flowers, and are resistant to climatic conditions. It is said that *R. damascena* satisfies these conditions,

even though in shape and appearance it is not so attractive as many other garden varieties. In point of fact, *R. damascena* is a cultural variety not known in wild state, and is presumably a hybrid between *R. gallica* Linn. and *R. canina* Linn. with the characteristics of the former predominating.

The cuttings from old bushes are made about the beginning of the cold weather (November) and planted in small pots or baskets. In about 3 months they root and by February leaves appear when they are removed from the pots, earth and all, and placed in beds of sandy loam, previously prepared, and manured with farmyard manure and superphosphate at the rate of one ounce to a square yard. The planting is done about 3 ft. apart and nearly double this distance between the rows, and in this way 2,500 plants can be accommodated in an acre. The plantation is watered freely and frequently during the hot and dry weather. Early and excessive drought is harmful to the rose crop and so is excessive rain that helps flourishing of several pests which may ruin the entire crop. By March the following year, i.e., 15 months after starting, the plantation should be ready for harvesting though the maximum amount of flowers is not to be expected till the fourth or fifth year.

In Bulgaria the plant is propagated through root cuttings. The old bushes are cut down and the roots pulled out and separated into as many as possible. These are then replanted four or five abreast, in long trenches about 18 in. wide and deep, manured and watered, so as to form one hedge 80 to 100 yds. long. The distance between two hedges is about 6 ft. In order to combat the fungal disease to which rose is liable, spraying with Bordeaux mixture (with 1 per cent. while the plant is dormant, with 0.5 per cent. just before the blossoms open, and again with 1 per cent. after flowering) is recommended (Georgieff, 1925 *Perfum. essent. Oil Rec.*, 16 : 441). Like in India, the planting is done in November, and by the following May young shoots appear and a year afterwards the plants are in full flowering. The maximum number of flowers appear between the fourth and tenth years. After about 10 years the plants are rejuvenated by cutting down the branches to ground level to allow new shoots to come up and form the original hedge. Rose shrubs thus handled produce flowers for 20 or 30 years.

It is well known that conditions of soil, irrigation, and climate are of prime importance in setting up this industry, since most extraordinary differences have been noted in the amount of otto yielded by the same rose when grown in different regions.

*Yield*: In Bulgaria one acre on an average yields 2,100 lbs. of flowers in one season (May) of 21 days (Gildemeister records 4,000 lbs. per acre in good plantations). A second flowering takes place sometimes in November, but, from the commercial point of view, it is said to be of little value. The yield of flowers in France varies from 1,800 to 4,500 lbs. per acre towards the fourth or fifth year, when the maximum production is realized. In Hathras, in the United Provinces, the yield of flowers is 500 to 600 lbs. per acre per year. Experiments on cultivation tried in Cawnpore, in the United Provinces (Srivastava & Sinha, 1921. *Perfum. essent. Oil Rec.*, 12 : 14), show that a yield of 1,200 lbs. may be readily obtained, and that there is a possibility of increasing this to at least 1,500 lbs. per acre per year.

*Collection*: Rose flowers are usually collected before they begin to open. The harvest lasts for 3 to 6 weeks and the crop must be worked up for the oil immediately. The gathering is done early in the morning before the sun gets hot. It is essential that the flowers be not picked with the hot sun on them because the flowers picked during the heat of the day not only give a lower yield of the otto but the otto also is not so sweet in fragrance. Not only are the yield and quality of the otto affected by picking the flowers during the hot part of the day but they are also affected by storage of the flowers.

Srivastava & Sinha (*loc. cit.*) have shown that the high temperature which prevails in Cawnpore, during April, has a marked influence on the yield of the otto. Experiments conducted right in the open under the heat of the sun and unprotected from the hot wind gave a yield of 0.01 per cent. of otto. Simultaneous experiments in a covered shed improved the yield to 0.015 per cent. When protected with wet screens to keep away the hot winds and to reduce the temperature of the air surrounding the receiver the yield improved to 0.02 per cent. Further, comparative experiments using freshly plucked flowers and flowers which had been kept for 24, 48 and 72 hours, respectively, showed that fresh flowers were the only ones suitable for distillation; those which had been kept

for 72 hours had lost the whole of the oil. Flowers picked before sunrise and distilled immediately gave 0.025 per cent. of otto. Flowers which are left for some time before distillation tend to get partially fermented though the fermentation can be prevented, to an extent, by salting and keeping in a cool place.

*Preparation of rose otto in Bulgaria:* According to Poucher<sup>7</sup> (1936. *Perfume, Cosmetics & Soaps*, 2: 212), there are four systems of extracting the perfume from the flowers, and all of these are practised in Bulgaria. They are as under:

1. Open fire with small alembics.
2. Open fire with large alembics.
3. Steam stills including vacuum and rotary apparatus.
4. Volatile solvent extraction.

*"Distillation with Small Alembics* — Up to 1902 all the rose otto exported from Bulgaria was distilled by the growers themselves in small tinned-copper alembics. At that time there were in all 2,800 distillers, operating over 13,000 small alembics having a total capacity of nearly fifteen million kilos of rose blossoms. Now there are only about 300 peasant distillers with under 2,000 stills, the old system of distillation being continually replaced by modern methods, involving the use of large improved stills. There are at present over 50 new modern plants, operating some 400 large open-fire stills and 30 steam stills. These modern factories distil nearly 80 per cent of the entire rose crop, and the growers only 20 per cent of it. The latter generally use the old-fashioned *gulapana*, which constitutes one of the out-buildings of the local farmer. The small alembics are some 40 to 43 inches high and have a base of about 32 inches diameter. There are two handles, one on each side for lifting when charging and discharging. They are constructed as near as possible to a stream so that the supply of water for distillation and condensation is assured. The apparatus is comparatively crude and consists of a truncated cone copper boiler having a capacity of about 120 litres. The helmet top is mushroom shaped and has a straight condensing tube fitted into one side. This runs through a wooden vat containing the cooling water, and where it emerges below there is a cavity in the ground which acts as a receptacle for the glass receiver. The stills are generally placed in rows on a low brick hearth. Wood is used as fuel.

" On arrival at the *gulapana* the red and white roses are mixed and distilled at once. During the busiest times it is often necessary to spread the roses out in a cool place to await distillation when they are moistened with cold water to prevent fermentation. Ten kilos of petals and 75 litres of water are placed in each boiler, the apparatus is fitted together and sealed. The fire is then lighted and it takes from 1½ to 2 hours for complete distillation. The yield of rose water and otto measures about 10 litres and this is placed aside. The apparatus is then emptied, the exhausted roses separated by strainer and the hot water returned to the boiler for the second distillation. This generally measures about 50 litres, and so another 25 litres of fresh water is added for the further charge of 10 kilos of rose blossoms. This process is repeated until the day's collection of flowers has been disposed off. The rose otto is separated from the reserved distillates by placing the products of four distillations (40 litres) together in the apparatus and carefully distilling the whole. The first 5 litres of cloudy distillate are reserved, and when this clears the otto is found floating on the surface. It is carefully removed and stored in glass flasks. The other 35 litres is left in the still and a fresh quantity of rose water added. The process is repeated and the otto separated.

