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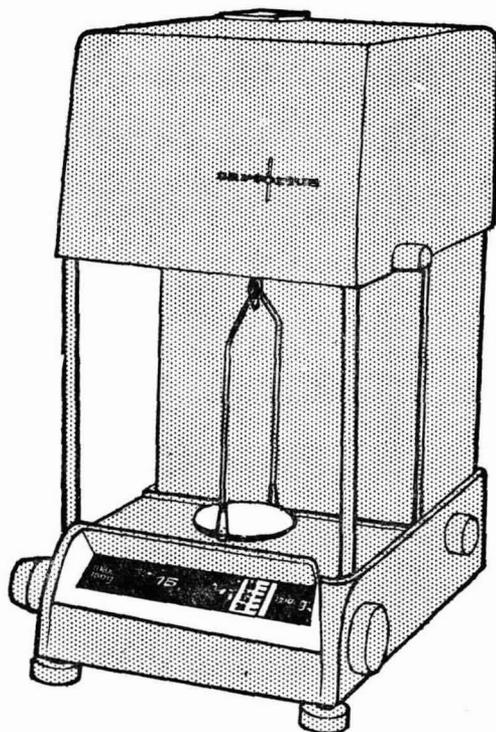
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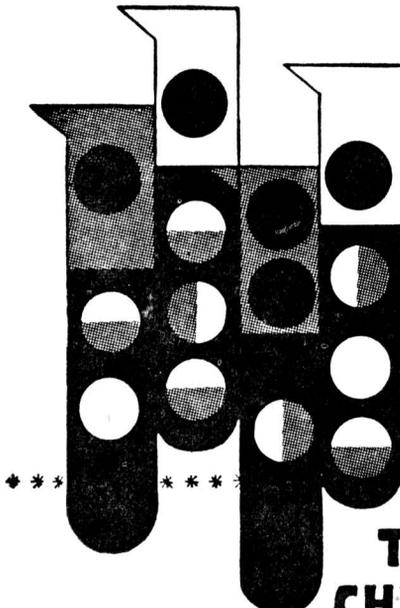
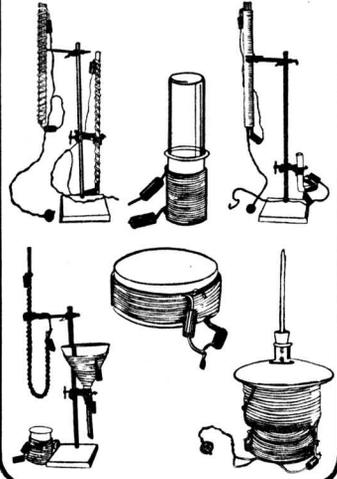
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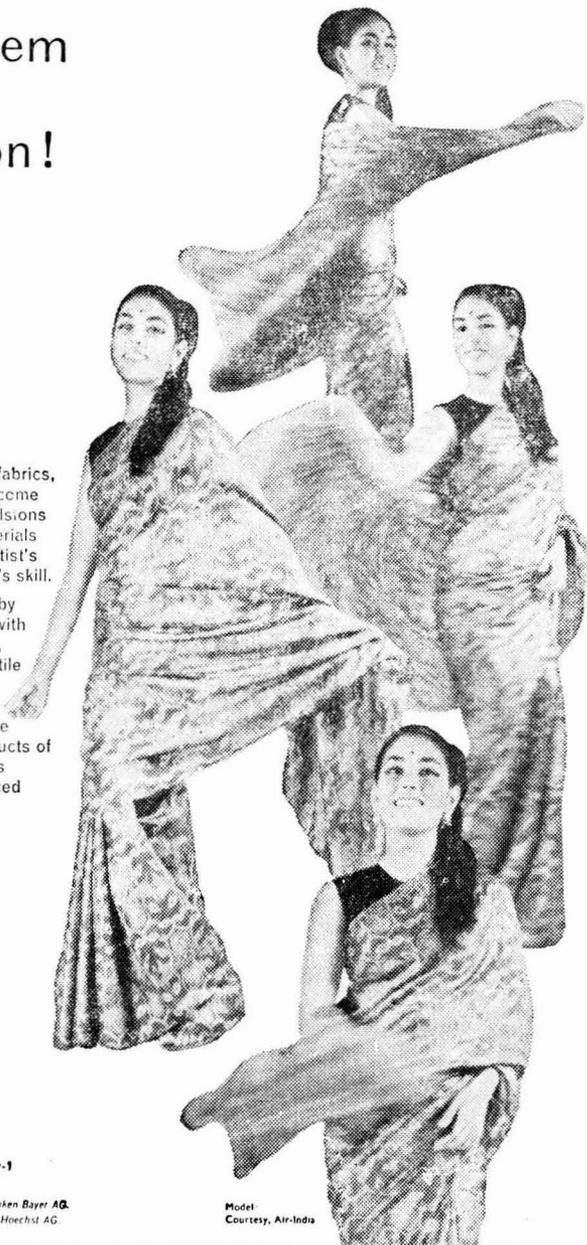
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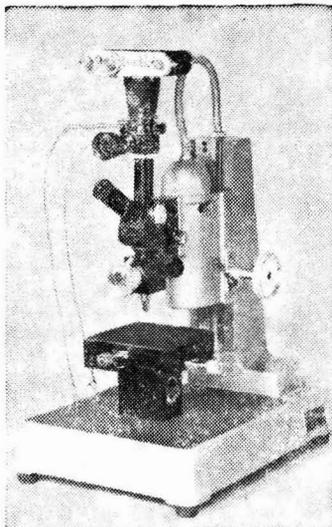
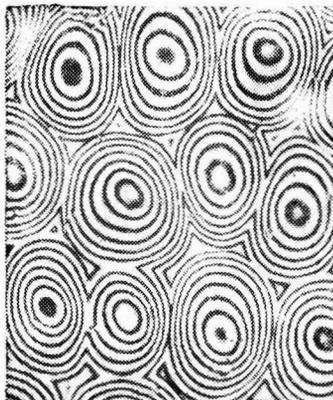
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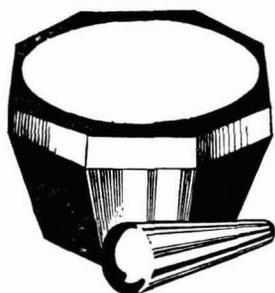
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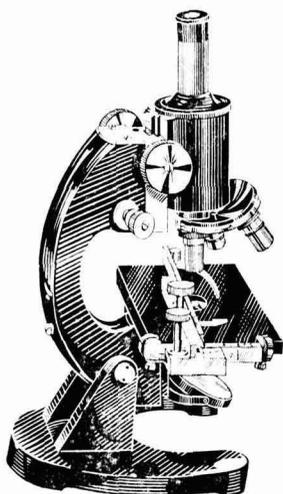
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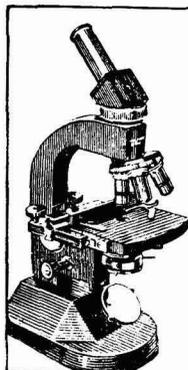
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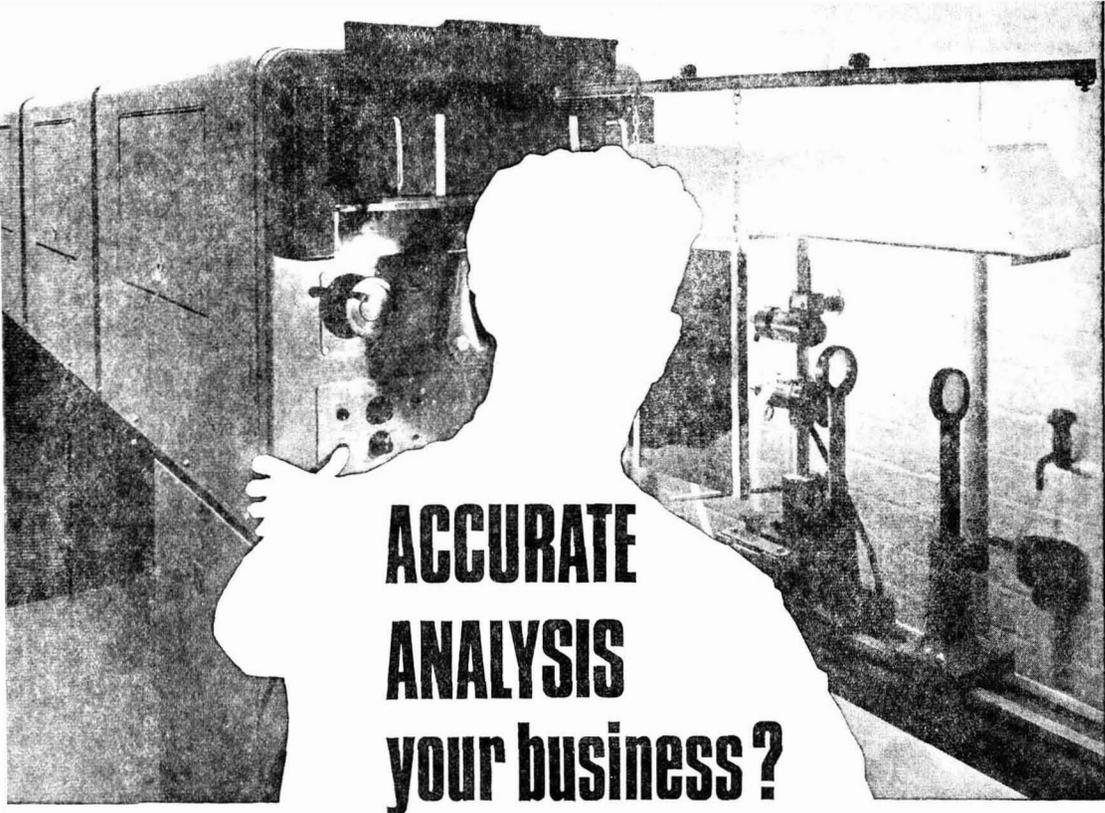
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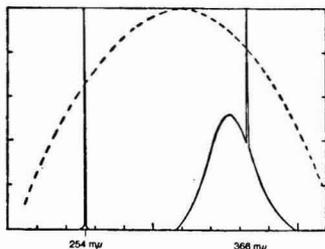
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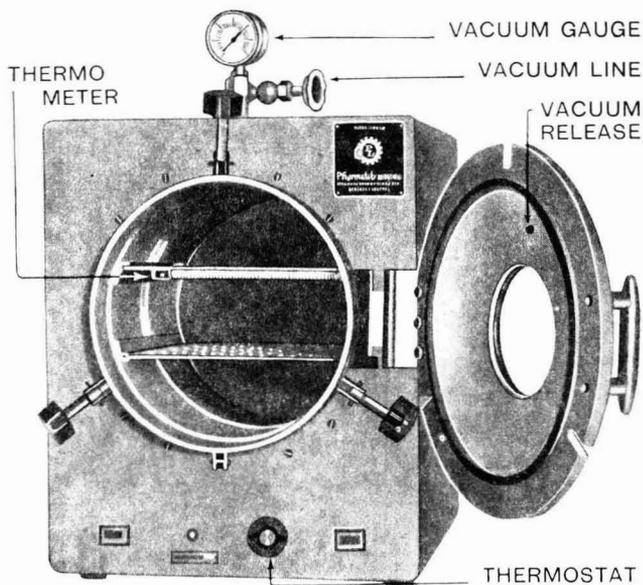
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Plant Sciences in the Service of Man*

IN the world of today, it is not necessary to dwell upon the importance of science in the life of a nation. Even the most primitive societies have started thinking about improving science education and the training of scientists. The very survival of developing countries today depends on adequate acquisition of scientific and technical knowledge and its full utilization in agriculture, industry, defence and other spheres. This has been brought out very clearly in the Science Policy Resolution of the Government of India adopted in 1958.

Around the beginning of the 17th century, when science was just entering upon its modern phase of active growth, Bacon listed what could be expected from science. His important expectations from science were the prolongation of life, the restitution of youth in some degree, the curing of diseases counted incurable, the mitigation of pain, the increasing of strength, the altering of constitution such as fatness, leanness and stature, making of new species, making better instruments of destruction for war, exhilaration of the spirits and putting them in good disposition, increasing the force of the imagination either upon another body or upon the body itself, acceleration of putrefaction and germination, making rich composts for the earth, making of new food out of substances not now in use, making new threads for apparel, etc.

While some of Bacon's notions are not shared by modern science and a few of his expectations are still not realized, it is obvious that we have achieved today almost everything that he desired and many things more. Bacon, for example, never imagined utilization of mechanical power, electricity, nuclear energy, acceleration of transport and communications, television, space travel, etc, which are common things at present. His major interest was in problems of health, freedom from common ailments and prolongation of life. The realization of all these expectations, nearly 250 years old, has not, however, solved the problems of mankind. The progress of science in one field has created new problems in another. Thus the advances in medical sciences have greatly increased the expectation of life. In advanced countries a newly born child expects to live now to the age of 75-80 years. Even in India the application of simple health measures in two decades since independence has resulted in nearly doubling the longevity from 27 to 53 years and the average male in Punjab expects to live for 60 years. The result has been a 'population explosion' unprecedented in history and mankind is faced with the grim possibility of dying from famine and starvation instead of disease.

*Address of Dr A. C. Joshi, General President, Fifty-sixth Session of the Indian Science Congress, Bombay, 3 January 1969.

The condition of many underdeveloped Asian countries, India, Pakistan, Indonesia, Philippines, China, etc, where civilization developed earlier than in other parts of the globe and which are already over-populated, is particularly precarious. Feeding their poor and malnourished millions has become a source of much anxiety to their governments. Foodgrains have had to be imported from outside in large quantities. Many experts forecast the occurrence of widespread famine in the early seventies.

During the last five years, however, a silent revolution has taken place in these countries through what has become known as the modern agricultural technology. This implies the use of high yielding dwarf varieties (wheat and rice), hybrid vigour (maize, jowar and bajra), dependable irrigation, fertilizers, pesticides and fungicides, better machinery and electric power. In 1967, world agricultural production set a new record and the underdeveloped countries accounted for most of that increase. The agricultural output in these countries rose by 7-8% over 1966. Per capita food production increased by 6%.

In Indian economic history also, 1967-68 is one of the most significant years. Ninety-five million tons of foodgrains were harvested. More than 15 million tons of wheat alone were harvested. This is more than double the production of 1951. In a few years, India aspires to be self-sufficient in foodgrains and even hopes to export some rice. The Philippines, which has been importing food in large quantities for many years, harvested a record rice crop in 1967 with only 14% of its rice-fields planted with the new 'miracle variety' IR8. It even exported some rice. Similarly, Pakistan harvested 5.5 million tons of wheat and it has good chances of achieving self-sufficiency in foodgrains in another year and is looking forward to an export potential in the Fourth Five Year Plan beginning from 1970. The first sproutings of the 'green revolution' have naturally generated all-round hope. We can say positively that many countries which were faced just a few years ago with the prospects of a large-scale famine are today on the brink of an unprecedented opportunity. The critical food problem of the next 20 years can be solved with known agricultural techniques.

If the present pattern of consumption continues, the foodgrain requirements of India by 1975-76 are expected to be 150 million tons. These will rise to 200 million tons by 1985. Both these targets are within reach by extending the cultivation of high yielding varieties already available in the country or likely to be produced at the plant-breeding stations in the near future and by providing the requisite inputs of water, fertilizers, etc. The task before the plant scientists now is to explore the

means of meeting the food requirements after 1985 and at the end of the century, as even with the full application of the known agricultural technology, people will outstrip even an expanded food supply.

The food problem could be solved if the world were to achieve a stable population in 20 or even 30 years. This is, however, not likely to happen. The average rate of population growth at present is about 2.5%. In large areas of the underdeveloped world the population growth is likely to shoot up to 3.5% as soon as essential health services are organized. Despite the fact that very determined efforts are being made in many countries to limit the size of families, the population is going to touch the 6 billion mark much before the end of the century. Whether it shall be stabilized by the end of the century is an open question. It will be hazardous to base plans of food production on such an assumption. We should be only too well aware of the fact that the extinction of many dominant species in the past has followed the explosion of population. Man by his skill has survived a series of such population explosions in the past following pastoral, agricultural and industrial phases of his cultural evolution. He can survive the present crisis of scientific revolution due to the progress in medicine by control of his environment and the proper planning of food production well ahead of his requirements. If the world is in a position to feed its population today, it is due to the basic discoveries made in plant sciences, that underlie agriculture, during the last 40 years. Planned scientific studies to feed a population of 6 billions or more at the end of the century must begin now.

While it may become possible to manufacture carbohydrates and other simple food materials in due course with the help of atomic energy or other sources of power without the aid of plants, one cannot hope much from such sources to feed the millions. Mankind will continue to depend for a long time for food largely on solar energy captured by the green plants.

Further, our efforts to utilize unconventional food, from *Chlorella* to wild tree leaves, though possible, pose formidable technological problems. Recent work of Odum and others has shown that the seas and the oceans are not so productive as they were once considered, since the holding of large proteinaceous biomass is no guarantee for sustained high rate of production. Indeed, they have been compared with deserts in the matter of rate of production among the terrestrial ecosystems.

Technically, it is possible to argue that land may be better utilized if we were to obtain our food resources largely from selected trees than from cereals. It is also sometimes said that the climate of tropical countries is better suited for 'tree culture' rather than for 'grass culture'. The argument runs like this: all materials used for food by man ultimately come from green plants which grow by utilizing the energy of the sunlight. Even the best farmlands use annually only a fraction of the solar energy that falls on them. Assuming the average annual input of solar energy for India as about 200 kcal/cm² and allowing for reflection from and

transmittance through the leaf of about 25% of the only 50% as photosynthesizable light energy, we get a figure of 12% of energy fixation on the basis of 10 quanta being used per CO₂ molecule. Roughly 25% of the fixed energy is used up in respiration, leaving only 9% of the fixed energy for growth of just 18 kcal of the input of 200 kcal/cm² for the whole year provided the land is covered with leaf all the time. However, in actual practice the average yield of paddy and wheat in India is 1 ton per hectare or roughly 0.01 g/cm² which corresponds to about 0.04 kcal of fixed energy per cm² (1 g plant tissue being equivalent to 4 kcal of energy). Trebling this figure to 0.12 kcal/cm² may give us a rough estimate of the total energy fixed during the growth of the whole plant during a season. If we raise two crops in a year, the annual yield can be 0.24 kcal of fixed energy per cm² against the potential of 18 kcal. The gap between the potential and realized values is staggering. The difference is mainly on account of light saturation of the photosynthetic apparatus so that most of the bright light during the day is not used. Nevertheless, it has been possible to raise plants which reach light saturation in more intense light. The rate of organic production in our forests has been shown to be of a higher order than achieved so far by any cereal, and possibly in trees the critical light saturation intensity is of higher order. Further, the wild vegetation does not need manuring, irrigation and labour input necessary for raising a crop. This is possible on account of the dynamic equilibrium of flow of energy and cycling of minerals in the natural ecosystem. It is said that man has only to learn the technology of extracting food from the wild vegetation in order to be free from the tedium of agriculture.

In spite of the higher productivity of the forests, it is not likely that men will change their food habits so drastically as to obtain the greater part of their energy requirements from trees. Conventional agriculture would continue to be the mainstay of food requirements of the people and the problem before our country as well as before other parts of the world is how to augment food production through conventional agriculture on a decreasing area of farmland*.

In the ECAFE region, during the period 1960-66, the average foodgrain output increased at the rate of 1.1% against 2.5% increase in the population. In India agricultural production increased at the rate of 3.1% against a rise in demand of 3.36% during the same period. The gap between food requirements and food output has been met by large-scale imports from outside. The heavy imports during the past years have affected very adversely our plans of economic development. Further, we are not sure whether foodgrains will

*We have no exact data for our country but it has been estimated that in the United States about one million acres of land are consumed every year by growing cities, suburbs, factories, roads, airports, etc. It is estimated that nearly one-tenth of the farmland will be used up in this manner in the next 40 years during which period the population will double itself. A similar situation may arise in India as we take to large-scale industrialization, housing of the ill-housed people, provision of roads, etc.

be available for us from other countries in the future. The food surpluses in USA are rapidly dwindling. All the production there may be required to meet its own growing population. Every country, unless it is heavily industrialized like Japan or Great Britain, would have to meet its food requirements largely by increasing production from its own fields.

Thus in most countries, two curves, one for decreasing farmland and the other of rising population, are on a collision course. The security of man lies only if new advances in plant sciences result in sufficient increase in productivity as to stave off this collision.

Fortunately, very rapid progress is being made in different branches of biology since World War II. Many thoughtful people have even begun to regard the second half of the 20th century as the commencement of the 'Age of Biology'. We are better aware now about the nature and diversities of life. The discoveries in botany are opening up entirely new vistas in our understanding of plants. Some of these findings are as important as the great discoveries in physics that have led to the tapping of nuclear energy.

When one attempts to classify these developments, it is seen that some of them are at present of a purely theoretical interest and have no direct bearing on agriculture. They greatly broaden, however, our knowledge of plant life. In this category can be placed the discovery of algae-like and bacteria-like fossils from pre-Cambrian rocks, which put back the origin of living organisms on the earth to about 3 billion years, that is almost a billion years earlier than was considered likely only a decade before. Another such example of great interest is the development of new biochemical techniques for characterizing and making visible the finest structures of the cells down to individual large molecules.

Other recent discoveries are of immediate practical value and hold out promise of revolutionary applications to agriculture and food technology. The most important among them are concerned with the understanding of the precise nature and the subtleties of heredity. As a result, the long-cherished goal of producing varieties of crops to exact specifications is now coming into view. This is making possible the production of many greatly improved strains.

A good example is the recent development of high yielding varieties of dwarf wheats. Scientific improvement of this cereal in India was started some 50 years ago at the Indian (then Imperial) Agricultural Research Institute. Eight years ago a critical study of factors responsible for the yield stagnation as well as instability in the production of this crop was made. It was concluded that the morphology and the developmental rhythm of the tall varieties cultivated in the country until then were not conducive to the production of wheat under conditions of high soil fertility and irrigation. The tall straw made the crop liable to lodging when 3-4 months old. Further, many of the common varieties were susceptible to rust and smuts. Whenever the farmers gave more irrigation and fertilizers, the intensity of attacks of rust and smuts increased.

In addition, in Northern India, rains and hailstorms, which are frequent in late March and early April, caused the lodging of the crop even under conditions of poor fertility. The analysis of this kind led the workers at the Indian Agricultural Research Institute in 1962 to the conclusion that dwarf wheat varieties were essential for breaking barriers to high yields in irrigated fields. A request was made to the Rockefeller Foundation for the seeds of the dwarf wheats already developed in Mexico. In 1963, after visiting the wheat-growing regions of the country, Dr N. E. Borlaug sent 100 kg each of 4 dwarf and semi-dwarf wheat varieties and small samples of 613 promising selections. The seeds were grown and the crops studied at Delhi, Ludhiana, Pusa, Kanpur, Pant Nagar, etc. In 1964, it was found that the Mexican varieties Lerma Rojo and Sonora 64 yielded over 4 tonnes per hectare at several places. Seeds were multiplied during summer months in the Nilgiri Hills in South India. During the *rabi* season, the dwarf wheats were tested at 155 locations and subjected to detailed pathological, physiological, agronomic and quality evaluation. In 1965, the results from yield trials conducted all over the country led to the release of Lerma Rojo and Sonora 64 for general cultivation. The yields obtained by farmers were so encouraging that it was decided in 1966 to import 18 thousand tonnes of seeds of Lerma Rojo and a few other varieties from Mexico. These were sown in 400,000 hectares during *rabi* season, along with the recommended doses of inputs, under the High Yielding Varieties Programme. In 1967, these dwarf wheats were cultivated in nearly 2 million hectares and thus started the great agricultural revolution in the country. Such rapid spread of new varieties has not been paralleled anywhere in the world. In the summer of 1968 nearly 17 million tonnes of wheat were harvested in the country as against the previous best of 12 million tonnes.

An amber-seeded mutant developed at the IARI by treating Sonora 64 with gamma rays and released under the name of Sharbati Sonora deserves special mention for its high protein content. Its yields have touched more than 6 tonnes per hectare. It possesses on an average 16.5% protein in contrast to about 14% in Sonora 64. Further, this variety has about 3 g of lysine in 100 g protein in contrast to about 2.4 g in Sonora 64. The mutant has helped to establish that protein quantity and quality in wheat can also be improved simultaneously with increasing yields. As the Indian diet is generally deficient in proteins, this discovery is of great significance in improving the nutrition of the masses. It also holds much promise for the future in developing high yielding as well as high quality crops.

The wheat revolution has helped to bring about a great change in the outlook and agronomic practices of farmers because new varieties can assist in doubling the yield and income. The farmers have become aware of the need for several fundamental changes with regard to the time and method of sowing and in the timing of irrigation and harvesting.

The recent developments in plant physiology are as revolutionary as those in the field of genetics.

The investigations on the different types of plant hormones—auxin, gibberellin and kinetin—are giving us new ideas about the growth and development. The basic studies on the auxins have led to the development of chemical weed killers. The discovery will save not only a good deal of back-breaking labour, but it will also greatly augment the yields of crops.

Rapid advances are being made with regard to the understanding of the influence of both internal and external environment on flowering. This has led, among other things, to the discovery of a new light-detecting pigment in plants, the phytochrome. These studies make it possible to have a precise control of the time of flowering of crops by chemical sprays or by exactly timed artificial illumination. This opens up the possibility of industrialization of agriculture, particularly of greenhouse crops.

Then there are discoveries about the occurrence and functions of the trace elements in soils—zinc, copper, boron, manganese, cobalt and molybdenum. These findings have helped to explain many problems of plant nutrition and hold out the possibility of bringing large areas of wasteland under fruitful cultivation.

The cultivation of isolated fragments and even single cells of plants marks a new advance on the very old problems of plant differentiation and organogenesis, namely how different tissues and organs can be formed from cells with the same heredity. The growth of whole plants from single cells has also many practical applications and holds out revolutionary possibilities in plant propagation.

There is a growing understanding of the master role of the nucleic acids in controlling growth, differentiation, pigment formation, etc. Then we have now a much clearer picture of photosynthesis, the ascent of sap and the translocation of nutritive elements, the chemistry of synthesis and breakdown of many of the major metabolites, the different kinds of enzymes, etc. These advances are enabling us to understand more fully many of the mysteries of plant life. While this knowledge is of fundamental importance for its own sake, it will in due course have practical applications in agriculture.

The mysteries of the biochemical reactions which lead to the fixation of nitrogen from the air have been gradually unravelled. These discoveries may open the way to vast new nitrogen fertilizer resources.

There are many questions about the nature of the plant world about which we still know very little. How is it, for example, that the green plants can manufacture from 16 elements an enormously complex series of organic compounds including, besides carbohydrates, fats and proteins, a variety of vitamins, many toxins, stimulants, drugs, scents, pigments, fibres, etc? What determines the sequence of germination, growth, maturity, fruiting and ageing of plants? How is it that the sea water kills most land plants if used for irrigation, whereas thousands of species of plants, including some flowering plants, live in the sea? Why different

species grow in specific habitats? Why certain plants cannot stand even slight frost, while others can live in subarctic conditions? It will take a long time before answers are found to these questions, but what has been learnt already in recent years holds out great promise for the future.

The application of the recent discoveries in different fields of plant science opens up vast possibilities of obtaining higher yields from the cereals as well as other crops several times the present level of production. In order to derive full advantage from this knowledge very sustained efforts to improve biological and agricultural education, research and extension are essential. The great increase in productivity in the United States during the last 40 years is mainly due to a highly developed biology and productive agricultural practices. In a country like ours, where agriculture is much less productive, the need to improve the study of and research in biological sciences is urgent. There has also to be created a climate of science in the country. Adjustments have to be made in land-use and cropping patterns. Irrigation facilities and rural electrification on a large scale have to be provided. Fertilizer production has to be greatly stepped up. Pesticides and fungicides have to be made easily available. Machines for quick harvesting, scientific storage and roads to villages have to be provided. Animal husbandry has to be greatly improved, keeping in view the fact that the cattle compete directly with man for food, that they are inefficient converters of energy and increase in their population is likely to deplete the primary production, but they have the capacity to digest the fibrous constituents of plants which otherwise are useless to man. Finally, all-out efforts are required to activate the vast population to the problems that it is facing, their possible solution, and to improving the farmer's ability. All this must be done without delay. We should not imagine that the food problem has been solved. It is going to be continuously with us and will need our earnest attention all the time.

Fortunately, man's position in the biosphere is unique. He has the capacity to manipulate the physical and biological environment to his advantage. His cultural revolution holds great promise provided he is conscious of his limitations, and plans to overcome them in good time. The remaining part of the century places him in a precarious position. Food production is undoubtedly increasing, but the population is growing as fast if not faster. Hence special efforts are required in developing countries to boost up agriculture in the hope that both food and population growth shall become stabilized by the turn of the century. This brief period may be regarded as a breathing spell in the race for man's survival. If he utilizes the opportunity wisely to his full advantage, making use of the scientific method and the technology that science has given him, he would overcome the present crisis of exploding population as he had overcome several other catastrophes in the past.

Conduction Mechanism in Organic Semiconductors

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THERE has been considerable interest lately in the electrical conductivity of organic semiconductors¹⁻⁹. Recently, Datt *et al*¹⁰ have discussed the various aspects of this interesting subject. The subject is interesting because the nature of charge transport in these materials is different in many important respects from that of conventional elemental and compound semiconductors. The theoretical understanding of electronic transport processes in organic semiconductors is a problem complicated by many subtle, interrelated effects. This difficulty still persists even if the discussion is restricted to the mechanism underlying the mobility of charge carriers in an applied electric field, thereby eliminating discussion of the processes of charge-carrier formation, recombination, trapping and other practical difficulties which exist in the total transport problem.

Experimental Facts

Any theory describing the process of electrical conduction in organic semiconductors must adequately account for the following experimental facts:

(i) The electrical conductivity can usually be fitted to an equation of the form $\sigma = \sigma_0 \exp(-E/kT)$, where σ is the electrical conductivity; σ_0 is a constant; E , the activation energy; k , the Boltzmann constant; and T , the absolute temperature. The activation energy E is found to be generally of the order of a few electron volts.

(ii) For some materials, ie naphthalene¹⁰⁻¹² and anthracene^{10,13}, a considerable anisotropy exists, so that conduction in a direction perpendicular to the cleavage plane is notably smaller than within any direction in the cleavage plane. For other materials, the anisotropy effect appears to be either much smaller or completely absent. The activation energy E is found to be independent of the crystal orientation within experimental error¹⁰⁻¹³.

(iii) The dark current is found to be ohmic up to the field strengths of 1500 V/cm (ref 1-15). Above this field strength the current increases at a more than linear rate. These trends in the variation of current with field strength are irrespective of the sample purity¹¹. The conductivities measured near room temperatures are very low ($\sigma = 10^{-7}$ - 10^{-18} ohm⁻¹ cm⁻¹ or even less). They change very little upon fusion, although exceptions do occur, as in the case of naphthalene^{10,16,17} and anthracene^{10,17}.

(iv) Though the effect of impurities has not been investigated very systematically by most of the authors, it appears that they affect both dark and photo-conduction¹⁻¹⁰. The studies of Datt¹¹, Northrop and Simpson¹⁴ and Gravatt and Gross¹⁵ on some organic semiconductors are significant in this direction. These studies conclusively show that the purity of the material has a marked effect on the magnitude of the electrical conductivity and on the low temperature activation energy, but the high

temperature activation energy seems to be relatively insensitive to the purity of the material. Hence, the high temperature activation energy is a characteristic property of the material and not a function of sample purity and can, thus, serve as a reliable parameter for comparison with other solids.

Comparison with Inorganic Semiconductors

Although more than 800,000 organic compounds have been characterized, the electrical conductivities of only a very small number have been studied¹⁻¹⁷. The major portion of the information on organic semiconductors of molecular crystal type published up to the middle of 1966 has been given by Datt *et al*¹⁰. The present paper is devoted completely to molecular crystals for three reasons. First, physicists are interested mainly in crystalline materials; secondly, maximum number of investigations have been carried out on the semiconductive properties of molecular crystals; and lastly, the shortage of space in the present paper does not permit the discussion of the other two types of organic semiconductors, ie charge-transfer complexes and polymers.

In the case of organic semiconductors of the molecular crystal type, it has been found that plots of $\log \sigma$ against the reciprocal of the absolute temperature yield one or two intersecting straight lines. This suggests a relationship between electrical conductivity σ and absolute temperature T of the form

$$\sigma = \sigma_0 \exp(-E/kT) \quad \dots \quad \dots \quad \dots \quad \dots \quad (1)$$

where the symbols have their usual meanings. From the formal similarity between this and the equation for the electrical conductivity of an intrinsic inorganic covalent semiconductors (eg germanium or silicon) the quantities in Eq (1) can be interpreted in terms of the model underlying Eq (2):

$$\sigma = \sigma_0 \exp(-\epsilon/2kT) \quad \dots \quad \dots \quad \dots \quad \dots \quad (2)$$

ie any conductivity is assumed to be intrinsic, since the material has been prepared as pure as possible. These model parameters are the effective mass m^* of the charge carriers, their mobility μ and concentration n . They are defined as follows:

$$\sigma = ne\mu \quad \dots \quad \dots \quad \dots \quad \dots \quad \dots \quad (3)$$

$$n = \frac{[2(2\pi m^* kT)^{3/2}] \exp(-\epsilon/2kT)}{h^3} \quad \dots \quad \dots \quad (4)$$

and

$$m^* = \frac{h^2}{4\pi^2 \frac{d^2 E'}{dK^2}} \quad \dots \quad \dots \quad \dots \quad \dots \quad (5)$$

where h is Planck's constant; E' , the energy associated with the wave vector K ; $K = 2\pi/\lambda$; and λ , the de Broglie wavelength of the electron.