" As will be observed, the peasant-farmer generally re-employs the residuary water remaining after each operation, and this constitutes a fundamental difference between his process and that of the larger manufacturers, where the exhausted flowers and *residuary water* are discarded. In the former case, the hot and dark coloured residuary waters give the farmer the advantage that he commences his new distillation with already heated water and so saves fuel. It will be obvious that the progressive concentration of extractive matter in these residuary waters will raise the boiling-point and cause constituents to be carried over with the distillate which increase the yield of oil. Since, however, such residuary waters in time assume a sharp and distinctly unpleasant odour, the increased yield of oil is largely discounted by the fact that the quality is impaired. Nowadays many farmers have observed this disadvantage and, like the larger distillers, completely discard both exhausted blossoms and water together, thus dispensing with the use of sieve or strainer as the case may be. The rose oil



made by this primitive method is known as *Peasant Quality* and possesses a soft, sweet, honeyed odour lacking in strength. It is cheaper than the oil distilled in the large alembics because, in the first place, the farmer does not 'cost' his labour and that of his family, and in the second place the capital involved is comparatively insignificant. This type of oil successfully competes with ottos produced by other means of distillation.

"*Distillation with Large Alembics* — There are over 50 large distilleries belonging to private firms or to co-operatives. These contain some 400 large open-fire stills having a capacity varying from 500 to 2,500 litres and are capable of distilling 100 to 500 kilos of roses at each operation. They are usually made of copper and may have either fixed or detachable heads. Different forms exist, some being the flat type of still and others the erect cylindrical apparatus. In all cases they differ from the small former type in that they are built into hearths so that no loss of heat occurs. Moreover, they have a perforated platform inside near the base to prevent the flowers coming into contact with the direct heat of the fires beneath. Worm condensers are almost invariably used, and the receivers resemble large florentine flasks having cylindrical glass tops for the observation of the oil as it collects on the surface. In the fixed-head type of still the flowers are charged through a manhole direct from the sacks and until the blossoms are within a foot of the top. Water is then run in until the flowers are completely covered.

"A hefty worker stirs the whole mass with a long pole to ensure the separation of the blossoms so that they will float freely once the operation has commenced. (Coagulated groups of flowers would interfere with the complete removal of the essence.) In normal times the ratio of water to flowers is strictly observed, being 4 to 5 litres to 1 kilo of roses. The ratio is only decreased with pressure of work when the crop is exceptionally abundant. The charging orifice is closed and the fire lighted. Considerable experience is here necessary. The application of heat must be gentle, otherwise a too sudden rise in temperature would drive out some of the lighter perfume constituents with the air remaining in the still and would thus be lost to the detriment of the subsequent oil distillate. As a rule it takes one and a half hours before any

condensation takes place, and from two to two and a half hours more to complete the distillation. The distillate passes through the condenser, which is comparatively hot at the top and not very cold at the bottom. The temperature of the distillate is kept at just over 35°C., otherwise the stearoptene would crystallise inside the condensing tubes. The distillation is not stopped until about 1 litre of rose water is collected for each kilo of flowers in the still, the actual volume depending upon pressure of work. The contents of the still, that is, exhausted flowers and water, are emptied from a side exit and pass down shoots into a river. The distillate flows from the condensers into the receivers, arranged in series of two for each still, and the rose water constantly passes from the second into open tanks having a capacity of 100 to 150 litres. When full, the contents are automatically pumped into large storage tanks for subsequent redistillation or cohobation. The oil floats to the surface of the receivers and appears in the glass cylindrical top already referred to. It is usually a pale yellowish-green crystalline mass, containing all the stearoptene and the more highly aromatic constituents. This is the **Direct Oil** and is known locally as *Surovo maslo*. It is removed by either pipette or spoon and transferred to glass bottles for the time being. The yield is comparatively small, because the major portion of the oil remains dissolved or is emulsified and in suspension in the rose water pumped into the storage tanks. This is known as the *first waters*, and when a sufficient quantity has accumulated it is transferred to the stills for cohobation or redistillation. About 1,200 litres are run into each still. The fire is lighted and heat applied gently. In an hour and a half the distillate begins to pass and the operation is completed in another hour. The condensers are operated cold since no stearoptene is present in this distillate. When some 150 litres of rose water have been collected, the distillation is stopped and roses added to the residual waters in the still. (The difference between this residual water and the peasant residuary waters above referred to will be apparent.) Distillation commences again for the production of direct oil. The above 150 litres of rose water constitute the *second waters* and are pumped into the storage tanks to join other first waters therein and to await cohobation. The oil which separates in the florentine flasks from the

distillation of the first waters is known as the **Water Oil** and is referred to locally as *Prevarka*. This is always fluid. It is removed and mixed with the **Direct Oil** to constitute the Rose Oil of commerce. The yield from the first and second distillation is, from the author's actual experience in Bulgaria, never a constant ratio; even though seemingly identical conditions were observed for repeated distillations. The approximate calculations made on the spot led to the estimate of 25 per cent direct oil as against 75 per cent water oil. These mixed runnings, as already stated, constitute the crude oil of roses. It is placed in glass bottles and exposed to the direct rays of the sun for a number of days, when impurities and colloidal matter are precipitated. The supernatant fluid is carefully decanted and filtered. Packing is done under the supervision of the local Excise authorities, who seal each of the well-known tinned copper vases and levy an export tax of about 4 per cent.

"*Steam Distillation*—This is carried out by a few of the larger manufacturers who have very efficient and modern apparatus, often specially constructed after much expensive experimental work. The steam is usually generated in a building adjacent to the distillery and the stills are as a rule larger than those employed for direct fire, sometimes having a capacity of 3,000 litres. Two types are in existence, one having a steam coil or steam jacket and the other having a perforated direct steam coil. The latter have the advantage of more quickly raising the temperature of the water in the still and incidentally replacing part of that distilled over during the operation of the plant. For all steam stills the process, flower ratios, etc., are much the same as those already described. Owing to the ease with which the steam can be controlled it is obvious that this constitutes a distinct advantage over the direct fire systems. It is not unusual for some of the larger manufacturers to have both steam and direct-fire stills in operation together. The oils from the two sources differ in odour and the latter are usually somewhat stronger and sharper, even though the bouquet is less fine. From the author's comparisons on the spot, a mixture of the two would seem to make the ideal rose otto, blended in the ratio of 2 of fire to 1 of steam oil. The largest plant has over 70 fire stills, 2 enormous steam stills and 4 concrete batteries,

all of which were in continuous operation a few years ago during the peak period of perfumery sales. To-day, alas, most of them are idle.

"*Vacuum Distillation*—There is one large plant in operation and the process differs slightly from the foregoing. In the first distillation no oil is separated, the product being entirely rose water which is pumped into storage tanks. It is then redistilled, and yields at one operation the whole of the oil. The odour of this rose otto is of a very special character and in the author's view would be exceptionally suitable for perfumes of the white rose type.