The three assumptions implied in substituting Eq (2) for the experimentally obtained Eq (1) are: (i) the energies of the electrons in the solid can be described in terms of the two-dimensional analogue of a system of Brillouin zones; ie by definite energy bands separated by a forbidden zone; (ii) since the basic excitation process in intrinsic conduction involves the generation of an electron-hole pair, it must be assumed that one type of carrier becomes trapped in order to explain the experimental fact that organic semiconductors are either *p*- or *n*-type (in fact, most are *p*-type, ie they conduct by means of positively charged electron vacancies); and (iii) the energy of an electron is a parabolic function of the wave vector *K*.

The mobility will depend on certain details of the model employed. The material is, therefore, assumed to be an intrinsic conductor and scattering of the charge carriers due to impurities must be negligible, leaving only scattering by the lattice and by dislocations and other structural faults to limit mobility. Since only a few organic semiconductors can be obtained in the form of single crystals, the latter mode of scattering would be expected to predominate. However, in view of the irregularities necessarily involved in such crystal imperfections, no reliable calculations which are valid for a compacted powder or a cast pellet have been published.

The mobility contribution due to dislocations alone has been computed by Dexter and Seitz¹⁸; if lattice scattering alone limits mobility then the mobility should be proportional to $m^{*-5/2} T^{-3/2}$. Thus, in view of the exponential factor in Eq (2), it is further implied that the mobility varies relatively little with temperature and that any such variation would be compensated by the $T^{3/2}$ term in Eq (4) which determines the concentrations of charge carriers.

The above approach which is based on the assumption that the organic materials are intrinsic semiconductors obeying the same laws as the inorganic intrinsic semiconductors is not universally accepted^{1,19}. According to Garrett¹ and Fox¹⁹ there is insufficient justification at present for making such an assumption and even if the organics are intrinsic semiconductors, it is possible to show that the factor of $\frac{1}{2}$ should not appear in the exponent. The factor of $\frac{1}{2}$ in the exponent in the case of an inorganic intrinsic semiconductor appears because the possible distributions of electrons in the conduction band are independent of the distributions of holes in the valence band. This factor appears also in the case of an equilibrium distribution of ion pairs. However, in molecular excitations, once one determines a distribution of electrons among the excited states of the various molecules, the distribution of 'holes' in the ground state is completely determined. In such a case the factor of $\frac{1}{2}$ does not appear. These conclusions are based on general statistical arguments, not restricted to solids. It is required only that the Fermi level be far, compared to kT , from any of the one-electron states. Hence, a serious doubt is thrown upon the efforts to correlate $2E$ with 'energy gaps' determined by optical methods such as absorption or phosphorescence. This has

been shown theoretically and experimentally by some workers^{1,11,20}. In addition, Mott and Gurney²¹ have pointed out that, because of the Franck-Condon principle, thermal and optical activation energies should not be expected to agree.

Band Structure in Molecular Crystals

Even if a purity sufficiently high to ensure intrinsic conductivity can be achieved, it is highly questionable to what extent a model based on studies of covalent crystals like germanium or silicon can be safely extrapolated for organic materials in the crystalline, glass or liquid form. It has been shown by Ioffe and Regel^{22,23} that serious contradictions arise when the low values of mobility obtained for most organic semiconductors are considered. A carrier of effective mass m^* and mobility μ , at ordinary temperatures, has a mean free path *L* given by the approximate relation²²:

$$L \approx 10^{-8} \mu \left(\frac{m^*}{m_0} \right)^{\frac{1}{2}} \dots \dots \dots (6)$$

where m_0 is the mass of the free electron. Values of *L* lie between 0.1 and 10^4 Å. Consider a free electron or hole moving with thermal velocity at ordinary temperatures. Since its de Broglie wavelength λ is of the order of 70 Å, it appears that *L* becomes less than λ if the mobility drops below about 100 cm²/V-sec. However, as an electron is 'smeared out' over distances of the order of λ , a motion over a distance *L* less than λ becomes meaningless. Moreover, most organic substances have values of mobility less than 5 cm²/V-sec so that the mean free path *L*, calculated from mobility data, is of the order of 0.1-1 Å; this value is appreciably less than the lattice spacing in a molecular crystal. Therefore, neither the concept of a mean free path nor that of a free electron mobility has any meaning in such a case, since the electron wave is completely damped out within less than a single lattice spacing.

Further difficulties arise if the change in conductivity upon melting is considered. If lattice scattering were indeed the rate-determining step, then a change in the conductivity similar to that observed in the case of metals would be expected. Most metals obey, at least approximately, the Mott²⁴-Harasima²⁵ relation

$$\frac{\sigma_{\text{solid}}}{\sigma_{\text{liquid}}} = \exp(-2L_f/3T_f) \dots \dots \dots (7)$$

where L_f is the latent heat of fusion per molecule or per atom, and T_f the melting point; numerically this ratio is about 2 for most metals. The loss of long-range order on melting, however, has even less influence on the conductivity of organic semiconductors than it has on metals where lattice scattering is the main mode of scattering. It has been shown^{16,17} that, though conductivity increases by some orders on melting, there is no change in the slope of the log σ versus $1/T$ curve on melting. Hence, it may be assumed that melting only raises the mobility to a higher value in the liquid state but leaves the band structure of the organic substance unaltered.

Therefore, it appears that the band structure arises not because of long-range order, but rather is determined by the chemical bonding, the type of coordination and the type of short-range order in the substance, and is thus preserved upon melting. Long-range periodicity may be a sufficient but is not a necessary condition for the development of energy bands. The concept of a conduction band separated from a valency band by a definite energy gap may, therefore, be applied to a molecular crystal. On this basis, it has been shown by Gubanov²⁶ that the width of the gap remains substantially unaltered if a crystal having long-range order melts into an amorphous liquid or glass having only short-range order, provided that the coordination number remains unchanged. The following expression for the conductivities in the solid and liquid states can be derived²⁷:

$$\frac{\sigma_{\text{solid}}}{\sigma_{\text{liquid}}} = \frac{\mu_{\text{solid}}}{\mu_{\text{liquid}}} \exp(\Delta\epsilon/2kT) \quad \dots \quad \dots \quad (8)$$

where $\Delta\epsilon$ is the change of the energy gap upon fusion. Since $\Delta\epsilon$ is small, the value of the fraction also becomes small and in the limit approaches the mobility ratio.

Mobility Problem and Conduction Mechanism

It is clear that mobility as such has little meaning in the case of organic semiconductors where intermolecular distances are of the order of several Ångströms. The very existence of organic semiconductors, however, shows that some means of charge transfer must exist to permit an electron or hole to migrate from one molecule to another. Whatever the nature of this process, it must be a very improbable event since the conductivity values are small.

The distinguishing feature of the organic semiconductors is their narrow bandwidths; specifically, bandwidths $\lesssim kT_r$, where k is the Boltzmann constant and T_r the room temperature. As a consequence of the small overlaps, it is not evident *a priori* that a conventional energy band model and Boltzmann equation treatment are applicable in describing their transport properties. It has been shown²⁸ that in semiconductors of sufficiently narrow bandwidths, for which the electron-lattice interaction is sufficiently strong, the latter must, in effect, be taken into account before the periodic potential, giving rise to the so-called small polaron mechanism of charge transport. For this case at sufficiently high temperatures, a band model is found not to apply. Rather, the transport mechanism is a thermally activated^{29,30} hopping motion of the charge carrier between neighbouring lattice sites in which the interaction of the carrier with the lattice plays an essential role.

It is, therefore, somewhat surprising that there is no experimental evidence of the above mechanism in the commonly studied organic molecular crystals, i.e. anthracene and naphthalene. Thus, although the electrical conductivity follows the standard semiconductor Eq (1), the temperature dependence of the mobility, as deduced from transit time^{31,32} and Hall measurements³³, varies typically like $\mu \sim T^{-n}$,

where $3 \geq n \geq 1$. In particular, there is no evidence that the mobilities have an activation-type temperature dependence.

Band Model

One line of attack on the conduction problem in molecular crystals was started by LeBlanc³⁴, who noted that electron and hole mobilities increase with decreasing temperature³¹ in a manner not unlike that to be expected from a band theory model. Charge transport by phonon-assisted hopping from one site to another would, on the other hand, result in an exponential increase of mobility with increasing temperature. This observation led LeBlanc and others to treat the conduction problem within the tight-binding approximation of the band theory³⁴⁻³⁶. Intermolecular overlap integrals for anthracene were calculated first by Murrel³⁷. Later calculations of this type are provided by Thaxton *et al*³⁵, and Rice *et al*^{36,38,39} for anthracene and naphthalene crystals, which have base-centred monoclinic crystal structure. Except for variations in the estimates of the intermolecular transfer integrals among the above workers, this aspect of the problem is relatively simple due to the simplicity of the tight-binding approximation. This theoretical work has indicated that the bandwidths for excess electrons and holes in molecular crystals of the naphthalene-anthracene type are very narrow, 0.2 eV at the largest, and possibly smaller than that by an order of magnitude or more.

The more difficult problem within the band approximation is a proper treatment of the scattering. Most of the workers^{35,36,38,39} have assumed the existence of a constant relaxation time τ . This assumption is often qualified by noting that τ may depend on K , the electron wave vector. However, Friedman⁴⁰ has pointed out that it is likely that under some circumstances, the relaxation time assumption may be entirely unjustified, and that, due to the narrow bandwidths, the scattering may not be amenable to a standard, one-phonon deformation potential treatment.

There are two important respects in which the narrow bandwidths must be taken into account. The first relates to the validity of the band picture. With the mobility μ given by⁴¹

$$\begin{aligned} \mu &= \left(\frac{e}{kT} \right) \langle \tau v^2 \rangle \\ &\cong \left(\frac{e\tau}{kT} \right) \frac{W^2 a^2}{\pi^2 \hbar^2} \end{aligned}$$

where v is the average thermal velocity of the electron; W , the bandwidth; and a , the lattice constant, the energy definition of the band states requires that

$$\left(\frac{\hbar}{\tau} \right) < W$$

Thus, a lower limit on the mobility is given by

$$\mu > \frac{ea^2}{\hbar} \cdot \frac{W}{kT}$$

With $a = 3 \times 10^{-8}$ cm, one finds that^{42*}

$$\mu \gtrsim 0.1 \frac{W \text{ cm}^2}{kT \text{ V-sec}}$$

Hence, mobilities of the order of unity are on the border-line of a band description. Mobilities of the order of 0.1-1.0 are observed experimentally^{31,43} and, therefore, their description by means of band motion is not in clear violation of the uncertainty relation.

But, since the bands are quite narrow in the absence of lattice vibrational effects of a self-trapping nature, one must consider that the standard formulae regarding phonon scattering may not be applicable. Rather one may be forced to use higher orders of perturbation theory⁴¹, or even a change in basis from the Bloch representation to a localized representation may be required²⁸. Friedman⁴⁰ estimated that the self-trapping effects of intramolecular vibrations would not be important in the aromatic solids of naphthalene-anthracene type, and went on to derive improved formulae for various transport phenomena in the narrow band limit. His estimates have since been not accepted and now it appears that self-trapping effects may actually be quite important⁴⁴.

Hopping Model

A second line of attack on the conduction problem pictures the electrons and holes as jumping from one site to another. As Eley first suggested, this could occur by tunnelling through the intermolecular barrier rather than by phonon-assisted hopping over the barrier^{10,45,46}. The tunnelling mechanism does not require that a charge carrier be raised a given amount above the ground state; therefore, the model cannot be rejected on the basis of the experimental temperature dependence of mobility. More generally, tunnelling probabilities will depend upon the vibrational quantum number and one might still expect to see a thermal activation effect. In addition, the population of acoustic phonons will not change much except at very low temperature (because of their low energy). Finally, the increase in the equilibrium thickness of the intermolecular potential (ie crystal expansion) with increasing temperature will tend to decrease mobilities with increasing temperature, an effect opposite to that produced by thermal vibrations in a crystal of constant volume. Apart from the opaque question of temperature dependence, quantitative calculation has shown that simplified hopping models can account reasonably well for electron mobilities in anthracene^{47,48}. But the state of the theory underlying a hopping model is still in a rather weak position. In this case, the lacuna is not that the bands are too narrow, but rather that they may not be narrow enough, and further, one does not know how to adequately treat the time-dependent problem to get quantitatively accurate expressions for the ump probabilities.

It is instructive to compare this condition with that applicable to the conventional wide band case. In this case with $\mu = e\tau/m^$, the requirement that $(h/\tau) < kT$ leads to the condition that $\mu > (m/m^*) \times 20 \text{ cm}^2/\text{V-sec}$ at room temperature [Eq (3) of ref 41; also ref 42]. While it is possible that $m^* > m$ so as to allow mobilities of the order of unity, $\mu \neq e\tau/m^*$ for the narrow band case, as discussed in the text.

Recently, Glaeser and Berry⁴⁹ have made an attempt to decide which of the two extreme models is the more useful as a first approximation to understand the conduction mechanism in organic molecular crystals. Anthracene was the principal solid examined, but similar calculations have also been given for naphthalene. A critical re-evaluation is made of the transfer integrals which have been calculated from molecular orbital wave functions, particular attention being given to include the effects such as electronic polarization, whose importance was not estimated earlier. These transfer integrals have been used to examine the conduction problem in the two extreme limits: (i) in terms of simple band theory, in which a delocalized, tight-binding representation is used; and (ii) in terms of a simple hopping model where site-to-site hopping by resonance transfer (tunnelling through the intermolecular barrier) is valid. Their calculations have shown that the usual model of a band representation and weak phonon coupling is probably not the best starting point for a discussion of charge mobility in organic solids of the naphthalene-anthracene type. The bands are apparently too narrow for the simple band model to hold; when the effects of polarization are included, the bands are narrowed considerably and interaction with optical phonon would probably narrow them further still. To take the narrowness of the bands into account and still retain the delocalized picture is not a trivial problem^{28,41}. On the other hand, a simple hopping model appears to be indicated by theoretical considerations associated with the small molecular interactions (ie narrow bandwidth)²⁸. The assumption that the resonance transfer is the time-limiting process leads to quantitative predictions regarding the mobility of excess electrons or holes.

It can, therefore, be concluded from the critical re-evaluation of the two extreme models that the hopping model is superior to the band model, and that the former gives a quantitative picture of the physical mechanism underlying electrical conduction in organic molecular crystals of the naphthalene-anthracene type. However, it is recognized, as pointed by Glarum⁵⁰, that the calculated transfer integrals and observed mobilities lie in a range intermediate between the domains of rigorous validity for either model.

Summary

A comparative study is made of the applicability of the two limiting models, band and hopping models, for obtaining a quantitative picture of the physical mechanism underlying charge transfer in organic semiconductors of molecular crystal type. In both the models, the transfer integrals are calculated from molecular orbital wave functions. But, most of the authors have calculated these transfer integrals without paying any attention to include the effects such as electronic polarization. However, if the effects of electronic polarization are considered in the evaluation of the transfer integrals and the modified transfer integrals are used to examine the conduction problem in the two extreme limits: (i) in terms of simple band theory, in which a delocalized, tight-binding representation is used;

and (ii) in terms of a simple hopping model where site-to-site hopping by resonance transfer is valid, then the hopping model is found to be superior to the band model, and gives a quantitative picture of the physical mechanism underlying electrical conduction in organic molecular crystals of the naphthalene-anthracene type. However, it should be pointed out that the calculated transfer integrals and observed mobilities lie in a range intermediate between the domains of rigorous validity for either model.

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Determination of Force Constants*

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&

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THE main techniques which have been used for the determination of intermolecular forces are based on (1) quantum mechanical calculations, (2) spectroscopic observations, (3) relaxation methods, (4) beam scattering, and (5) macroscopic properties.

The discussion in the present review will be confined to the determination of force constants through the macroscopic properties of gases. The dependence of such properties of gases on the force laws provides a convenient means of obtaining reliable information about these forces. The physical properties thus used are: (1) equilibrium, (2) transport, and (3) crystal properties. Out of these the equilibrium properties find a definite preference over the others due to the existence of accurate and well-understood theories correlating the force constants and the macroscopic properties. The transport properties, on the other hand, are less suited to an investigation of intermolecular forces because of the presence of internal degrees of freedom. The theory of crystal properties involves certain assumptions like pair-wise additivity, etc, which may not be strictly valid.

The various intermolecular forces are described in terms of different potential functions which have been suggested on semi-empirical basis. All sorts of omission and commission made in the choice of a potential model have to be absorbed into the numerical values of the adjustable potential parameters whose values are obtained by the best fit of the experimental data on some appropriate macroscopic property. The criticality of the method in fitting considerably affects the reliability of the force constants thus determined. These methods of determining the potential parameters from the experimental data on bulk properties of gases have been discussed by Strehlow¹, Whalley and Schneider², Srivastava and Srivastava³, Saxena⁴, Srivastava and Madan⁵, Mason and Monchick (private communication) and others. The methods which have been often used for the determination of potential parameters from the experimental data on bulk properties are: (1) translational method, (2) intersection method, (3) ratio method, and (4) least squares method.

The above methods are reviewed and compared with the other methods (suggested by the present authors) in this paper.

The central potentials can be represented in the form

$$\phi(r) = \epsilon f(\sigma/r) \dots \dots \dots (1)$$

*Paper presented at the convention organized by the Physical Research Committee of the Council of Scientific & Industrial Research at the Andhra University, Waltair, in February 1968.

For such potentials the second and third virial coefficients, the isothermal Joule-Thomson coefficient, coefficients of viscosity, thermal conductivity and diffusion can be given by the equations of the type

$$\xi = R\psi(T^*) \dots \dots \dots (2)$$

where ξ is the concerned coefficient; R , a constant which depends on σ or r_m ; and $\psi(T^*)$, some function which depends only on T^* .

Translational Method

In the translational method, also named after Lennard-Jones⁶ who first employed it, log of the experimental values of ξ is plotted against log T on a transparent graph paper. Another plot of log $\psi(T^*)$ against log T^* is drawn on the same scale as for the previous plot. The former plot is then superposed on the latter by simple translations parallel to the two axes. The translation along the log $\psi(T^*)$ axis gives log R and hence the value of σ , while translation along the log T^* axis gives log (ϵ/k) . This method requires data over a large temperature range and unless there are some distinguishable peculiarities in the curve it is not possible to successfully exploit the method. The method thus is not always very reliable though it is a quick one and permits easy comparison of several potentials if the theoretical curves are plotted on the same graph paper.

Intersection Method

The intersection method of Buckingham⁷ requires experimental data only at two temperatures at which the quantities

$$\log R = \log \xi - \log \psi(T^*) \dots \dots \dots (3)$$

and

$$\log (\epsilon/k) = \log T - \log T^* \dots \dots \dots (4)$$

are determined for various values of T^* around the expected value. If log R is plotted against log (ϵ/k) , two curves will be obtained for the two temperatures. The intersection point will give the values of R and ϵ/k satisfying the experimental data at the given temperatures. If the process is repeated for different pairs of temperatures, the mean of all the intersections will give the best fit values of σ and ϵ/k . This method thus requires accurate data at few temperatures only to give reliable values. Another simplified way to use this method is to tabulate experimental values of $\psi(T^*) = \xi/R$ for a series of arbitrarily chosen values of σ for the two temperatures. A comparison of these experimental values of $\psi(T^*)$ with the theoretical values will give the value of T^* and hence

Their calculated values of ϵ/k and σ show a definite and significant dependence on temperature. However, scarcity of reliable data on Joule-Thomson coefficient over a wide temperature range for CO_2 makes it difficult to explain this variation with certainty and in all probability the simple L - $J(12-6)$ potential is not adequate for this complicated molecule

In the light of the above discussion this method finds preference over others wherever reliable experimental data on the two equilibrium properties, viz second virial coefficient and Joule-Thomson coefficients, are available.

All the above methods, except the preceding one, employ a single bulk property for the determination of the force constants. The constants thus determined from one property vary markedly from the constants determined with the help of another property. Consequently, it has become desirable that the parameters obtained from one transport property, preferably viscosity, should be used to interpret the data on transport properties only; similarly the parameters from an equilibrium property should be used for calculations of equation of state and thermodynamic properties⁸. This suggests that these force constants are not the true representatives of the molecular interactions which are common to all these physical phenomena. To overcome this it is preferable to use simultaneously two macroscopic properties belonging to different classes (transport, equilibrium or crystal properties) to evaluate the force constants. With this view Nain *et al*¹⁴ have suggested a method which utilizes the experimental second virial coefficient and viscosity data simultaneously. They have tested the appropriateness of the method on the L - $J(12-6)$ potential function by calculating second virial and viscosity coefficients for Kr.

The second virial coefficient, $B(T)$, for the central L - $J(12-6)$ potential is given by

$$B(T) = \frac{2}{3}\pi N\sigma^3 B^*(T^*) \quad \dots \quad \dots \quad \dots \quad (10)$$

The different symbols bear the usual explanation.

The coefficient of viscosity, η , for pure gases is given by

$$\eta \times 10^7 = 266.93 \frac{(MT)^{1/2}}{\sigma^2 \Omega^{(2,2)*}(T^*)} \quad \dots \quad \dots \quad \dots \quad (11)$$

where M is the molecular weight and $\Omega^{(2,2)*}(T^*)$ the reduced collision integral. The values of $\Omega^{(2,2)*}(T^*)$ for the (12-6) potential are given by Hirschfelder *et al*⁸.

With the help of Eqs (10) and (11) the final expression can be written as

$$\frac{[B(T)]^2 [\eta]^3}{(MT)^{3/2}} = 3.0367 \times 10^{-14} \frac{[B^*(T^*)]^2}{[\Omega^{(2,2)*}(T^*)]^3} \equiv Q^*(T^*) \quad \dots (12)$$

The right-hand side of Eq (12) is a function of T^* only, while the left-hand side involves simply the experimentally determined quantities. Thus, the knowledge of $B(T)$ and η at a common temperature T and the molecular weight M gives the value of left-hand side in Eq (12), which when compared with the theoretical values of $Q^*(T^*)$ will yield the value of T^* and hence ϵ/k . This value

of ϵ/k in conjunction with Eq (10) or (11) will enable the determination of the value of σ . These parameters thus determined will correspond to the particular temperature T at which the experimental data were used.

The success of the method and the parameters thus determined was tested by calculating the viscosity and the second virial coefficients using the same parameters. From these calculations on Kr, CO_2 and CH_4 , Nain *et al*¹⁴ concluded the following:

(1) The set of parameters determined from combined viscosity and virial data are capable of interpreting both the viscosity and second virial coefficients with desirable accuracy.

(2) The force constants determined from pure viscosity data are unsuitable in predicting the second virial coefficients and vice versa.

(3) In certain cases (Kr) the combined fit reproduces $B(T)$ and η better than the 'best fit' for $B(T)$ and η separately. This deviation may be due to either poor curve fitting in the determination of the force constants from $B(T)$ and η individually or the use of a smaller temperature range as compared to that employed in the present investigation.

(4) The values of ϵ/k determined at different temperatures varied insignificantly for Kr and CH_4 , while for CO_2 the variation was as much as $\pm 5\%$ from the mean value without any systematic trend. Thus the temperature dependence of the parameters for CO_2 discussed earlier could not be confirmed here.

(5) The method lays more emphasis on the viscosity data as compared to the second virial coefficients. This, therefore, will result in more satisfactory prediction of the viscosity than the second virials.

Summary

Various methods used very often for the determination of potential parameters from the macroscopic properties of gases are briefly reviewed. Two additional methods, one involving the two equilibrium properties, viz second virial and Joule-Thomson coefficients, and the other involving the equilibrium and transport properties, viz second virial and viscosity, simultaneously suggested recently are discussed at length. Calculations performed on several gases including monatomic through complicated molecules are discussed.

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Arc Techniques in the Preparation of Inorganic Materials

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THE arc technique was first used by Hare¹ in the synthesis of inorganic materials like CaC₂, Ca and P. Later, Moissan² developed the technique of using carbon electrodes in air or carbon monoxide atmosphere to attain temperatures of up to 4000°C, a technique still being widely used. Kroll³ has described an arc melting furnace for processing refractory materials in inert atmospheres, employing tungsten electrodes and a water-cooled hearth. Apart from these isolated examples, electric arcs seem to have been little used in inorganic preparative chemistry. Recently, interest in this technique has been revived, as exemplified by some recent publications⁴⁻⁶. Using a single arc, Reed⁴ has melted a wide variety of materials including metals, alloys and compounds such as ThO₂, BeO, CeO₂, Al₂O₃, ZrO₂, etc. It was felt that the attention of a wider group of scientists needs to be drawn to the simplicity of the technique, and its great utility in carrying out a large number of high temperature chemical reactions. The present paper reviews the design and techniques followed in the construction and operation of the arc furnace. The observations reported are based largely on experiences gained at the authors' laboratory with equipment adapted from the design by Reed⁴ and Reed and Pollard⁵.

Single Arc

Basically, an arc is generated by the passage of current from a small thoriated tungsten cathode to a crucible containing the materials to be reacted, acting as an anode (Figs. 1 and 2). The temperature of the plasma exceeds a minimum of 10000°K required to maintain sufficient ionization and conduction, and an upper limit near 30000°K, set by the energy density that can be maintained. The shape of the cathode tip is particularly important in the melting of non-conductors, since the high velocity magnetohydrodynamic flame which issues from the tip is a result of the high current density there. Therefore, the tip should be ground to a

point with an included angle in the range 30-60°.

Power is conveniently supplied by rectified power sets used in standard welding operations, though motor generator sets or batteries with series resistance ballast are satisfactory in many instances. Typical operating conditions involve currents of the order of 70 amp at 15 V passing through a gas. These gases circulate in a chamber enclosed at the bottom by the cold hearth, at the top by an aluminium frame holding the cathode, and surrounded on the sides by a cylindrical pyrex or vycor glass sleeve.

Arcs may be maintained in inert or reducing atmospheres such as argon, helium, nitrogen or hydrogen in the range 100-760 torr. Traces of oxygen or volatilized carbon attack the tungsten electrode. Thus, it is important to getter the gases by passage over heated active metals (Ti at 500°C). If it is desired to operate in oxygen, a carbon electrode must be used, but it will produce carbon monoxide by oxidation of the cathode and thus affect the oxygen partial pressure in the system.

The crucible, hollowed out from a cylindrical copper hearth, is maintained near room temperature by water flow through cooling channels. An arc is first struck by lowering and swivelling the electrode so that it touches a spot on the cold hearth. The current is raised slowly while the cathode is simultaneously withdrawn, so as to maintain the arc, which is then moved until it bathes the materials to be reacted in the crucible. The current is increased until the material has reacted in the molten state; on turning off the current, one generally obtains a frozen button.

With a water-cooled hearth, enormous temperature gradients develop at the interface between the melt and the crucible. In all probability, a very thin skin of the sample separates the melt from the copper hearth; in this sense the sample forms its own crucible, and serious contamination with copper is avoided. The method thus lends itself to the synthesis of material in highly purified form.

Details concerning the construction of the cathode are shown in Fig. 1. The principal problems are

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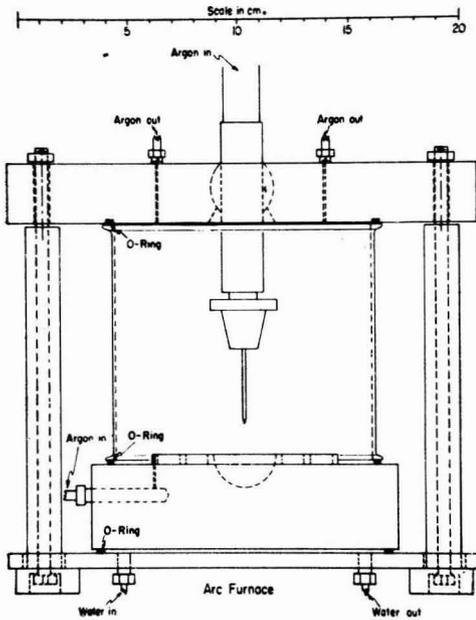


Fig. 1 — Side view of single arc melter drawn approximately to scale [Crucible is in the central, hollowed-out section of the water-cooled hearth; the cathode is fitted through a spherical holder that can swivel in the aluminium frame forming the top. A pyrex or vycor sleeve separates the top and bottom]

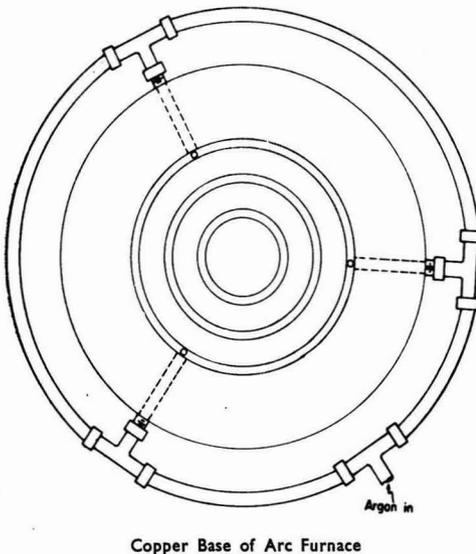


Fig. 2 — Top view of single arc hearth [Outermost ring represents feed line for circulation of inert gases through the hearth into the reaction chamber]

(a) to insulate the cathode from the remainder of the system at ground potential by means of a ceramic sleeve, and (b) to keep the cathode tip reasonably cool, by directing a flow of cool gas over it via nozzles in the tip. Gas circulation through the chamber is achieved through these openings, as well as through holes in the hearth itself; the location of the ports is indicated in Fig. 2. For more efficient cooling to reduce the possibility of contamination of the sample by tungsten vaporized from the cathode, we have constructed a water-cooled cathode which has a replaceable thoriated tungsten tip (Fig. 3). Swivelling action is achieved by inserting the electrode in a steel ball which is allowed to rotate on a support in the top part of the frame resting on the hearth. Although we have not done so in this laboratory, flexible brass or stainless steel bellows could be substituted for the steel ball bearing to connect the cathode to the furnace chamber and allow the necessary freedom of movement for the cathode. It is necessary to shield operating personnel from the intense UV radiation generated by the arc.

We have found the arc technique to be of particular value for the preparation of metal oxides with varying degrees of stoichiometry. Thus, we have prepared the oxides TiO_x ($0 < x < 2$), VO_x ($0 < x < 2.5$), NbO_x ($0 < x < 2.5$) and mixtures of these by using stoichiometric quantities of the metal and its corresponding oxide in the highest valency state. Other compounds made in this laboratory

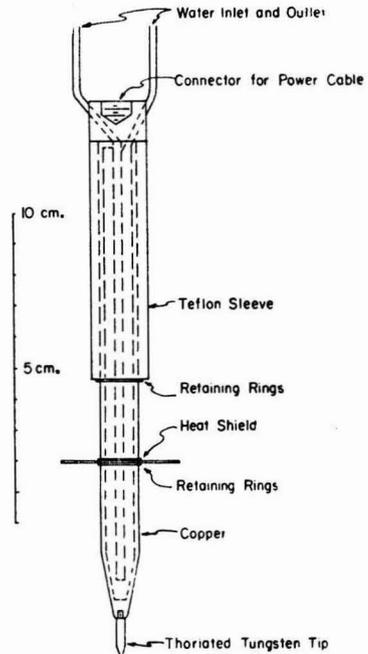


Fig. 3 — Water-cooled cathode [Section through centre, approximately to scale]

include Ln_2O_3 , LnCrO_3 , LnMnO_3 , LnFeO_3 (Ln = rare earth) and $\text{CeO}_2\text{-Y}_2\text{O}_3$ and $\text{TiO}_2\text{-Y}_2\text{O}_3$ mixtures. The buttons obtained from arc melting are often quite satisfactory for many electrical and other measurements. The method also has been found to be very convenient for carrying out inorganic syntheses requiring high temperatures. For example, when equimolar quantities of TiO and VO are melted together the resulting bead shows a phase separation into a dark outer region of the composition $(\text{V}_x\text{Ti}_{1-x})_2\text{O}_3$ and a silvery inner portion $(\text{V}_x\text{Ti}_y\text{O}_z)$. This illustrates that the arc technique can be quite useful in the study of high temperature phase equilibria.