"*Rotating Apparatus*—This a modification of the plant used in Grasse for the extraction of flowers with volatile solvents, and was introduced into Bulgaria by Charles Garnier many years ago. It consists of a fixed vertical drum, inside of which perforated metal drums containing the blossoms revolve on a horizontal axle. They continually dip into the boiling water at the bottom of the apparatus and the flowers are rapidly exhausted. The actual period of extraction is considerably reduced, roughly to half that required for the fixed apparatus. Moreover, the ratio of water to flowers is less, being somewhere between three to two and two to one. Furthermore, the quantity of rose water distilled over is only half of that necessary in the fixed apparatus. Whereas in the latter the ratio of water oil to direct oil is about three to one, it is about two to one in the rotating apparatus. The principal difference in the oil obtained is that of a much lower stearoptene with a consequent increase in the odour to weight ratio."

*Volatile solvent extraction*: According to Poucher, a few of the modern factories employ this process which was first introduced into Bulgaria by Garnier in 1903. He installed a factory at Kare Sarli, containing six 12-tray extractors (the *roue* type). Details of this method have been given by Poucher (*loc. cit.*) and also by Naves & Mazuyer (1947. *Natural Perfume Materials*: p. 85, 235). According to Naves & Mazuyer six more factories were built after 1919, all but one equipped with stationary extractors; three of these were still working in 1939. Large quantities of flowers were treated in these factories, particularly from 1922 to 1930. In some years, the production of the concrete reached as high as

2,200 lbs., but fell to 450 to 650 lbs. between 1932 and 1935.

*Preparation of rose otto in Germany:* The Bulgarian peasant process is quite primitive and leaves much more room for improvement. In Germany roses picked in the morning are transferred directly to the large copper stills, each of which has a capacity of about  $1\frac{1}{2}$  tons of roses, in addition to the requisite water. The stills are not heated over direct fire but with steam; water is changed with every charge of flowers, and the otto is collected in a series of florentine flasks arranged as cascade. All this care results in producing a very superior otto and, although it has a high stearoptene content, it is twice as intensive in odour and it goes twice as far in perfumery blending. The yield of the otto is about 0.02 per cent.

*Preparation of rose otto in France:* The cultivation of rose is a very old industry in Grasse, the home of French essential oil industry. The rose which had till fairly recently been employed for distillation, almost to the exclusion of other varieties, was the well-known *R. centifolia* Linn.\* usually known as the May rose or *rose de Mai*. This rose is of exquisite fragrance, but requires much care in cultivation, and does not give a high yield of flowers. Its odour, however, is so charming that it is almost exclusively used for enfleurage purposes and for the preparation of rose water (Parry). Over and above *rose de Mai*, many new hybrids have been developed and they too are employed for the purpose of manufacturing concrete or otto. *Roses de Jardin*, which is a general name for all the garden roses like Brunner van Houtte and tea roses, have appeared in the market because of the higher yield of otto, even though their fragrance does not match that of *rose de Mai*. For instance, the fragrance of otto of van Houtte roses recalls that of a bouquet of mixed roses. This otto is distinctly green in colour when freshly distilled, but becomes yellow on keeping, melts at a lower temperature ( $15^{\circ}$  to  $16^{\circ}$ ), and has a high optical rotation frequently reaching  $-11^{\circ}$ .

\*According to several books on perfumery and essential oils, such as those by Finmore, Parry, and Poucher the species cultivated mostly in France and known by the name of *rose de Mai* is *R. centifolia* Linn., but, according to the latest book by Naves & Mazuyer (1947. *Natural Perfume Materials*: p. 234), it is *R. damascena* Mill. These authors also state that *rose de Mai* is the name for *R. damascena*.

According to Naves & Mazuyer (1947. *Natural Perfume Materials*: p. 234), whereas the greater part of the Bulgarian harvest is used in the production of rose otto, in France only a small amount of the essential oil is produced, distillation being practised mainly to obtain the rose water. The major portion of the crop in France is used in the manufacture of concrete, but a small proportion is also used to produce pomades and oils by digestion. According to Poucher, approximately 70 per cent. of the flowers are treated by volatile solvents (principally petroleum ether), 20 per cent. by maceration, and the remainder by distillation.

*Preparation of rose otto in India:* Otto of rose and rose water have been prepared in India for the past several centuries, and the methods followed have been more or less similar in principle to those followed in Bulgaria. An account of a more recent attempt to establish this industry is given by Srivastava & Sinha (1921. *Perfum. essent. Oil Rec.*, 12: 14), and Gadre & Mukerji (1922. *J. Indian Industr.*, vide *J. Soc. chem. Ind., Lond.*, 1922, 41: 192R). Srivastava & Sinha (*loc. cit.*) state as follows:

"The two main centres of the rose industry in Upper India are Ghazipur and Hathras. Rose plantations exist in the neighbourhood of both these places, but the industry is confined to the manufacture of rose water and small quantities of attar (mixture of sandalwood oil and otto of rose).

"Very little, if any, pure otto is made at either of these places, the reason being that the yield of otto is so small as to make the manufacture quite unremunerative.

"Ghazipur was once renowned all over the world for its rose products, but now the industry has practically died out, and much of the so-called Ghazipur rose water really comes from Hathras, where roses can be grown more cheaply.

"(1) A series of experiments was carried out on the ordinary native still (called 'deg'), provided with an outlet pipe made of bamboo, and connected to a receiver, which is kept immersed in water. The experiments confirmed the results obtained last year, and a yield of 0.004 to 0.0045 per cent. of otto on the weight of fresh flowers was obtained.

"In all these experiments, freshly-plucked flowers, free from moisture, were used, and the conditions as regards quantity of flowers used, time of distillation, water used in the

still, were kept identical. The same distillation (*sic*) was used three times over, the quantity of flowers being the same in each case.

"(2) A second series of experiments was carried out, using the same apparatus as above and keeping all factors identical, with the improvement that the water used to cool the receiver was kept running. This brought up the yield to 0.005 per cent. This shows that the native practice of not changing the cooling water is responsible for some loss of the otto. Strange as it may appear, native distillers do not care to change the water, because they have an idea that cold water impairs the aroma of the otto.

"(3) An improved direct-fired still (cap. 20 gallons), devised and constructed by the Indian Essential Oils Company, provided with a worm condenser, was next tried. Several experiments were carried out, and the yield was found to have risen very greatly. All other conditions being the same, the yield was found to be 0.015 per cent. on the weight of the flowers.

"(4) Experiments carried out at Cawnpore on steam still using 10 lbs. steam pressure, gave a yield of 0.016 per cent."

Experiments on improved still (3 above) further showed that if the receiver was protected from hot winds and kept cool, using 100 lbs. of water and 25 lbs. of fresh flowers plucked before sunrise, the best yield was obtained, namely, 0.025 per cent. Hope is expressed that with a little further care the yield can be improved to even 0.03 per cent., which is the Bulgarian figure. Further, by improving the methods of cultivation it may be possible to increase the yield of flowers per acre as well as the oil content of the flowers (Srivastava & Sinha, *loc. cit.*).