Tri Arc

Reed⁴ has pointed out that if a material is suitable for growing single crystals from the melt, buttons of the material will cool with the formation of large crystal grains. One can construct a furnace suitable for pulling single crystals of these materials by slightly modifying the single arc furnace described previously. The side view of the tri arc furnace is shown in Fig. 4. The main differences between the single and tri arc furnaces involve the substitution of a water-cooled pulling rod (a) in the position occupied in the single arc melter by the cathode, a crucible (b) of high density graphite which can be raised and lowered and the use of three symmetrically arranged thoriated tungsten cathodes (torches) instead of one. The pulling rod can be raised and lowered by turning the handle (c) which is connected to the pulling rod through a rack and pinion gear drive mechanism. Turning the attached crank (d) allows the pulling rod to be rotated on its axis. The lower part of the pulling rod consists of two coaxial tubes, with a water inlet to the annular region, and an outlet from the central tube through a rotating seal (e). The bottom of this assembly is threaded to accept a short replaceable tungsten rod, the only part of the pulling assembly which ever touches the molten sample. A water flow rate sufficient to keep the tungsten pulling rod cool, thereby minimizing tungsten contamination, is maintained during the course of pulling a crystal. A gas-tight seal through the aluminium top of the arc furnace is maintained by the tight fit of the pulling rod in a teflon bushing. In series with each of the torches is a water-cooled ballast resistor ($R \approx 0.5$ ohm) which serves to allow the striking of the arc from the second and third torches once the first has been struck and also to stabilize the plasma arc once all three torches are on.

The crucible consists of a high density graphite cylinder with a slight concavity machined on the top. The cylinder fits tightly in a hole bored through the copper base of the arc furnace but can be moved upward using the screw drive (f) at the bottom of the furnace. This allows the level of the melt to be maintained as material is withdrawn in the form of a solid single crystal.

In practice the pulling of a single crystal is accomplished by starting all three torches and adjusting the current and the placement of the torches so that the top surface of the sample is slightly above its melting point. This requires

some practice, but the colour of the molten sample can serve to help in determining when the proper temperature is attained. The pulling rod is then cranked down until the tip just touches the melt. The melt usually wets the rod at this point and the slow pulling process can be started. If the plasma arc is properly adjusted, an irregular cylinder of solid sample (most likely polycrystalline) will emerge from the melt. By controlling the rate of pull and crank rotation, the sample can be necked down until only one single crystal remains; this also requires practice. This is equivalent to the method of starting the pulling of a single crystal using a single crystal seed on the end of the pulling rod. Once the sample has been necked down, slow, careful turning of the crank should produce a single crystal boule whose diameter is determined by the rate of pull and the placement of the torches. The screw drive must be turned periodically to maintain the level of the melt. Nice single crystal boules of Ti_2O_3 , NbO and Ti_2O_3 have been grown in this laboratory using the techniques described

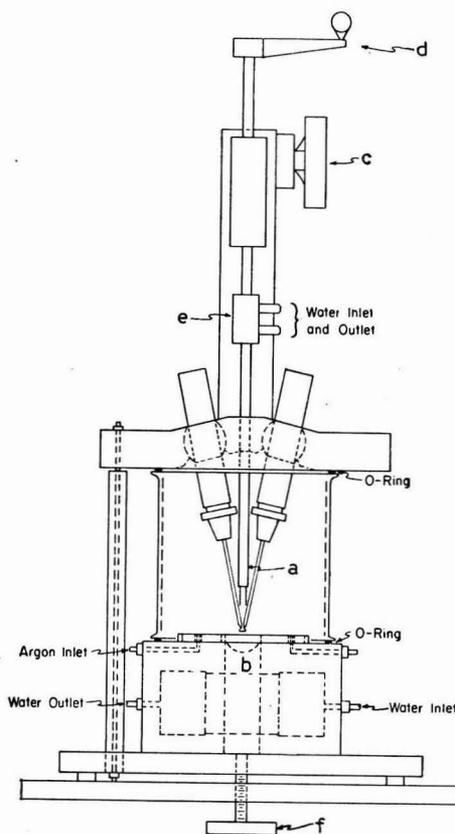


Fig. 4 — Side view of tri arc crystal pulling furnace approximately to scale [a, water-cooled pulling rod; b, crucible of high density graphite; c, handle; d, crank; e, rotating seal; and f, screw drive]

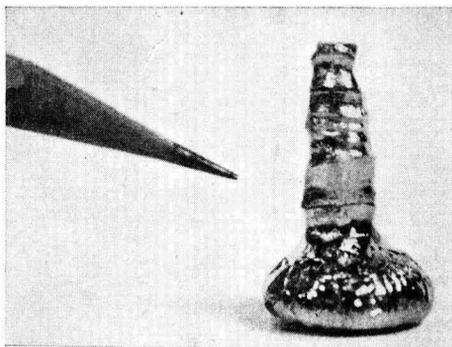


Fig. 5—Single crystal boule of NbO still attached to the polycrystalline bead from which it was pulled

here. In Fig. 5, a crystal boule of NbO obtained by this technique is shown as a typical example.

Conclusion

Modifications of the apparatus described here can readily be made to allow the arc technique to be applied to the melting of fine powders, zone refining, arc casting, rapid quenching or for use as a plasma torch⁴.

It may be of interest to compare the arc to other heat sources available for the preparation of inorganic materials. An electron beam could be more

intense than an arc, and the high vacuum in the former case could be useful in removing gases from the solid samples. However, the inert atmosphere in the arc is helpful in minimizing evaporation of components. The arc technique has the advantage over the induction furnace in that it is able to heat non-conductors directly. The economy and convenience of the arc furnace compared to other methods should make it a valuable tool in any materials preparation laboratory.

Summary

Techniques for the ready preparation of inorganic materials for solid state research are discussed. With the single arc furnace, polycrystalline beads may be made easily, while the tri arc furnace allows one to pull single crystals of a variety of materials. The problem of reaction between sample and crucible material is eliminated in both these methods.

Acknowledgement

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The Applications of Radioisotopes in Metallurgy in India—Present Position & Future Potentialities

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TO review fully the present status and future potentialities of the applications of radioisotopes in metallurgy in India is by no means an easy task. It was only in 1958 that a beginning was made in the production of radioisotopes in this country, using the first reactor APSARA. The availability of the powerful CIRUS reactor in 1960 made it possible to produce most of the commonly required radioisotopes in quantities and specific activities adequate to meet the needs of industrial applications. Simultaneously, the development of nuclear electronic equipment at Trombay stimulated more and more institutions to work on the application of radioisotopes. A number of training programmes in certain areas of industrial applications like radiography and activation analysis, radiochemical techniques and handling of isotopes have been organized at the Bhabha Atomic Research Centre (BARC). Besides this, actual assistance is being provided, whenever requested, in the planning and installation of radioisotope laboratories.

Radiography

Industrial isotope radiography has been the most important industrial application in this country and is in regular use today in more than 65 different undertakings either for production control within factories or for inspection during the construction of new installations. The users include all the major fertilizer factories and oil refineries, several thermal and nuclear power stations, electrical and heavy machinery industries, and important defence industries involving the production of aircraft, tanks, guns, ships, etc. More than 100 radiography cameras, most of them containing 5-20 C of ^{192}Ir and some up to 10 C of ^{60}Co obtained from Trombay, are in regular use. The demand for cameras is expected to rise steeply in the coming years, and the future developmental effort will be in the design of improved models of lighter weight, of cameras taking higher curiages of ^{60}Co and ^{192}Ir , and of new cameras taking isotopes like ^{170}Tm . In its efforts to promote and actively encourage the spread of inspection using isotopes, the Isotope Division also maintains a small service group to carry out mainly non-routine and specialized radiography jobs and those requiring guaranteed quality. The Division also assists the Indian Standards Institution in evolving standard codes of practice in radiography.

However, achievements to date are modest compared to the achievements in other countries. The economic benefits reported in some of the advanced countries by the use of isotope radiography are truly enormous. In UK, the figures for 1961 indicate that about 700 radiography cameras were in use, effecting an estimated saving of about

\$ 3 million. In USA, the savings in 1963 were estimated at about \$ 7 million and in the USSR, where 3000-4000 cameras are in use, the savings are estimated to be about \$ 22 million¹. Direct savings result from lower capital cost for alternative equipment for inspection; savings in scrap and increased productivity; and savings in labour. Besides this, large intangible savings are effected by increased safety and fewer complaints. Important applications like welding control of steam boilers and pipelines and ship plates have helped in decreasing hazards to life and property and have shown the importance of isotope technology in a modern industrial society.

Tracer Techniques

Radioisotope tracer techniques have found a wide variety of applications in metallurgical processes and research. The ease with which various materials and phases can be labelled with radio-tracers, the extreme sensitivity with which the radiations can be measured, and the valuable knowledge and benefits obtainable have made radioisotopes an indispensable tool for such investigations. Notwithstanding all this, very few metallurgical institutions and industries in this country make frequent use of tracer techniques.

Apart from the BARC and the Indian Institute of Technology, Bombay, radioisotopes are employed as tracers for metallurgical investigations only at the Indian Institute of Petroleum, Dehra Dun. Radioisotope work in these institutions is mainly directed towards diffusion and metal wear studies. In the industrial field, it is only at Bhilai that tracer techniques are employed for studying the wear of refractory linings, the mixing of molten metal and in certain other processes. Tracer laboratories are in the process of being set up at the National Metallurgical Laboratories and the Tata Iron & Steel Co in Jamshedpur and at the Indian Institute of Science, Bangalore.

A few typical instances of how valuable results can be obtained by using radioisotopes as tracers can be cited. By the addition of radioactive tracers to a product, it is possible to follow it through several steps in the production line and afterwards confirm its origin by a simple activity measurement. This has practical use in the identification of special qualities. One interesting example is the tagging of steel carried out in Sweden. It was required that two steel works, which delivered tubes for heat exchangers to a nuclear reactor, should certify that every tube at delivery had the approved composition. Analysis of the highly alloyed steel was very time consuming. It was, therefore, preferred to add a small quantity (<10 mC/ton) of a radioisotope with medium life and suitable metallurgical

behaviour. ^{182}Ta was chosen as tracer, the amount added not affecting the quality of the steel. Later, one of the works received an order for tubes with a different composition, for which a similar identification demand was prescribed. It was tagged with ^{192}Ir . The final products, tubes and tubing details were measured in the second case with a scintillation counter with variable discriminator level. When this was set for measuring pulses corresponding to γ -energies above 0.65 MeV, only the radiation from ^{182}Ta was recorded. With the discriminator set to accept γ -energies above 0.10 MeV, both ^{192}Ir and ^{182}Ta tagged tubes showed activity by which they could be distinguished from untagged material. The usefulness of the tagging method was clearly demonstrated for the identification of two different qualities of steel².

In other cases, tagging of steel baths has been performed, for instance, to avoid having experimental heats being treated and delivered as standard qualities by mistake. For this purpose, various short-lived nuclides, such as ^{64}Cu , ^{198}Au or ^{187}W have been used³.

Metal Wear

Tracer techniques have been widely employed to study metal wear in order to improve the performance of engine parts in automobiles, tractors, ships and aircrafts. Owing to their very high sensitivity, radiotracers allow the detection of extremely small values of wear and considerably reduce the time of investigation. The conventional technique involves irradiation of the engine part under investigation in a reactor. This makes the whole component radioactive and the activity is then traced in the lubricant or metal powder produced.

A recent development, the differential radioactive method, has increased the scope of the tracer technique by obviating the high radioactivity of the component which used to be a major drawback of the technique. The method enables the study of metal wear after reducing the radioactivity by a factor of 1000 or more, making it possible to carry out investigations without any radiation protection. Components are irradiated in an accelerator by a thin beam of charged particles, and a small spot of relatively low radioactivity is activated on the part to be tested. The part is then mounted directly on the running mechanism, and the radiometric devices record the decrease in the activity of the surface layer caused by wear, depending on the actual operating conditions. This novel technique for continuous or periodic control of wear under working conditions without stopping or dismantling the machine is now being widely used in the engineering industry in USSR⁴.

Studies with tractor engines in relation to quantities and abrasive qualities of dust and the rate of wear on piston rings have shown that dust particles of average diameter 0.1-0.2 μ caused the maximum wear. The life of CSEPEL engines of Budapest buses has been doubled by the introduction of filters, chosen after investigations with radioactive tracers. Changes in the concentration and nature of dust in an engine could lead to variation between

1 and 10000 in its life. Wear was found to be practically independent of the loading⁵.

Radiorelease Technique

Considerable improvements in labelling techniques have taken place in recent years. Tagging of a variety of materials, without regard to their chemical or physical form, may be accomplished by incorporating ^{85}Kr into the solid substance to be labelled⁶. This is performed by bombardment of the material with ionized krypton gas or by diffusion of the gas under high temperature and pressure.

The versatility of ^{85}Kr as a tracer lies in the fact that almost any material may be labelled with it. The degree to which absorption of the gas occurs is a function of the physical structure of the solid material. Thus while pyrolytic graphite has been labelled with ^{85}Kr up to 1 C/g, for steel samples, the specific activities obtainable are far less, and correspond to only a few microcuries per gram as the absorption of gas here occurs only within a few microns of the surface. Any process, chemical or mechanical, which disturbs the surface, causes a proportional loss of krypton and, consequently, of its radioactivity. This technique of labelling has rendered possible applications like the measurement of the erosion of turbine blades, the wear of bearings and the kinetic properties of the refractory metals at high temperatures⁷.

Non-active Tracer Method

Besides the normal tracer techniques utilizing the addition of radioactive substances, the isotope technology provides another tracing method. This is based on the addition of a non-radioactive substance which can be determined subsequently by activation analysis. For such 'non-active tracers', elements are chosen which can be activated giving a high yield of radionuclides with suitable half-lives and radiation properties. The sensitivity of the activation analysis makes this method superior to conventional combinations of tracer substances and analytical techniques, but it is much more laborious.

The production of steel with the lowest possible content of non-metallic inclusions is an important and urgent task of modern metallurgy. Tracer and non-active tracer techniques have been used to determine the source from where these inclusions penetrate into the liquid steel during smelting and pouring. For this purpose, the lining of steel melting furnaces and ladles as well as slag or any other suspected source is labelled with ^{45}Ca mixed as oxide or carbonate in the lining material. The subsequent separation of inclusions from the metal produced, measurement of their radioactivity and autoradiography of the ingots give a direct quantitative idea of the steel contamination sources.

As non-active tracers, La_2O_3 and Dy_2O_3 have been used, as La and Dy have high activation cross-sections. Studies by Russian investigators have shown that the exogenic inclusions are of minor importance in killed steel, while they are mainly responsible for the inclusions in rimming steel.

Non-active tracer technology has certain advantages. On the one hand, it is advantageous in very large-scale investigations where it would be complicated to handle the large amount of radioactivity needed; and, on the other, in investigations where a tracer is released in nature and the safety of the public could not, with absolute certainty, be guaranteed.

Radioisotope Instruments

Radioisotope instruments like thickness, density, concentration and level gauges have enabled modern technology to derive great economic benefits. The principle of these gauges is well known and depends on the interaction of the radiation emitted with the materials being controlled, i.e. attenuation in transmission gauges or back-scattering of the radiation in back-scatter gauges. These radioisotope instruments are made up of (i) a radioactive source — radiation detector system, the source and detector being mounted with respect to the medium controlled; (ii) suitable electronics with a recording unit; and (iii) a controlling or actuating unit.

The economic benefits derived from the use of radioisotope gauges result from a number of factors. Their use increases plant efficiency, where the conventional process gauging is a bottleneck, decreases the manpower necessary for monitoring the process and accelerates analytical work. Savings in raw material and scrap are important, particularly where the raw material cost is a significant fraction of the final product cost. Finally, increased quality, the most quoted advantage of nuclear gauging, makes the product more competitive.

The total number of nuclear gauges in all types of industries in India is estimated to be less than 200, as against an estimate of about 4000 gauges in UK and about 10,000 in USA. The annual saving from isotope gauging is estimated at about \$ 50 million in USA in 1963 and at about \$ 100 million in the USSR in 1961 (ref 1).

It is estimated that in India about 120 nuclear gauges have been installed in different metallurgical industries, of which nearly 100 are used for level detection in sintering plants and hot metal cupolas, the largest single user being the Bhilai steel plant. This includes about 50 level gauges. However, in the field of thickness gauging, while these gauges are considered to be efficient and economical in other countries, the benefits are yet to be recognized in India. The thickness of even hot rolled steel in the range 4-100 mm is now being controlled by ZITRA thickness gauges in the USSR. In this gauge the radiation source is ^{137}Cs of 1 C strength, the detector being a scintillation counter with NaI crystal. The detector set-up is water-cooled. Precision of the order of $\pm 0.5\%$ is claimed⁸. The main users of thickness gauges are Messrs India Foils Ltd of Calcutta, where 10 beta ray thickness gauges, using ^{204}Tl sources, measure the thickness of aluminium foils during the rolling process. These gauges cover the range 0.0002-0.02 in. with an accuracy of 1%. Besides these, it is understood that the Rourkela steel plant is also planning to install thickness gauges for steel

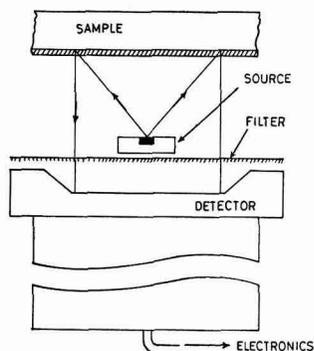


Fig. 1 — Schematic representation of the sample-source-detector assembly in an X-ray fluorescence gauge

sheets during rolling and back-scatter gauges for measuring the thickness of zinc coating.

A remarkable development in recent years has been the growth of radioisotope instruments for analysis, both in the laboratory and on-stream. They are now often used as an alternative to X-ray machines, because they are smaller, cheaper and more reliable. For instance, in isotopic X-ray fluorescence analysis, isotopes giving low energy γ - and X-radiation can be used to excite characteristic X-rays of the element being sought in the sample.

X-ray fluorescence gauges (Fig. 1) are used for measuring the thickness of metallic coatings like tin on steel, either by measuring directly the X-rays excited in the coating or by measuring the absorption (in the coating) of the X-rays excited in the base material. In the case of Sn on steel, thicknesses down to 0.1μ can be measured. For gauging Zn on steel, suitable filters are used for energy selection.

In UK 'dragonite' gauges are used in the Steel Company of Wales, Port Talbot, for measuring the thickness of electroplated Zn coating on steel, using a $^3\text{H}/\text{Zr}$ source to excite the characteristic Fe X-rays in the steel base, which are attenuated by the Zn coating before detection by a scintillation counter. Zn thickness in the range 10^{-5} - 10^{-3} in. can be measured with 95% confidence to an accuracy of $\pm 5\%$ in 10 sec⁹. Apart from those required for important on-line applications like Sn on steel and Zn on steel, most of the instruments have been designed to be portable to enable wide use in plant, laboratory and the field. Other applications are the continuous analysis of process streams such as mineral slurries, and the field analysis of metalliferous ores. The isotopes used often for such applications are ^{109}Cd (1.3 yr), ^{57}Co (270 days), ^{241}Am (470 yr), and $^3\text{H}/\text{Zr}$ (12.3 yr). There is not a single X-ray fluorescence gauge in this country, but one will soon be installed at BARC to demonstrate to the industry the wide utility of such gauges and to explore the possibility of developing them indigenously.

Analytical Techniques

There has been considerable progress in this respect on account of the developments in radioisotope methodology and their impact on metallurgy has been wide and useful. The technique of isotope dilution is well known and the principle is simple. A known quantity of the actual substance, labelled with a radioactive nuclide at a known specific activity, is added to the sample. After equilibrium is reached, a sample of the product is removed, weighed and its radioactivity and consequently the specific activity determined.

Isotope dilution techniques have been used for the assay of various constituents like Mo, W, Nb and Ta in steel¹⁰. In the determination of phosphorus, ³²P is added to the melt in a suitable chemical form. After it has mixed uniformly with all the phosphorus present, the phosphorus content of the steel and slag can be followed easily during the refining process. In one sample, the phosphorus content is determined chemically after the activity has been measured. The activity of the steel and slag samples can then be used as a measure of the phosphorus content, using the chemically analysed sample as a standard. The measurement can be performed on site in the steel plant rapidly. It is thus possible to determine the phosphorus content of the slag, which is of value when the slag is to be used as a fertilizer¹¹.

Sulphur in steel and slag can also be determined in the same way, but the method is less attractive on account of the soft beta radiation of sulphur. Isotope dilution techniques with tritium have also been used for hydrogen determination in aluminium^{12,13}.

Using dilution techniques, it is possible to weigh easily the amount of slag in open hearth furnaces. A known amount of a radioactive substance is added to the slag phase, where it rapidly dissolves in the molten slag. It then remains quantitatively in this phase. The concentration of the added substance is determined in samples and, from the result, the total amount of slag is calculated¹⁴.

Dilution analysis can be performed with any substance that fulfils the metallurgical requirements. The oxides of rare earth metals are used as radioactive tracers. The choice of the rare earths for use as tracers is governed by their resistance to reduction — no risk of losses by transfer into the metallic phase exists. Radiotracer techniques using active ¹⁴⁰La and non-active tracer techniques involving activation analysis are used. The amount of molten metal in an electric furnace for the production of highly alloyed steels is measured in Sweden on a routine basis with short-lived ⁶⁵Ni. The calculation for adding expensive alloying compounds can be made with this method more accurately than is otherwise possible. The amount of molten aluminium in an electrolytic cell has been determined in the same way with ¹⁹⁸Au (ref 15). It is also possible to use inactive gold and carry out the determination by subsequent activation analysis¹⁶.

Activation Analysis Techniques

A significant advance in metallurgy in recent years has been the production of ultra-pure metals

by means of zone refining techniques. Metals used in semiconductors must be free from certain impurities and must contain accurately known quantities of certain other elements. The influence of trace impurities or of alloying elements upon the properties of metals is of great theoretical and practical importance and radioactivation analysis has provided information in this direction which could not be obtained by any other method.

The principle of activation analysis is quite simple. The material under reference is subjected to neutron bombardment in a nuclear reactor; the mixture of radioisotopes produced is analysed by gamma spectrometry directly or after suitable and rapid radiochemical separation. With activation analysis, it is possible to determine concentrations of 10⁻⁷ (ie 0.1 ppm) of rare earths, Sb, Mo and Zn and 10⁻⁹ (ie 0.001 ppm) of Cu and As in germanium.

A comparatively recent development in this field is the portable neutron generator, in which a stream of monoenergetic 14 MeV neutrons is produced by bombarding the radioisotope tritium (³H) with deuterium (²H) ions. Such neutron generators have immense potentialities in the metallurgical industry and in particular in metallurgical analyses. Normally 3-5 C of tritium are adsorbed on zirconium targets and these are then bombarded with deuterium ions accelerated to 150-400 kV. Fluxes of 2.5 × 10¹¹ neutrons/sec are obtainable. (The cost of such a generator would work out to about Rs 3 lakhs.)

Oxygen in steel is an important contaminant that must be determined rapidly and accurately during production. Using neutrons from portable neutron generators, oxygen can be activated to form the radioisotope ¹⁶N whose determination is then carried out rapidly by gamma spectrometry. With neutron generators, large samples (up to 10 g) can be accurately analysed in half the time needed for much smaller samples with other techniques. The system is non-destructive and the same sample can be re-run if necessary.

At the steel factory at Cockerill-Ougree-Providence near Liège in Belgium, producing about 2 million tons of steel per year, the oxygen content in steel is determined by such techniques. It is claimed that the analysis of each sample takes only 2 min. One hundred and fifty samples, each in triplicate, ie about 450 samples, are analysed per day. At 20 ppm levels, 5-7% precision is obtained, while at 1 ppm level, 20-30% precision is claimed [Personal communications, Messrs Sames, Grenoble, France].

Another field where neutron generators are finding increasing application is neutron radiography. The higher transmission of neutrons through thick steel, lead, uranium and other heavy metals makes this technique preferable to the conventional X-ray and gamma radiography techniques in specific applications. The technique of neutron radiography is basically the same as gamma radiography except that, since neutrons have little effect on photographic emulsions, indium foils placed close to the emulsions act as image converters. The radiations from indium are picked up by the emulsion

which is then developed [Personal communications, Messrs Sames, Grenoble, France].

Summary

Radioisotopes are being used at present in India in some metallurgical industries, mainly for quality control by non-destructive isotope radiography and for gauging either the thickness of metal sheets or the level of materials in closed vessels. Besides these, tracer investigations of metal wear, wear of refractory linings, mixing of molten metals and diffusion phenomena have been carried out in some of the institutions. The present position in this regard is surveyed and the potential applications indicated, based on the experience in other countries, on the use of radioisotopes in metallurgical research and industry. These include recent developments like radioisotope tagging of steel, wear studies in machines under actual working conditions using differential radioactive tracer methods, radiorelease techniques using 'kryptonated' solids, slag weight determinations by isotope dilution, measurement and analysis of thin coatings by radioisotope X-ray fluorescence gauges, routine analysis of oxygen content at low levels by fast neutron activation analysis using portable neutron generators and neutron radiography.

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Some Aspects of Carbohydrate Metabolism in Haemoflagellates

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ANTONY VON LEENWENHOCK, who for the first time reported the occurrence of free-living fresh water protozoa, is commonly recognized as the father of protozoology. Through the painstaking efforts of various investigators over the last 60 years, it has now been established that some 25 species of protozoa occur in man. More recently, interest has shifted towards studies on intermediary metabolism, with emphasis on the enzymatic relationship existing between different classes of protozoa and many other haemoflagellates.

The main difficulty in pursuing the biochemistry of haemoflagellates is that only a few of the haemoflagellates can be cultured in well-defined media outside their hosts. Since successful metabolic studies can be carried out only on species obtained without contamination, such investigations have been confined largely to trypanosomes and the malarial parasites. A few haemoflagellates have been grown in pure cultural form and some information regarding their growth and nutritional requirements has been obtained. The earliest observation regarding the requirement of blood in haemoflagellates was by Novy and McNeal¹ who used blood-agar medium for the growth of *Trypanosome lewisi* and *T. brucei*. Subsequently, Nicolle² grew *Leishmania tropica* and *L. donovani* in the same medium. Adler³ and Senekjic and Lewis⁴ claimed the growth of various *Leishmania* and trypanosomes on peptone medium enriched only with rabbit serum. But different groups of workers have established that no *Leishmania* or trypanosomes can grow in a completely haemoglobin-free medium⁵⁻⁸. The requirement of haemin in the haemoflagellates seems to be due to their inability to synthesize it^{9,10}. It seems that the Trypanosomidae have a tendency towards loss of synthetic ability due to their parasitic mode of life and this has not affected their survival.

Utilization of Different Sugars in Haemoflagellates

All defined media of trypanosomatids and most undefined media contain a sugar. Glucose is most frequently used, but often sucrose at a proper pH (7.6-8.1) can replace glucose. Both glucose and sucrose are utilized for growth by all trypanosomatids growing in defined media at room temperature, but differences in utilization occur with rise in incubation temperature¹¹. The glucose consumption by the blood stream trypanosomes has been determined in a number of organisms, viz *T. lewisi*¹², *T. evansi*¹³, *T. brucei*^{14,15}, *T. gambiense*¹⁶ and with 11 species of African trypanosomes¹⁷. Glucose consumption by the cultural forms of Trypanosomidae has been demonstrated with *Leishmania* and *T. gambiense*¹⁸ and *T. rhodesiense*¹⁹.

The intensity of sugar metabolism is higher in the *congolense*, *evansi* and *brucei* groups than in the *lewisi* group^{12-14,16}. Reiner and Paton²⁰ and Hawking²¹ found that African pathogenic trypanosomes consume sugar corresponding to 15-30% of their wet weight in 1 hr. There exists a definite difference in carbohydrate requirement between the blood stream form of African pathogenic trypanosomes and their cultural forms. Schern and Bozzalo²² found a small amount of stored glycogen in African trypanosomes, but Krijgman²³ and Moulder²⁴ found little indication of stored polysaccharide.

Apart from glucose, fructose and mannose have been shown to be well utilized by various trypanosomatidases²⁴⁻²⁸. Pentoses and polysaccharides are also utilized in some isolated cases. In many cases, maltose and saccharose are also utilized. Ryley²⁹ has shown that the blood stream form of *T. rhodesiense* can utilize glycerol, glucose, fructose and mannose, whereas the cultural forms can utilize some tricarboxylic acid cycle intermediates, especially under acid conditions. Apart from glucose, fructose and mannose, glutamine can be well utilized by *T. lewisi*³⁰, whereas glutamate, glycerol and asparagine were shown to be utilized to a lesser degree.

It has been observed that in *L. donovani* mannose is utilized at a similar rate as glucose³¹. The presence of mannose phosphorylating enzyme and phosphomannose isomerase has been observed in the cell-free extract of this organism. The utilization of ribose and galactose in this organism is low³². The organism *L. donovani* has been shown to utilize sucrose well and the presence of a sucrose splitting enzyme has been demonstrated³³. Studies by Chang²⁸ on four haemoflagellates (*L. donovani*, *L. brasiliensis*, *L. tropica* and *T. cruzi*) have shown that the organisms can oxidize glucose and levulose, but not maltose and lactose.

End Products of Fermentation in Haemoflagellates

The carbohydrate metabolism for all protozoa is characterized by incomplete oxidations. This is well marked in the case of African pathogenic trypanosomes. It is a bit surprising that the Trypanosomatidae, though living in well-oxygenated surroundings, are characterized by incomplete oxidation. Hence, von Brand *et al*³⁴ have classified the Trypanosomatidae among the aerobic fermenters from the standpoint of metabolic types. Some of the trypanosomes and Leishmanias, in which the common Krebs cycle enzymes are lacking, should accumulate the end products of glycolytic cycle. In other parasites, however, such explanation does not always hold good. Thus, in the incubates of malarial parasites, lactic acid has been

shown to be present, though they possess all the enzymes of the Krebs cycle³⁵. Succinic dehydrogenase³⁶ and another succinate splitting enzyme³⁷ are present in *T. cruzi*, yet in the incubates of the organism succinic acid accumulates. In the intact organisms either the enzymes responsible for the formation of succinic acid are more active than those related to its utilization or by some intracellular regulatory mechanism the latter are prevented from working at their maximum capacity. Excretion of organic metabolites is a highly uneconomic process, since a large part of the potential energy of the parent substance remains unutilized by the parasite. This also may be the reason for the trypanosomes needing large amounts of carbohydrates for their growth.

Pyruvate formation—Pyruvic acid is the most important aerobic metabolic end product of blood stream forms of pathogenic African trypanosomes^{38,39}. Anaerobically, the excretion of pyruvate is reduced and part of the glucose carbon is accounted for by glycerol.

Chang²⁸ made an exhaustive study on the three species of *Leishmania* and *T. cruzi* and found that appreciable amounts of succinic, pyruvic and lactic acids and carbon dioxide are produced by *L. donovani*. Fulton and Joyner⁴⁰ could not demonstrate the formation of pyruvic acid in this organism. But in a subsequent study, Crowther *et al*⁴¹ demonstrated the formation of pyruvic acid along with succinic and acetic acids in the culture medium. The formation of radioactive pyruvate³² has been demonstrated when cells of *L. donovani* are incubated in the presence of uniformly labelled glucose-¹⁴C. It has also been observed that the cell homogenate of *L. donovani* produces lactic acid anaerobically. The formation of lactic acid is considerably diminished in the presence of iodoacetate, but not in the presence of fluoride⁴².

In *T. equiperdum*⁴³, *T. hippicum*⁴⁴ and *T. evansi*⁴⁵, glucose dissimilation proceeds up to the pyruvate stage, while with *T. cruzi*, *T. lewisi*, *T. congolense*, *T. vivax* and *S. oncopelti*, glucose degradation proceeds beyond the pyruvate stage. This distinction has been clearly shown by Ryley¹⁷, who studied the end products of both aerobic and anaerobic metabolism of glucose in 11 species of trypanosomes. In six of the species, pyruvic acid and glycerol were shown to be the end products and the respiratory quotients in all these cases were very small. Working with *T. equiperdum*, Reiner *et al*⁴³ also made a similar finding. Pyruvic acid was also shown to be produced in *T. evansi*⁴⁵.

Succinate formation—Succinic acid is the most commonly produced acid by the haemoflagellates during glucose catabolism; its production is often much more pronounced under anaerobic conditions^{17,29,46}. Agosin and von Brand³⁶ could not demonstrate the formation of succinic acid under aerobic condition. Succinic acid was shown to be produced in *T. rhodesiense* along with lactic, formic and acetic acids⁴⁷. *T. lewisi* also produces succinic, pyruvic and lactic acids, besides several one- and two-carbon fragments^{43,48}.

The formation of succinic acid aerobically may be via acetyl CoA condensation and is an oxidative

process⁴⁹. But succinate cannot originate under anaerobic conditions by the reactions of that cycle as they proceed under aerobiosis. Various pathways leading to anaerobic succinate formation have been suggested by different workers^{37,46,49,50}. Seidman and Entner⁵⁰ suggested the anaerobic formation of succinate and pyruvate from malate in the presence of ADP and inorganic phosphate. Succinate formation has been noted upon anaerobic incubation of a particulate system from ascaris muscle with fumarate and reduced DPN⁵¹. This indicates the reversal of succinoxidase system, a feature possibly representing an adaptation to the worm's anaerobic life.