The above experiments were repeated by Gadre and Mukherji (*loc. cit.*) and they state as under:

"The flowers used for distillation were those of *R. damascena*, the species employed in Bulgaria, and were of the red or pink variety exclusively, which is the commonest Indian garden rose. The flowers from Hathras were packed in tins with common salt and sent by rail to Cawnpore; they were distilled by the Indian Essential Oils Co., and the distillates were forwarded to the Government Technical Laboratory at Cawnpore for examination. The flowers grown around Cawnpore were distilled at the Government Technical Laboratory in a steam still and a direct-fired still which was

a modernised improvement of the Deg still. The flowers were collected early in the morning and distilled as soon as possible after collection. Distillation usually occupied from 3 to 5 hours and the weight of the distillate collected was the same as the weight of the flowers used. Double and triple distillations were also tried, the once distilled and double-distilled waters being mixed with more flowers and distilled from a copper still. In preparing these a layer of paraffin oil was placed in the receiver to minimise the escape of essential oil owing to the prevailing temperature and high winds. These layers of paraffin oil were afterwards collected and the amount of rose oil computed by determining the iodine-absorption figure of the paraffin oil. The oil content of the rose-water was similarly evaluated. Determined in this manner the average yield of otto obtained by distilling Cawnpore flowers amounted to 0.01 per cent. on the weight of the flowers.

"It was found that the iodine value determined by Wij's iodine solution gave satisfactory and concordant results. Calculations were based on 190 as the iodine-value for pure otto of rose, this figure being the mean values determined by Hudson-Cox and Simmons on various samples (Analyst, 1904, 29, 175) and agreeing with figures obtained by the authors for Bulgarian otto and a sample of pure otto made from Hathras roses.

"It was concluded from these investigations that rose flowers grown at various localities in the Aligarh District are superior for distillation to those available at Cawnpore or Ghazipur, the superiority being due to richer soil and better irrigation. By means of a preservative like common salt, the flowers can be stocked undamaged for 3 or 4 days after plucking. The high percentage yield of otto by the Indian Essential Oils Co. could not be substantiated by the authors; the average calculated yield of otto did not exceed 0.015 per cent., and the optimum yield was found to be much lower than that usual in Bulgaria and elsewhere. Determination of the iodine-absorption of rose-water in the manner described was found to be a ready and reliable method of evaluating the rose-water.

"The following types of stills were tried in the experiment:—The Deg type; the Bulgarian still; the steam-still in which steam is blown through the flowers; and an improved direct-fired still. All three stills gave practically the same results with a given variety of flowers. Live steam is

unsuited for making otto from rose-flowers. A steam-jacketed still would be ideal, but its use may not be practicable on rose fields. The fourth type of still was a modification of the Deg still with a separate top, containing a fairly wide leading tube which could be connected with a worm condenser. It is portable and easily worked."

In actual practice hardly any pure rose otto is manufactured in India. The so-called attar or "itr" of rose is a mixture obtained by the distillation of sandalwood oil and rose oil.

*Preparation of rose otto in other countries:* Otto of rose is also prepared in many other countries including Persia, Turkey, U.S.S.R., Spain, and N. Africa.

*Production of rose otto and its trade in important countries:* From 1927 to 1937 the area under rose cultivation in Bulgaria varied from 14,000 to 18,000 acres and the otto produced ranged from 4,000 lbs. to 7,000 lbs.

The total world production of rose oil during 1935-37 is said to be in the neighbourhood of 9,400 lbs. per annum.

According to Schimmel & Co. (1945. *Ann. Rep.* : p. 50), the area devoted to the cultivation of roses in Bulgaria decreased from 15,600 acres in 1939 to 5,700 acres in 1945. The yield of flowers was also below normal, because of neglect of plants, lack of fertilizers, and the planting of food crops among the rose plants. Only 770 lbs. of rose oil were produced in 1945 and no rose concrete was manufactured in that year or in 1944. Schimmel & Co. (*loc. cit.*), quoting from an article in the *Foreign Commerce Weekly*, 1946, 23(7), p. 41, give the following figures of production of rose oil and rose concrete in Bulgaria from 1938 to 1945:—

|      | ROSE OIL<br>lbs. | ROSE CONCRETE<br>lbs. |
|------|------------------|-----------------------|
| 1938 | 3,785            | 5,090                 |
| 1939 | 8,310            | 190                   |
| 1940 | 4,065            | 4,630                 |
| 1941 | 1,840            | 2,650                 |
| 1942 | 595              | 2,790                 |
| 1943 | 2,490            | 4,435                 |
| 1944 | 1,615            | ...                   |
| 1945 | 770              | ...                   |

In 1945 Bulgaria exported 1,408 lbs. of rose oil valued at \$753,300 to the U.S.A. and 284 lbs. to France. The U.S.A. did not import rose oil from Bulgaria in 1944, but France obtained 282 lbs. of the oil and 167 lbs. of rose concrete.

The crop of rose harvested in the Grasse area of France in 1945 amounted to only

858,000 lbs. as compared with 981,200 lbs. in 1944. Exports of rose oil from France in 1945 totalled 1,170 lbs. valued at Frs. 14,252,000.

Imports of rose oil from Morocco into France during 1945 totalled 1,980 lbs.

Imports of rose oil into the U.S.A. during 1945 included, in addition to the Bulgarian otto of rose (see above), 420 lbs. of rose oil from Turkey, 440 lbs. from the U.S.S.R. and 287 lbs. from France.

*Rose odours:* According to Parry, Blondel has grouped the rose odours as follows: rose, musk, mignonette, violets, lily of the valley, hyacinth, fruit, bugs, and clove.

According to Poucher, "Slight differentiations in odour value are not perceptible to every one. The sense of taste is, however, generally more developed, and inconsiderable modifications of flavour are more frequently noticed. To the trained specialist, however, the merest graduation of odour is appreciable, and an expert florist will name the variety of rose *even in the dark*. The real rose odour is unique, and represents a *type* which is undefinable, incomparable, and at present inimitable by synthesis. This type is best represented by *Rosa Damascena*, cultivated in Bulgaria, and approximated to very closely by *Rosa centifolia*, cultivated in Provence. There are many variations from this type, and some of the roses which exhibit slightly different floral notes are enumerated below; they represent practically all those that the perfumer is required to imitate:—

"*R. arvensis*, also known as the Ayrshire rose—some varieties are myrrh-scented; *Banksian*, recalling violets; *Canina*, resembling mignonette; *Desprez*, fruity; *Eglantine*, whose leaves recall jasmin; *Macartnean*, apricots; *Marechal Niel*, the most delightful of the 'tea' class, having a somewhat fruity odour, resembling raspberry; *Moschata*, growing wild in Tunis and believed by some to be musky and by others to resemble pinks; *Muscosa*, moss-scented; *May*, recalling cinnamon; *Safrano*, recalling pinks; *Socrates*, resembling the peach; *Souveraine*, the melon; *Unique jaune*, the most charming of the 'noisette' type and having an odour of hyacinths. During 1929 a new rose, known as 'Portadown Fragrance', made its appearance. It is alleged to have taken eleven years to 'produce' and is the result of ingenious crossing by Samuel McGreedy & Sons. According to report the odour is a complex of tea rose and verben. In the

majority of cases red roses are more odiferous than white ones, but a peculiarity possessed by a number of both kinds is that, when cut and placed in water, their fragrance appears more pronounced than when growing. Roses cultivated in a hot climate have a more powerful perfume, and it has been noticed that those flowers grown under glass develop a finer aroma than those which thrive in the open air. Some roses, e.g. *R. gallica* develop their perfume when dried, while others, e.g. *R. Damascena*, lose it under similar circumstances. Sawyer states<sup>1</sup> that before a storm the odour of a rose seems strangely increased, and suggests that this may be due to the oxidising influence of the ozone in the atmosphere or to the perceptive faculties being sharpened at such moments. Other peculiarities of the rose odour are (a) that no two flowers emit the same fragrance; (b) that different flowers from the same plant have never exactly the same perfume."