Most of the available evidence for the anaerobic formation of succinate in haemoflagellates is by reversal of some reactions of the Krebs cycle along with some auxiliary reaction, such as the formation of oxaloacetic acid. Oxaloacetic acid is generally formed by carbon dioxide fixation. The importance of transamination for the generation of oxaloacetic acid cannot be assessed properly in the organisms at present. First evidence for the fixation of carbon dioxide was provided by Searle and Reiner⁴⁸; it was confirmed by the manometric experiments of Ryley¹⁷. In the blood stream form of *T. rhodesiense*, the fixation of carbon dioxide was demonstrated by the use of radioactive bicarbonate. By the use of variously labelled glucose and bicarbonate, Bowman *et al*⁴⁶ demonstrated that both aerobic and anaerobic formation of succinate was due to carbon dioxide fixation to pyruvate. The relative importance and distribution of the different carbon dioxide fixing enzymes, i.e. pyruvate carboxylase, phosphoenol pyruvate carboxylase, malic enzyme and other related enzymes, are not properly known in this group. However, Raw⁵² demonstrated the presence of malic enzyme in *T. cruzi*. By the use of radioactive glucose and radioactive bicarbonate, it has also been found that in *L. donovani* carbon dioxide fixation takes place during succinate formation. The presence of malic enzyme has been found in the cell-free extract of this organism, but the presence of phosphoenol pyruvic carboxylase and phosphoenol carboxy transphosphorylase could not be detected³².

Glycolysis in Haemoflagellates

Studies made so far on the protozoal metabolism have proceeded along two different lines. The first concerns with studies on the overall metabolism of the entire organisms and the second involves investigations on the intermediary metabolism requiring the preparation of cell-free extract. From such studies it appears that though the anaerobic sugar catabolism by the trypanosomes and other protozoa follows essentially the same pattern as found in the majority of free-living organism, still it has some differences and distinct characteristics of its own.

There is evidence for the existence of modified glycolysis in trypanosomes. In normal glycolysis, pyruvic acid is reduced to lactic acid in the presence of reduced DPN and thus maintains the supply of DPN. But in *T. brucei*, glyceraldehyde phosphate accepts the hydrogen of reduced DPN, a

process leading to glycerol formation. The presence of the required enzymes, glycerophosphate dehydrogenase and phosphatase, has been demonstrated in trypanosomes^{44,53,54}.

Harvey⁴⁴ has shown that in *T. hippicum*, glycolysis follows the classical EMP pathway. Fractionation studies on acid-soluble phosphorus revealed that the main phosphorylated compounds were glucose-6-phosphate, triose phosphate and glycerophosphates. Specific enzymatic analysis confirmed the presence of hexokinase, aldolase, triose phosphate dehydrogenase and glycerophosphate dehydrogenase.

Searle and Reiner^{48,51} have demonstrated the importance of carbon dioxide as an activator of anaerobic glycolysis in trypanosomes. These investigations have shown clearly the diversity in the end products of glucose metabolism in various species of trypanosomes, but have provided little indication of the intermediary processes by which these products are formed. Chen and Geilling⁵⁵ have shown that lysed trypanosomes transform glucose to fructose 1,6-diphosphate and triose phosphate and oxidize phosphoglyceraldehyde to phosphoglyceric acid. Chen⁵⁶, working with a lysed preparation of *T. equiperdum*, showed the presence of the enzymes hexokinase, triose phosphate dehydrogenase and adenosinetriphosphatase. The presence of an active adenosinetriphosphatase may at least in part explain the failure of Christophers and Fulton⁵⁷ to demonstrate the occurrence of glycolysis with lysed *T. rhodesiense*. This reason may also be fitting with the negative results of Moulder¹² with lysed *T. lewisi*.

In *T. lewisi*, glucose breakdown follows a different pattern from that in *T. evansi* or *T. equiperdum*. Reiner *et al*⁴⁸ have shown that in *T. lewisi*, glucose is anaerobically broken down into one molecule of succinic acid and presumably one molecule of glycol. Glycol is probably decomposed further to acetaldehyde, which undergoes dismutation and forms acetic acid and ethyl alcohol. The same reactions take place under aerobic conditions; in addition, acetaldehyde is oxidized to formic acid, carbon dioxide and water. It is surprising that no indication of phosphorylation was found in this glucose breakdown. It is also obvious that the evidence for the existence of a complete glycolytic sequence is in many cases incomplete, only one or a few of the required enzymes having been demonstrated. In some cases, a few phosphorylated intermediates of glycolysis have been demonstrated. Some of the glycolytic enzymes present in this group of protozoa have been listed in Table 1.

Among the glycolytic enzymes, aldolase has been studied in greater detail. The aldolase of *T. cruzi* and *T. lewisi* appears not to be metal-activated and is not inhibited by dipyriddy^{58,59}. The aldolase of *L. donovani* also is not inhibited by dipyriddy or EDTA³². It has also been reported that in *L. donovani* a single enzyme, hexokinase, phosphorylates glucose, fructose, mannose and glucosamine⁶⁰.

Finally, studies with radioactive materials and subsequent elucidation of radioactivity in the metabolic products have given some insight of the related metabolic pathways. In *T. rhodesiense*,

TABLE 1—SOME KEY ENZYMES OF GLYCOLYSIS IN HAEMOFLAGELLATES

| Enzyme | Organisms |
|--------------------------------|--|
| Hexokinase | <i>L. donovani</i> , <i>T. cruzi</i> , <i>T. hippicum</i> , <i>T. rhodesiense</i> |
| Phosphohexose-isomerase | <i>L. donovani</i> , <i>T. cruzi</i> |
| Phosphohexokinase | <i>L. donovani</i> , <i>T. lewisi</i> |
| Aldolase | <i>L. donovani</i> , <i>T. cruzi</i> , <i>T. equiperdum</i> , <i>T. rhodesiense</i> , <i>T. hippicum</i> |
| Phosphotriose-dehydrogenase | <i>T. rhodesiense</i> , <i>T. cruzi</i> , <i>T. equiperdum</i> , <i>T. hippicum</i> |
| Lactic dehydrogenase | <i>T. cruzi</i> |
| Glycerophosphate dehydrogenase | <i>T. hippicum</i> , <i>T. rhodesiense</i> |

anaerobic incubation with uniformly labelled glucose leads to the formation of pyruvate and glycerol, both labelled to the same extent, while with glucose-1-¹⁴C under anaerobic condition, most of the radioactivity appears in glycerol³⁸. Using ¹⁴C-glucose, Shaw *et al* have demonstrated the formation of radioactive hexose, mono- and diphosphates in *L. tropica*, *T. cruzi* and *T. rhodesiense*⁶¹.

Pentose Phosphate Cycle

Very little work has been done to find out whether this pathway of glucose metabolism has any significant role in these organisms. Raw³² could not demonstrate the utilization of ribose-5-phosphate in *T. cruzi* and hence speculated the absence of this pathway in this organism. However, the utilization of 6-phosphogluconate was observed. But Ryley²⁹ has shown the absence of such faculty in *T. cruzi* and *T. rhodesiense*. In the case of *L. donovani*, the formation of ketohexose, ketopentose and sedoheptulose has been demonstrated, indicating the presence of such a pathway in this organism³².

Tricarboxylic Acid Cycle

The main function of the tricarboxylic acid cycle is complete oxidation of pyruvic acid to carbon dioxide and water by a series of cyclic reactions. The cycle involves the condensation of a two-carbon unit with oxaloacetic acid to form dicarboxylic acids, with ultimate regeneration of oxaloacetic acid. As mentioned earlier, this group of protozoa excrete large amounts of succinic acid and synthesize oxaloacetic acid for the proper running of the cycle. This may be the reason for the synthesis of oxaloacetic acid by carbon dioxide fixation, which may be an auxiliary process going on along with the TCA cycle.

From the available evidence it cannot be concluded whether a typical tricarboxylic acid cycle is present in the haemoflagellates. The presence of different Krebs cycle enzymes has been demonstrated in *L. tropica*⁶², *T. cruzi*, *T. rhodesiense*²⁹ and *T. lewisi*³⁰.

Some of the key enzymes of Krebs cycle present in different organisms are listed in Table 2. Among the various Krebs cycle enzymes, isocitric dehydrogenase and succinic dehydrogenase have been studied

TABLE 2 — SOME ENZYMES OF THE KREBS CYCLE IN HAEMOFLAGELLATES

| Enzyme | Organisms |
|-------------------------|--|
| Aconitase | <i>T. cruzi</i> , <i>T. rhodesiense</i> |
| Isocitric dehydrogenase | <i>T. cruzi</i> , <i>T. lewisi</i> , <i>T. rhodesiense</i> |
| Fumarase | <i>T. cruzi</i> , <i>T. rhodesiense</i> |
| Malic dehydrogenase | <i>T. cruzi</i> , <i>L. donovani</i> |

in great detail. The purified isocitric dehydrogenase of *T. cruzi* has been found to be TPN-linked and to require Mn⁺⁺ for its activity⁶³. The succinic dehydrogenase of *T. cruzi* is essentially particle-bound and can function in the presence of cresyl blue, but not in the presence of cytochrome *c*. The enzyme could be inhibited by malonate, even at a very low concentration.

Apart from enzymatic studies, the detection of Krebs cycle intermediates and their utilization have been taken as evidence for the elucidation of the cycle in different protozoa. In the cultural form of *T. cruzi*, almost all the intermediates of TCA cycle can be utilized⁶². Similarly, Ryley²⁹ has demonstrated the utilization of citric acid, α -ketoglutarate, and succinate in *T. rhodesiense*. In *T. hippicum*, the utilization of most of the Krebs cycle intermediates could not be demonstrated⁴⁴. It has been observed that in *L. brasiliensis* and in *L. tropica*, citrate, α -ketoglutarate and succinate can be utilized⁶³.

Evidently, the demonstration of a few enzymes of this cycle or utilization of some of the intermediary substrates cannot be taken as conclusive evidence for the presence of a complete cycle. Enzymes of the Krebs cycle may also be used for the synthesis of amino acids or for some other processes having no connection with the terminal respiration. Similarly, it may be equally premature to assume the absence of the cycle when the presence of a few of the enzymes could not be demonstrated or some intermediates were not utilized. As it has often been found, difference in technique may lead to erroneous conclusions. In the case of utilization of substrates the question of permeability is of paramount importance.

In conclusion, it can be stated that a functional Krebs cycle has not yet been demonstrated in any member of this group of protozoa, with the same definiteness as in mammalian or bacterial systems. The cycle seems to be operated in cultural forms of most trypanosomids. It is expected to be absent in the blood stream forms of trypanosomes of *brucei* and *lewisi* groups.

Summary

The work done to date on carbohydrate metabolism in the parasitic protozoa, haemoflagellates, has been reviewed. The haemoflagellates are well marked for incomplete metabolism and have been classified as aerobic fermenter from metabolic standpoint. Some scattered studies have been done on Krebs cycle enzymes, but the presence of this cycle in this group has not yet been fully established.

Hardly any work has been done so far on shunt pathway. Broad indications are provided for future studies in this field.

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Steroid Saponins of Some Indian Plants*

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THE research activity on steroid saponins was intensified after the discovery of the beneficial effect of cortisone by Kendall and his coworkers¹. Particular mention may be made of Marker *et al*²⁻⁴ for their pioneering efforts in this field. In recent years considerable attention has been focused on the conversion of steroid saponins into useful steroid hormones. The work carried out in India on survey and isolation of steroid saponins by the authors has been reviewed in this communication.

With the object of exploring the possibility of producing steroid hormones in the country, a survey of the Indian *Dioscorea* yams (Fam: Dioscoreaceae), commonly known as jungle roots, was undertaken around 1950. The yams, collected mostly through the courtesy of forest officers, were investigated⁵⁻¹². The yams of *Dioscorea prazeri* Pr. and Burk. (*kukur torul*) of Darjeeling area and *D. deltoidea* Wall. (*kins*) of Kashmir were found to be rich sources of diosgenin, a steroid saponin useful as a starting material for the preparation of a number of steroid hormones including cortisone. Using diosgenin, it was possible to prepare a number of steroid hormones, including progesterone, for the first time in India¹³. Laevulinic acid¹⁴ was obtained in considerable amounts as a useful byproduct¹⁵ during hydrolysis of *Dioscorea* saponin into diosgenin. It is formed obviously by the action of hot hydrochloric acid on the sugars of the saponin and the large quantities of starch present in the yams.

Dioscin was isolated by Tsukamoto and Ueno¹⁶ as the chief saponin of *Dioscorea tokoro* Makino. Later, *D. gracillima*^{17,18} yielded gracillin along with

dioscin. Barua *et al*¹⁹ isolated pure crystalline saponins from Indian *dioscoreas*, *D. prazeri* and *D. deltoidea*. The saponins on hydrolysis with 6% hydrochloric acid yielded diosgenin, glucose and rhamnose²⁰.

The saponin obtained from *D. prazeri*, mp 276-7° (decomp) $[\alpha]_D^{22} -90^\circ$ (pyridine) and that from *D. deltoidea*, mp 288-90° (decomp) $[\alpha]_D^{22} -95^\circ$ (pyridine) had different solubilities²¹ and appeared to be different from each other. The identities of these with dioscin¹⁶ and gracillin¹⁸ have not yet been established, although the saponins obtained by the authors yielded rhamnose at the end of the chain of sugars as in the cases of dioscin and gracillin.

Yams of *D. prazeri* var *glauca* also yielded a crystalline saponin which was found to be practically identical with that of *D. prazeri*.

In view of the presence of physiologically active alkaloid in the stems and leaves of *Cestrum parqui*^{22,23}, the investigation was extended to the *Cestrum* species in India. The leaves of *Cestrum nocturnum* (Fam: Solanaceae) (night jessamine or *hasna-hena*) have been found to be rich in saponin. On fractional solvent extraction they yielded a mixture of saponins from the alcoholic extract. The product after repeated crystallizations was still found to be a mixture, mp 260-69°, $[\alpha]_D^{22} -57^\circ$ (pyridine). It gave a beautiful purple colouration with concentrated sulphuric acid and on hydrolysis yielded a mixture of yuccagenin and tigogenin. These two were separated by chromatography over Brockmann alumina when tigogenin was obtained by elution with a mixture of benzene and ether, and yuccagenin by elution with absolute alcohol. Tigogenin and yuccagenin²⁴ thus obtained were purified by crystallization and their identity established by the melting points (207-8° and 242-3°C respectively), preparation of the derivatives, tigogenin acetate (mp 200-201°C), tigogenin benzoate (mp 228°C), tigogenone (mp 204°C), yuccagenin

*Paper presented at the Symposium on Glycosides and Saponins held in Calcutta during 2-4 April 1964.

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‡Present address: Department of Chemistry, Bethune College, Calcutta.

acetate (mp 178-9°), yuccagenin benzoate (mp 220-22°) and by comparison of infrared (IR) spectra.

The sugars produced by hydrolysis of the mixture were found to be xylose, glucose and galactose by paper partition chromatography using ethyl acetate-pyridine-water as the eluent. It may be quite likely that the saponin mixture consists of tigonin and yucconin.

The plant *Cestrum diurnum* (Fam: Solanaceae), a plant similar to *Cestrum nocturnum*, grows wild in abundance throughout the country. The leaves of the plant yielded, on fractional solvent extraction, ursolic acid²⁵ in the ether extract and a crystalline saponin, tigonin²⁶, in the alcoholic extract. On hydrolysis, the saponin yielded tigonin. The presence of xylose, glucose and galactose was established with the sugar part of the saponin by paper partition chromatography. The position of xylose in the chain of sugar units could be indicated by partial hydrolysis at a point farthest from the saponin residue. In other words, the saponin consisted of one tigonin molecule attached to xylose molecule through glucose and galactose molecules.

Pharmacological Studies

Pharmacological examinations of the purified crystalline saponins obtained from *D. deltoidea* (D), *D. prazeri* (P) and *D. prazeri* var *glauca* (G) were carried out at the Central Drug Research Institute, Lucknow (Mukerji, B., private communication). Saponins P and G, which are soluble in propylene glycol, produced a fall of blood pressure of about 45 and 25 mm of Hg respectively in anaesthetized cats when given intravenously in a dose of 0.5 mg/kg. This fall of blood pressure could be prevented by atropinization. Saponin D being insoluble in propylene glycol, its suspension in propylene glycol was used. There was no effect on blood pressure when it was given intravenously in the same dose. Further, these drugs had no action on isolated guinea-pig ileum.

It may be mentioned that the saponin from a Caucasian *Dioscorea* yam²⁷ (*D. caucasia*, Lipsky?) has been found to be useful in USSR in cases of atherosclerosis, particularly cerebral atherosclerosis. This may probably be explained as being due to the removal of the deposited cholesterol by complex formation with the saponin.