*Rose perfume*: The rose perfume is marketed in several forms, e.g., otto or attar, concrete and absolute, rose water and oil of rose waters, and pomade. The best and most powerful form in which the perfume can be obtained is undoubtedly the otto. The otto, however, does not accurately represent the odour of the flower, since, during the process of distillation, a large proportion of phenylethyl alcohol is lost. Phenylethyl alcohol is a constituent of the rose oil and is highly soluble in water. During distillation of the otto it remains dissolved in water, and thus disappears to a very large extent from the otto itself. This fact accounts for the difference in odour, for example, between otto of rose and rose concrete or rose water. The odour of the rose absolute, which is prepared by using volatile solvents, is more nearly approximate to that of the flowers. The rose perfume may also be extracted in the form of pomade by maceration, and, in a few instances where rose water is prepared in Grasse, the oil is separated which contains the most highly odorous constituents and all the stearoptene. This is the costliest otto obtainable (Poucher).

One lb. of rose oil is obtained by distilling 3,500 lbs. of flowers in Bulgaria (Poucher).

*Characteristics of otto of rose*: The characteristics of otto of rose vary widely according to the locality in which the plants are cultivated, and also in one locality from season to

season, mode of distillation, and other conditions. All these factors have to be taken into consideration, and, therefore, the published data should not be regarded as final or complete. The odour of the otto also is subject to considerable variation. According to Poucher, there are two well-defined types, possessing: (1) the soft honeyed sweetness of the peasant oil, and (2) the intense sharpness of the essence (those distilled from the large fire stills). These two comprise the main part of the oils distilled, but in addition there are those which have (3) the aldehydic tone of the steam-distilled oils, and (4) the leafy heaviness of the product from vacuum stills.

Poucher states that in the first two types there are further variations depending upon (a) the ratio of different types of roses, (b) the time and care taken between plucking and distillation, (c) the particular locality from which the distiller obtains his flowers in the preparation of his own special bouquet, (d) the possibility in large fire stills of slight burning of the flowers, due to the contact with the walls, and (e) the type of the fire still, some preferring the detachable and others the fixed head. In order to obtain otto of reasonable uniformity, some of the larger distillers, therefore, prefer to blend oils obtained from different districts.

*Physical and chemical data for otto of rose*: The Bulgarian rose otto, generally speaking, is light yellow in colour, occasionally with a greenish tinge. It has the consistency of oil of almonds at 21° to 25°C. In taste it is pungent and balsamic and in odour strong. At about 18° to 21°C., acicular or lamellar crystals separate which are shiny and ir-radiant. On account of their low specific gravity they collect at the surface forming a thin film which breaks up readily when shaken. When cooled to a lower temperature the oil congeals to a transparent, soft mass which again liquefies with rise of temperature.

Higher stearoptene content tends to lower the specific gravity of the oil. On account of the difficultly soluble paraffins which it contains, oil of rose yields turbid solutions, even with very large amounts of 90 per cent. alcohol.

According to Simmons (1925. *Perfum. essent. Oil Rec.*, 16: 341), the following figures will be found to cover most oils of good quality; figures given by Poucher are also recorded for the purpose of comparison:—

1. "Odorographia" (1892), 24.

|                                 | SIMMONS        | POUCHER        |
|---------------------------------|----------------|----------------|
| Specific gravity <sup>20°</sup> | 0.850 to 0.860 | 0.849 to 0.865 |
| Optical rotation                | -1.3° to -4°   | -1° to -5°     |
| Refractive index <sup>20°</sup> | 1.458 to 1.465 | 1.452 to 1.466 |
| Melting point                   | 19° to 24.5°   | 15° to 24°     |
| Saponification value            | 7 to 18        | ...            |
| Total alcohols, as geraniol     | 70 to 75%      | 63 to 84%      |
| Citronellol                     | 30 to 40%      | 24 to 64%      |
| Stearoptene                     | 14 to 20%      | 7 to 25%       |
| Melting point of stearoptene    | about 32°      | ...            |
| Acid val.                       | ...            | 1.5 to 3.8     |
| Ester val.                      | ...            | 3.7 to 17.5    |

According to Simmons (*loc. cit.*), the most important constituents of the rose otto are geraniol (40 to 50 per cent.), rhodinol—citronellol (30 to 40 per cent.), nerol, phenylethyl alcohol, together with small amounts of esters, linalool, citral, nonylic aldehyde, eugenol, a sesquiterpene alcohol, and stearoptene.

Poucher records the following constituents in commercial samples of the rose otto: *l*-citronellol (24 to 64 per cent.), total alcohols as geraniol (63 to 84 per cent.), nerol (up to 10 per cent.), phenylethyl alcohol (up to 1 per cent.—more in oils by vacuum distillation), esters of geraniol (up to 3.5 per cent.), eugenol (up to 1 per cent.), stearoptene (7 to 25 per cent.), and traces of *l*-linalool, farnesol, citral, and nonylic aldehyde.

The characteristic honey odour has been attributed to traces of phenylacetic acid produced by oxidation of the phenylethyl alcohol. The proportion of these constituents varies in oils obtained in different countries. The presence of ethyl alcohol is reported in some cases, but it is ascribed to the decomposition of flowers, especially when the roses get heated and begin to ferment during transportation to the stills. If the petals were distilled as soon as they are picked, the oil contains no ethyl alcohol (Simmons, *loc. cit.*).

It must, however, be mentioned that the chemical analysis of an otto of rose can never be taken as a final criterion of purity, but should always be supplemented by a critical evaluation of its odour by an expert.

*Artificial otto of rose*: While no synthetic preparation has yet been able to displace the natural otto of rose, efforts to manufacture a real artificial otto have been unremitting. For this purpose combinations of the following have been employed: geraniol, linalool, the higher homologues of phenylethyl alcohol, citronellol, the various esters of these alcohols and of phenylethyl alcohol, phenylacetic acid and its esters, and substituted citronellols. A near approach to the desired rose smell is

obtained by suitable combination of these, but all such preparations lack in the freshness which comes only out of live flowers. In order to impart this freshness a little of the natural otto is always added to the artificial otto, and in good oils a generous proportion. Without the natural otto, the synthetic oil is of little value to the perfumers.

*Standards and examination of rose oil*: Rose oil with its high price has always been a profitable article for all adulterators. In this connection it may be mentioned that, in Bulgaria, the amount of otto exported in many years exceeded the amount actually produced. This is explained by the fact that adulteration was practised. In point of fact, at one time adulteration was the rule and pure otto of rose a rare exception. The universal adulterant employed in Bulgaria was the palmarosa oil. Pure geraniol and similar compounds are also used, and "artificial mixtures" are today available which are marketed solely for the purpose of encouraging adulteration. Phenylacetic acid, which is one of the constituents of the natural oil, is commonly used to impart to the adulterated product the honey odour, which is characteristic of the natural otto. Other adulterants employed are guaiacum wood oil, gurjun balsam, benzyl benzoates, nonylic and decylic aldehydes, etc. According to Schimmel & Co. (1938. *Ann. Rep.*: p. 91), adulteration by ethyl alcohol has been greatly on the increase during recent years, and that geraniol, geraniol fractions, and also citronellol are favourite adulterants. They also report a crude case of adulteration wherein copious amounts of easily detectable terpineol had been added.

Since 1935, the State Agricultural Bank of Bulgaria holds a monopoly in that country for superintending the manufacture and marketing of pure rose oil, and it may be hoped that it will be possible to obtain rose oil of pure quality. Nevertheless, rose oil is, and remains, an article that must be taken on trust, and ought only to be purchased from dealers of first-class reputation. With so costly a product as rose oil the danger of adulteration is very great.

As mentioned earlier the characteristics of rose oil vary a great deal, and it is difficult to draw up standard figures, even within very wide limits, to cover every genuine otto of rose. The bulking of oils from different districts, usually carried out

by the dealers in Bulgaria, eliminates most oils with very abnormal properties, and tends to something more approaching uniformity. Although no limits can be definitely laid down for pure otto of rose (even excluding French otto and those oils produced in steam vacuum stills), the characteristics of the commercial oils given earlier would be found to cover most oils of good quality. The effect on these figures of the various adulterants which are likely to be met with have been dealt with by Simmons (1925. *Perfum. essent. Oil Rec.*, 16: 341). He states as under:—

“SPECIFIC GRAVITY—The specific gravity of otto of rose is low, and is raised by practically all adulterants, except ethyl alcohol and added wax. . .

“OPTICAL ROTATION—This figure has been useful in the past in detecting the presence of gurjun balsam, which considerably raises the laevo-rotation, one sample examined by the writer showing as high a rotation as  $-19$  deg. The presence of the balsam can be readily confirmed by Dodge and Olcott's test, adding to 5 drops of the otto 10 cc. of glacial acetic acid and 3 drops of pure nitric acid, and well shaking, when with as little as 0.5 per cent. of gurjun balsam oil a reddish violet colour develops in about 30 secs. The introduction of gurjun balsam oil into otto appears to owe its origin to the adulteration of the otto with a rectified palmarosa oil, which had in turn been adulterated with gurjun balsam oil.

“REFRACTIVE INDEX—This figure, like the specific gravity, is raised by most adulterants except ethyl alcohol and added wax, which reduce it. Addition of ethyl alcohol is very easily detected by determining the refractive index before and after washing with slightly warm water, when an increase of .0015 after washing may be taken as equivalent to about 1 per cent. alcohol (vide ‘P. & E.O.R.’, 1915, p. 74). With genuine otto containing an appreciable amount of phenylethyl alcohol, the refractive index is very often slightly reduced by washing due to the extraction of this substance. . .

“MELTING POINT—The congealing point, which was formerly usually determined in the examination of an otto, seems now quite generally to have given place to the determination of the melting point. These points are usually defined respectively as the temperature at which the first crystal appears as the liquefied oil cools, and the

temperature at which the last crystal disappears as the solidified otto melts. For genuine oils the melting point rises with increasing proportion of stearoptene, but is reduced by the addition of geraniol, palmarosa, or geranium oils, and is raised by the presence of ethyl alcohol, since the melting point is really the temperature at which the other constituents, sometimes termed the ‘eleoptene’, dissolve the stearoptene, and the solubility of the wax is much reduced by the addition of alcohol. The solidifying point has been much more correctly termed by Siedler, the ‘crystallisation point’ (Schimmel, ‘Semi-Annual Report’, April, 1913, p. 90). The effect on the melting point of geraniol or similar adulterant is sometimes counterbalanced by the addition of stearoptene, spermaceti, or other wax. Provided the amount of stearoptene is not excessive, the presence of added stearoptene is difficult to detect, but the addition of spermaceti raises the saponification value of the otto, and is also readily revealed by the melting point and saponification value of the separated wax and the melting point of the fatty acids obtained after saponification. A sample of such wax separated from otto examined by the writer gave melting point 49 deg. C. and saponification value 118.7.

“SAPONIFICATION VALUE—The proportion of esters in genuine otto of rose is extremely small, and the determination of the saponification value therefore readily detects the presence of any saponifiable adulterant, such as spermaceti (vide supra), diethyl phthalate (found by Schimmel, ‘Semi-Annual Report’, April, 1913), or oils containing much ester, e.g., geranium oils.

“TOTAL ALCOHOLS—The determination of the total alcohols by acetylation serves to reveal the presence of added geraniol, palmarosa or similar adulterant, as with a genuine otto this figure rarely exceeds 75 per cent.

“CITRONELLOL—Umney has shown (‘P. & E.O.R.’, 1913, p. 328) that the determination of the percentage of citronellol by formylation gives distinctly useful information, this figure being depressed by adulteration with geraniol or palmarosa oil.

“STEAROPTENE—The significance of the proportion of this has been already pointed out. It is best determined by Burgess's process which consists in extracting 5 grms. otto with 25 cc. 85 per cent. alcohol, cooling



in ice for 5-6 hours, filtering off the stearoptene, washing once with cold 85 per cent. alcohol, and drying in vacuo over sulphuric acid for 12 hours.

"In addition to those mentioned above, other adulterants which have been noted in otto of rose include guaiac wood oil, and nonylic or decylic aldehyde (Parry, 'Chem. & Drug.', 1910, 531), though as nonylic aldehyde is a natural constituent of otto, adulteration with this substance might be difficult to prove.

"The chemical analysis of an otto of rose can never be taken as a final criterion of purity, but should always be supplemented by critical examination of its odour by an expert. How fallacious a chemical analysis only may be is well shown by the following figures, which were obtained for a mixture, prepared by the writer, of 85 parts genuine otto and 15 parts palmarosa oil, and all of which fall within the above-mentioned limits for a genuine otto of rose :

|                             |          |
|-----------------------------|----------|
| " Specific gravity at 30°C. | 0.8554   |
| Optical rotation ..         | -1°30'   |
| Refractive index at 25°C.   | 1.4592   |
| Saponification value ..     | 11.2     |
| Melting point ..            | 21.3°C." |

According to Naves (1948. *Mfg. Chem.*, 19 (8) : 371), for many years a rhodinol (citronellol) content of between 25 to 40 per cent. was accepted as a standard for a genuine oil, and oils with higher content were rejected as adulterated. Subsequent work has shown that the reverse was, in fact, true and that a low rhodinol content could be an indication of adulteration. Naves (*loc. cit.*) advocates a rhodinol content of not less than 34 per cent. as the criterion of a genuine Bulgarian rose oil. It has, however, been shown that the content of rhodinol varies between 23 to 40 per cent. in the products of the old type of apparatus, and between 45 to 55 per cent. by the modern apparatus; a value up to 63 per cent. might be obtained for the products of rotating apparatus.

Several authors have stressed the point that the ratio of rhodinol to geraniol falls during distillation. This accounts for the low figures for oil produced in "peasant" apparatus when the operation is pushed to the limit in order to obtain high yields at the sacrifice of quality of the essential oil. It is, therefore, correct to state that

the increase in the rhodinol content has been mainly due to improved distillation methods (Naves, *loc. cit.*).

According to Naves (*loc. cit.*), the products of rose are characterized by the presence of laevorotatory rhodinol, the rotatory power of which in sodium light is 4° to 4.50°. It is not possible to obtain such a product economically from another source. The rotatory power of rhodinol from geranium oil is never more than 2.5°, and the product from other sources is either racemic or dextrorotatory. It has, therefore, been recommended that rhodinol be isolated and its rotatory power checked by measurements of other constants, such as density, refractive index, etc. Isolation is effected as follows :—

The oil is heated at 140° to 160°C. in presence of an excess of benzoyl chloride, when all the alcoholic constituents are destroyed with the exception of the rhodinol and a part of the phenylethyl alcohol, which get converted into the benzoic esters. The low-boiling products are eliminated by distillation and the residue saponified. The alcohols are rectified by entrainment with steam, the phenylethyl alcohol is destroyed by heating at 140° over potash, and rhodinol isolated by fractional distillation (Naves & Glichitch, 1933. *Parfums de Fr.*, 11 : 156).

Addition of rhodinol from any other source than rose oil depresses the rotatory power (Naves, *loc. cit.*).

#### *Rose concrete*

The rose concrete is extracted with petroleum ether or benzene. According to Naves & Mazuyer (1947. *Natural Perfume Materials* : p. 234), the yield of petroleum ether concrete in Provence varies between 0.17 and 0.27 per cent. (usually between 0.24 to 0.265 per cent.). This concrete gives 55 to 65 per cent. of an absolute. They also state that in Bulgaria the yield of concrete by the same process is 0.22 to 0.25 per cent., and that this gives 50 to 60 per cent. of absolute. The concrete extracted with petroleum ether ranges in colour from light brown to dark brown, while that obtained with benzene varies from light green to greenish brown; the latter method gives a slightly higher yield. It is also stated that the roses cultivated at higher altitudes yield more concrete.

The petroleum ether concrete from May rose in Provence has the following characteristics: congealing point  $43^{\circ}$  to  $48^{\circ}$ , methoxy val. (Ziesel)  $49^{\circ}$  to  $54^{\circ}$ , acid val. 9.8 to 14.4, and ester val. 19.6 to 25.6. The Bulgarian concrete has the following characteristics: congealing point  $41^{\circ}$  to  $46.5^{\circ}$  and saponification val. 31 to 56. The commercial products are said to frequently contain a considerable amount of petroleum ether. The presence of petroleum ether depresses the congealing point (Naves & Mazuyer, *loc. cit.*).

The results of determinations of different samples, as given by Naves & Mazuyer (*loc. cit.*), show that absolutes from the Bulgarian rose have the following characteristics: sp. gr.<sup>15°</sup> 0.9682 to 0.9916,  $[\alpha]$   $+9.4^{\circ}$  to  $+14.3^{\circ}$ ,  $n^{20}$  1.50633 to 1.51556, acid val. 3.5 to 11.2, ester val. 19.6 to 30.9, and total alcohols 69.3 to 71.8. The absolutes are generally brownish green, and are limpid and syrupy. They have a remarkable dextrorotatory power, whereas the essential oils and the distillates are laevorotatory.

The distillates from the concretes and absolutes are colourless and limpid. The results of determinations of different samples, as given by Naves & Mazuyer (*loc. cit.*), show that the distillates from the Bulgarian rose have the following characteristics: sp. gr.<sup>15°</sup> 0.951 to 0.992,  $[\alpha]$   $-0.51^{\circ}$  to  $-2.4^{\circ}$ ,  $n^{20}$  1.5046 to 1.5190, acid val. 2.1 to 5.1, and ester val. 5.6 to 10.4 (Naves & Mazuyer, *loc. cit.*).

#### Rose water

In some countries, especially in India, the rose is distilled not for its otto but for the production of rose water. During distillation, the distilled perfumed water is not returned to the still, as is the case when the manufacture of otto is aimed at, but sold as rose water. Rose waters of different strengths are prepared. If, for example, 100 lbs. of rose petals are distilled to yield 100 lbs. of rose water, the first 50 lbs. distilling over is labelled "quadruple" and the next 50 lbs. "triple".

On the basis of his experiments performed in France, Poucher states that it requires 12,000 kilos of flowers to produce 9,000 litres of rose water, plus 1 kilo of direct oil.

The rose water can be evaluated "by repeatedly shaking with chloroform and

determining the amount of iodine absorbed by the chloroform extracts with Wij's solution, the time of interaction always being an hour, a period sufficient for complete absorption. Five successive extractions with chloroform were found completely to exhaust the rose-water of its oil. Once-distilled water made in the laboratory gave figures such as 0.0574, 0.0688, 0.0605, 0.0434 grams iodine absorbed by 450 c.c. rose-water, and thrice-distilled water gave 0.2156 gm. Thus a classification of samples of rose-water can be made by the iodine-absorption method" (Gadre & Mukerji, 1922. *J. Indian Industr., vide J. Soc. chem. Industr., Lond., 1922, 41: 192R*).

The districts of Ghazipur, Hathras, and Aligarh are the three main centres of the rose industry in Upper India, where considerable quantities of rose water are manufactured.

#### Oil of rose waters

In France, part of the large amount of rose water manufactured in Grasse is extracted with volatile solvents. The product obtained is the oil of rose water, which has different uses in perfumery than the essential oil. The oil of rose water can also be used for the reconstitution of rose water, and has the advantages of an economy of storage space, packing, transportation, and an assurance of better conservation. According to Naves & Mazuyer (*loc. cit.*), the yield by petroleum ether extraction is from 0.200 to 0.280 per cent. for the so-called "single" water manufactured at Grasse. This product consists mainly of phenylethyl alcohol.

### 3. *Rosa moschata* Herrm.

*R. moschata* Mill., Fl. Brit. Ind., II, 367.

(Musk Rose, *Kuja*)

This is a large, thorny, climbing shrub with terminal compound corymbs of very sweet-scented, white flowers 1 to  $1\frac{1}{2}$  in. in diameter. It is found in the W. Himalayas at altitudes of 3,000 to 9,000 ft., but is more common between 4,000 to 6,000 ft.

According to Gupta (1928. *Kanjilal's Forest Flora of Chakrata, Dehra Dun, etc.:* p. 222), "An excellent otto is extracted from the flowers at Nahan in Sirmoor". The shrub flowers in May and June.

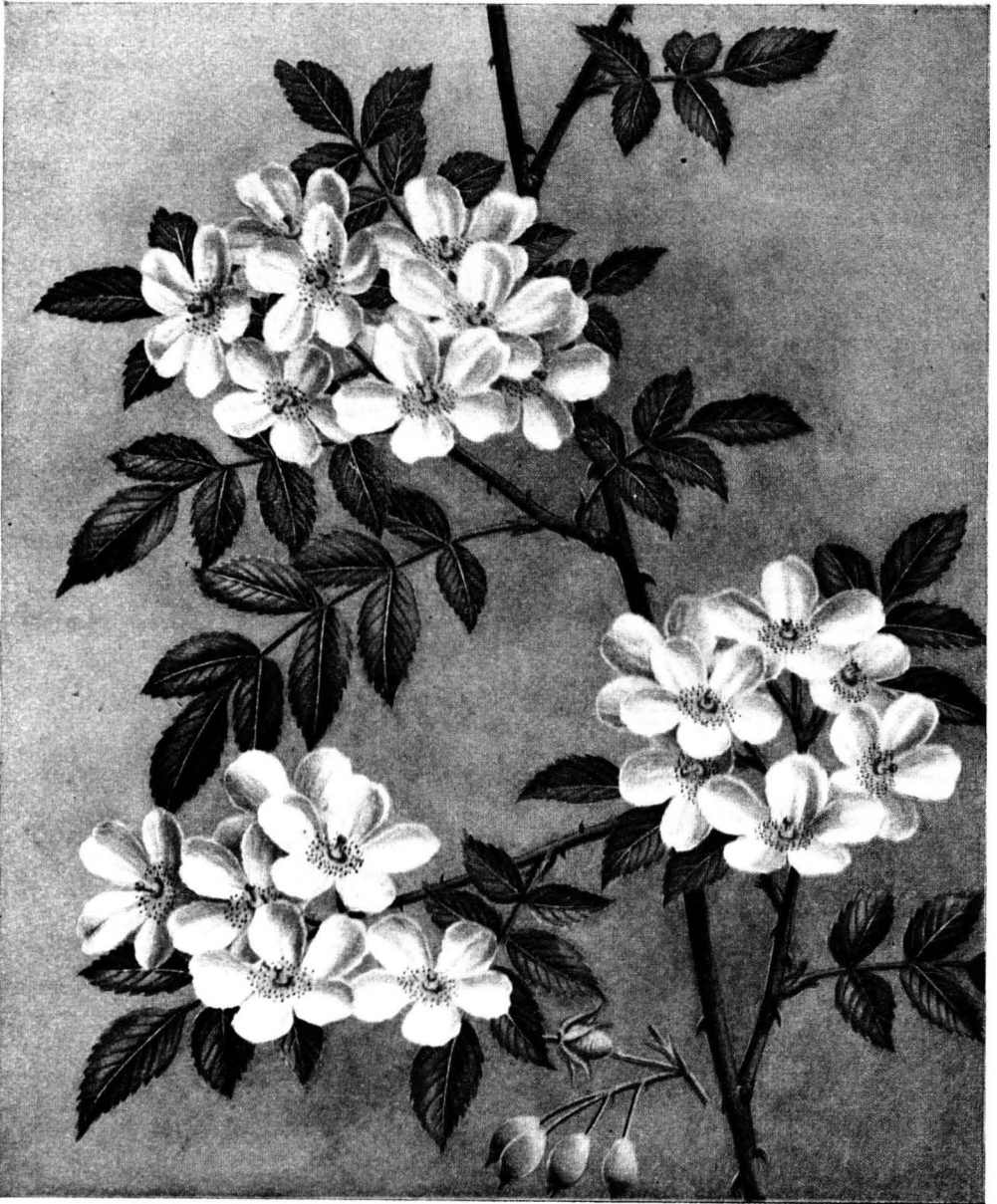


FIG. 41 — *Rosa moschata* Herrm.

#### 6. RUBUS (Tourn.) Linn.

(The Latin name for the bramble, derived from *ruber* — red; referring to the colour of the fruit in some species.)

This is a large genus of often prickly shrubs, including the brambles, the black-

berry, raspberry, etc. Its members have 3- to 7-foliolate or simple lobed leaves, white or pink flowers with a persistent calyx bearing the numerous stamens, and a mass of carpels ripening into a multiple fruit composed of many drupelets. This genus

is abundant in N. Hemisphere but rare in S. Hemisphere. Over 40 species grow wild in India, and some of them yield edible fruits.

The true raspberry of Europe is *R. idaeus* Linn. The fruit pulp, after extraction of the juice, yields on distillation a very small amount of a greenish oil of strong odour. The oil has sp. gr. 0.883,  $[\alpha] +2.8^\circ$ , saponification val. 193, the same after acetylation 215, and is soluble in 30 parts of 80 per cent. alcohol (Haensel, 1904. *Apothekerztg*, *Berl.*, 19: 854).

There are a number of species of *Rubus* growing in India which possess the odour and flavour of true raspberry, but it is not known if any of them yields an essential oil.

### 7. SPIRAEA Linn.\*

(From the Greek *speiraia*, from *speira* — a spire, something twisted; in allusion to the flexible branches.)

This is a large genus of unarmed shrubs or perennial herbs, with small perfect white

\* The genus as defined here is nowadays usually divided into a number of genera, the herbaceous members falling under *Ulmavia* and *Aruncus*, and the shrubby species under *Spiraea*, *Sorbaria*, etc.

or pink flowers in dense racemes, corymbs, cymes or panicles. Its members are found in the temperate and cold regions of N. Hemisphere, and about a dozen species are represented in India.

Members of this genus are hardly of any economic importance. Some of the Indian species, such as *S. bella* Sims, are ornamental and are cultivated in gardens. The Chinese *S. cantoniensis* Lour. is also occasionally cultivated. Both are shrubs; the former with pink, rarely white flowers, and the latter with white flowers.

The different parts of some species are stated to yield hydrocyanic acid. Thus, for example, the leaves, twigs and roots of *S. aruncus* Linn.\* of the temperate Western and Central Himalayas and flowers and leaves of *S. sorbifolia* Linn. of the temperate Western Himalayas are stated to yield hydrocyanic acid (Wehmer).

Salicylaldehyde has been reported from the leaves and flowers of some foreign species of this genus.

\* Some authors prefer to call it *Aruncus sylvester* Kostel.

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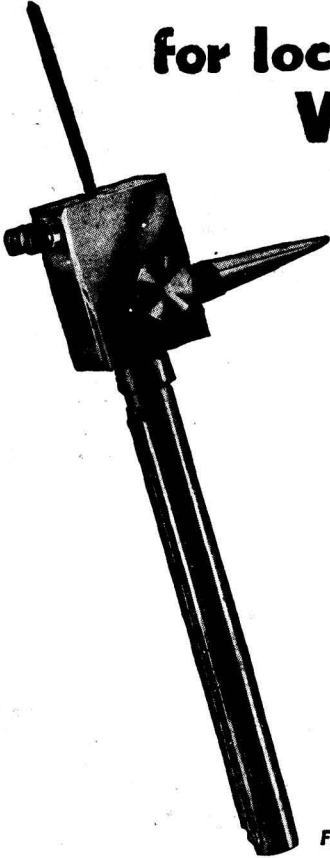
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