Pharmacological examination of the saponin from the leaves of *Cestrum nocturnum* showed²⁸ that the saponin possesses cardiac activity similar to that of ouabain.

Summary

The work done by the authors on survey and isolation of steroid saponins from *Dioscorea* and

Cestrum species, and pharmacological examination of the saponins have been reviewed.

Acknowledgement

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REVIEWS

THE PHILOSOPHY OF QUANTUM MECHANICS by D. T. Blokhintsev (D. Reidel Publishing Co, Dordrecht, Holland), 1968. Pp viii+132. Price Dfl 32

The present monograph is devoted to a critique of the philosophy of quantum mechanics. It is more concerned with theoretical physics rather than philosophy. Determinism which has been the basis of classical physics is illusionary, because the basic assumption that it is possible to determine uniquely the future state of a system from its initial data is based on an abstraction which does not include randomness. But the effects of randomness cannot be disregarded in the general case, because the uncertainty arising from the initial data increases with time and the prediction becomes meaningless after a lapse of a certain finite time. However small the random deviations in the initial data or random forces may be, their consequences always predominate after some time. This is in particular true for a system with large degrees of freedom. When a large number of random factors dominate, a new law 'the statistical regularity' emerges. According to Gibbs, a large number of identical and completely mutually isolated systems, each of which interacts weakly with a large macroscopic system at a set temperature (an ensemble), describe the thermodynamic behaviour of the system in terms of a canonical probability distribution, which gives a completely definite value for any thermodynamic measurement.

Quantum mechanics is essential to explain the zero-point energy of a system at absolute zero, as is shown by experiments on light scattering due to thermal motion at low temperatures and at absolute zero. Thus, a Gibbs ensemble spontaneously becomes a quantum ensemble at absolute zero. A very important characteristic feature of a quantum ensemble is Heisenberg's uncertainty principle. In fact, we can define the totality of systems in which the uncertainty principle holds as a quantum ensemble. Such an ensemble can be described in terms of a wave function ψ or a density matrix ρ . Using the Heisenberg relation or Bohr's principle of complementarity it can be shown that this wave function can be a function of either the coordinates or the momenta describing the system. Both ψ and ρ satisfy differential equations which express causality in quantum mechanics. The principle of superposition of states plays a fundamental part in quantum mechanics and the symmetry conditions for a system of identical particles can be conveniently incorporated in the theory. One cannot, therefore, consider quantum mechanics as a form of classical mechanics supplemented by some quantum force, because any such attempt faces serious obstacles, as it employs, in one way or the other, equations which are nonlinear with respect to the wave function. Moreover, the wave function can be measured. For instance, in the simple case of bound states of an atom, the amplitude can be

measured in terms of the mean particle density and the phase can be measured by the magnetic moment of the atom. In general, the wave function can be measured by the differential cross-section for elastic scattering of probe particles.

The usual problem in quantum mechanics is that of finding the eigenfunctions and the corresponding eigenvalues of the Hamiltonian of the systems. The inverse problem is to find the Hamiltonian from the experimental data on the energy states and the corresponding eigenfunctions. A method for finding the potential function is given and illustrated by reference to scattering in the S state for a system. But the method is, in general, not of much practical importance.

There is a detailed discussion on the role of the measuring instruments in quantum mechanics. The output of any instrument is always a macroscopic effect, eg deflection of a pointer in a metre, formation of droplets in a Wilson cloud chamber, blackening of grains in a photographic emulsion, etc. Thus, we use a macroscopic instrument to measure a microsystem. A detailed analysis is given of the action of the measuring instrument both as an analyser of a quantum ensemble and as a detector of individual events. It is shown that a macroscopic measuring instrument must be an unstable system, for only then a microparticle can alter its state and this change of state is a macroscopic event. A microparticle is powerless to act as an instrument represented by a stable macroscopic system; for instance, it cannot displace the pointer from the zero position. The observer himself does not get involved in any measurement.

Einstein gave an example, known as Einstein-Rosen-Podolski paradox, to show that quantum mechanics is not a complete theory. It was shown by Bohr and later by Mandelstam that the arguments given in the example regarding the possibility of simultaneous measurement of the coordinate and momentum of a particle are fallacious, because they contradict the principle of complementarity. The problem of latent parameters in quantum mechanics is then discussed and it is shown that it is impossible to introduce latent parameters which are, in principle, observable without causing a conflict with the principle of complementarity. However, this does not rule out the presence of unobservable latent parameters. An example is given to illustrate the point. Further, the individual history of a particle can be given as a sequence of macroscopic events; for instance, the history of a π -meson which collides with a proton and so generates two new π -mesons in a bubble chamber. One should not hope for a more detailed description of the events, for each bubble in the bubble chamber reflects a catastrophe in the microcosm. We cannot point to a single experimental fact that would indicate that quantum mechanics is incomplete within the range of atomic phenomena of the microcosm.

F. C. AULUCK

SEMIMICRO QUALITATIVE ANALYSIS by F. J. Welcher & R. B. Hahn (East-West Press Private Ltd, New Delhi), 1968. Pp x+497. Price Rs 10.00
 A book of the above title is expected to emphasize the practical schemes of qualitative analysis embracing all the common and many of the rare elements. But the present edition falls short of this expectation. The authors have divided this work into three parts, namely Part I: Theory; Part II: Reference; and Part III: Experimental. Part I comprises 15 chapters, Part II, 11 chapters (16-26 both inclusive), and Part III, 15 chapters. The importance given to the analytical part itself is very much diluted.

A book on qualitative analysis should aim at providing analytical chemists with proper background of theory rather than help produce laboratory technicians and, therefore, any book on the subject should necessarily include chapters on theory pertaining to that branch of analysis. However, after going through Part I of the present work, it is observed that many of the chapters in it are not directly relevant to the title of the book. Chapters 1-4, 9, 14 and 15 will be more appropriate in a book on general chemistry, dealing with volumetric, gravimetric and physical chemistry experiments. Chapters 10 and 12 could as well have been included as subtitles of Chapter 8. Inclusion of not very relevant chapters further depreciates the value of the work.

In Part II, Chapters 16 and 17 are tables, which are useful for analytical chemists in general. Chapter 18 on mathematical operations has been written with commendable simplicity and it will be very useful for quantitative analytical chemistry students rather than semimicro qualitative analysis workers. Apart from these three chapters, the other chapters (19-26) deal with the properties of metals and their ions. Their placement under Part II is of doubtful expedience. A better arrangement would have been to include the material of the latter chapters under Part III in appropriate chapters like 30, 31, etc. Also, the following material could have been brought under Part II: Table 5 (p 15); Table 9 (p 24); Tables 16 and 17 (p 39); and Table 22 (p 54); all these are useful for general analytical chemists.

The authors claim to deal with the 'large semimicro scale' (preface, p v). This is a new coinage by the authors, as there is no such division as large and small semimicro scale. On going through the volumes and quantities recommended for work in the book, the procedures seem to be more or less on a macroscale. One feels that the title of the book is a misnomer as compared with the books of Holness and Belcher.

Semimicro analysis is of special significance in modern analytical chemistry, as it introduces a scheme for the detection of rare elements. Complete exclusion of the rare elements with the exception of titanium limits the usefulness of this book considerably. Description of the apparatus and techniques used in semimicro analysis given in Chapter 27 is very inadequate. Companies like BDH have put in the market semimicro kits (known as 'Meso-chemical analytical kit') comprising a set of useful apparatus and organic reagents for confirmatory tests. In the light of these developments, Chapter 27 is inadequate.

Advantages claimed in page vi of the preface are of doubtful usefulness. Holness and Trewick's method using Li(OH) and KNO_3 is not advisable for Asian laboratories, as Li(OH) is several times costlier than NaOH. Moreover, sodium hydroxide works as well in the separation of elements of group II A and B.

On page 386, the authors proceed to precipitate members of groups III and IV of the regular qualitative analysis collectively as mixed hydroxides and sulphides. The advantage of this mixed precipitation procedure is very doubtful. It has definite disadvantages, such as (i) having to do an additional separation, and (ii) creating confusion in the minds of students who have been already trained in the regular qualitative analysis, because groups IV and V of the authors correspond to groups V and VI of the conventional qualitative analysis. (Vogel is probably the inspiration for the authors in this respect.)

Willard and Goodspeed's nitric acid method for the separation of calcium and strontium adopted by the present authors seems to be elusive, since even a small quantity of water on the semimicro scale may give negative result.

On page 413, the authors admit that spot tests are not given prominence in this work, and, in fact, they make use of only 'three organic reagents' (preface, p vi), namely dimethyl-glyoxime, Aluminon and Magneson. No doubt, organic reagents are vulnerable to interferences, but a large number of precise tests under controlled conditions are possible in semimicro analysis with the help of 20-25 reagents. This again is a point of deficiency in the book.

The applications of hydrolysis in analytical chemistry given under 'Principles of hydrolysis' are too far fetched and unusual to be of any practical necessity. No doubt, they are interesting examples to enrich the theoretical background.

The questions and worked-out problems at the end of each chapter are commendable. Lucidity of expression and presentation show the experience of the authors as teachers. Figs. 6-8 in Chapter 3 are specially commendable. Figs. 18 and 19 on pages 165 and 166 respectively giving the electronic configuration for the tetrahedral, both as sp^3 and d^2sp^3 , are confusing; d^2sp^3 corresponds to fc octahedron. Para 174 on page 172 gives two formulae for the product of reaction of ferric ion with thiocyanate, $[\text{Fe}(\text{CNS})_6]^{3+}$ and $[\text{Fe}(\text{CNS})]^{2+}$, which are again confusing in the absence of correct depiction of conditions. On page 175, the inclusion of NH_4^+ radical along with aquo complexes is misleading. NH_3 can form NH_4^+ radical not only with water but also with any protonic acid. The authors could have adhered to the conventional way of representing electronic configuration, eg on page 244, $6s^26p6p$ could have been better represented as $6s^26p^2$ or $6s^26p_x6p_y$. The same remark will apply to other instances in the book (eg pp 253 and 264). Similarly, the structure of oxygen as an example of double bond given on page 13 is also not satisfactory. At times the oxygen structure is also :O:::O: to explain its paramagnetism. Ethylene or carbon dioxide would have been a better example of double bond.

In the tables depicting complexes (eg on pp 244, 248, etc) the coordination number and the oxidation states could also have been included as two additional columns. In page 252, the expression 'dissolves giving colourless solution' would have read better than 'dissolved with colourless solution'. This error has been repeated in other places also (eg on p 254, Table 35). 'The analysis of the general unknown' is a repetition of the material given on page 418 and hence it is superfluous. In page 432, under flame test, colours of the flames for a few common ions could have been given. Under ammonia test (p 437), the hanging-drop cell method could have been indicated. Chapters 39 and 40 could have been amalgamated. There are quite a few printers' devils at various places.

To summarize, the material inside does not justify the title given to the book. The authors have dissipated their energies in too many theoretical chapters, which are not directly useful for semimicro qualitative analysis. Analytical (practical) part has been presented in a rather diffuse form. If the book is aimed at helping semimicro analytical students, it definitely fails in the purpose. It cannot be said that it has added anything more to the technique of semimicro analysis than what has been done by Holness, Vogel and Belcher.

However, it should be admitted that many of the chapters given under Part I will be of immense use to the general chemistry students. The authors have meticulously represented all equations in ionic forms as befitting modern theories. The theoretical chapters are very lucid, which any student can follow unaided. Several questions are presented at the end of each chapter. Worked-out examples provided will be of great help to the concerned students, though not to the students of semimicro analysis.

P. B. JANARDHAN

MEDICINAL PLANTS by S. K. Jain (National Book Trust, India, New Delhi), 1968. Pp xii+176
+plates: black and white, 29 and coloured, xii.
Price Rs 5.75

This book is the latest in the series 'India — The Land and People' being brought out by the National Book Trust, India. As the author says in his introduction, the purpose of the work is 'not to prepare a book of prescriptions for different diseases but to apprise the reader with some of the more common reputed medicinal plants of India'. Some 80 plants have been dealt with in as many chapters. The trade name or the most common name of the drug forms the title of the chapter. The botanical name, common names and botanical description are followed by the most important part of the chapter — that dealing with the properties of the drug and giving all the uses which it finds in the traditional systems of medicine and folklore.

The book is a valuable addition to the series. The value of the book to all classes of users as a popular handbook would, however, have been enhanced if the author had identified under each drug the properties and uses substantiated by chemical investigations or mentioned in one or the other of the pharmaceutical codices so as to separate them

from the other uses which could be attributed to indigenous system or folklore only. Further, it would perhaps have been quite profitable if a qualified medical man conversant both with allopathic and ayurvedic/unani systems were also associated with the author in compiling the sections dealing with medicinal uses for a critical assessment of the medical literature on the subject. Thus in page 57, *Embelia tsjeriam-cottam* is said to be 'effective only against tapeworms, and not hookworms and roundworms', but in the very next sentence we are told that 'it should be useful in ascariasis'. Ascariasis is, as we know, the medical man's term for roundworm infestation.

In spite of the seeming profusion of text-figures and also black and white and colour plates in the book we find that some 30 out of the 80 plants dealt with are not illustrated at all and this reduces the utility of the book to that extent. For example, there is no illustration of *Ocimum sanctum* and that of *O. Kilimandscharicum* is so poor that it could well have been omitted. The illustration of *Ephedra gerardiana* is also very poor and shows more rock and soil than plant. A reference to appropriate text-figure or plate should have been given in each chapter. The botanical name of each plant should have been given in parentheses after the title of each chapter in the contents page, particularly since the serial arrangement of the chapters has been based on the alphabetical order of the botanical name of the drugs.

R. S. CHAKRAVARTHY

ANIMAL GAMETES (FEMALE) by Vishwa Nath (Asia Publishing House, Bombay), 1968. Pp xix+195.
Price Rs 60.00

Like its predecessor, *Animal gametes (Male)*, the present volume is yet another monumental contribution by Dr Vishwa Nath, the pioneer animal cytologist of the country. It is devoted primarily to a morphologic and cytochemical account of yolk formation during oogenesis in invertebrate (insects, spiders, crustacea) and vertebrate (fishes, amphibia and mammals) forms, based mostly on outstanding researches by the author and his associates. The classical cytologic techniques and the recent sophisticated cytochemical and electronmicroscopic procedures have been used judiciously and pertinently to elucidate the morphology, ultrastructure and cytochemistry of organelles (nucleolar extrusions, yolk nucleus, mitochondria, Golgi complex, vacuome and the endoplasmic reticulum) involved in vitellogenesis. Particular attention has been paid to synthesis of protein yolk by RNA-rich nucleolar extrusions, mitochondria, yolk nucleus and the endoplasmic reticulum. The role of Golgi complex in biogenesis of yolk triglycerides has likewise been described in detail.

The presentation of the textual matter and the illustrations are superb and the coverage of literature has been extensive. The treatise will be a valuable source of information to the cytologist specially interested in female gametes. Dr Vishwa Nath deserves our gratitude for this excellent, scholarly and timely treatise.

A. B. KAR

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- STRUCTURE AND BONDING: Vol 4, edited by C. K. Jørgensen, J. B. Neilands, Ronald S. Nyholm, D. Reinen & R. J. P. Williams (Springer-Verlag, Berlin), 1968. Pp iv+229. Price DM 48 or \$ 12.00
- HANDBOOK OF EXPERIMENTAL PHARMACOLOGY edited by O. Eichler, A. Farah, H. Herken & A. D. Welch (Springer-Verlag, Berlin), 1968. Pp xxviii+1178. Price DM 320 or \$ 80.00
- CHEMISTRY OF COMPLEX EQUILIBRIA by M. T. Beck (D. Van Nostrand Co Ltd, London), 1968. Pp 220. Price 65s
- SOLVENT EXTRACTION OF METALS by A. K. De & S. M. Khopkar (D. Van Nostrand Co Ltd, London), *under preparation*
- MODERN METHODS IN ORGANIC MICROANALYSIS by J. P. Dixon (D. Van Nostrand Co Ltd, London), 1968

NOTES & NEWS

Lasers for measuring rates of ultra-fast chemical reactions

A laser beam has been proved by workers at the University of California to be superior to an ultrasonic probe for determining the rates of ultra-fast chemical reactions. The analysis of reactions, occurring within 10^{-12} sec, may now be possible by measuring the quasielastic scattering of laser light by salt solutions. Even when the solutions are at equilibrium, as far as temperature, pressure and concentration are concerned, they are never entirely free from minute local fluctuations. The return to equilibrium of these spontaneous fluctuations involves a characteristic time that is characteristic of the kinetics of the system.

The new technique depends on the Doppler broadening of the laser wavelength caused by localized fluctuations in the solutions through which the laser passes. The degree of wavelength stretch is a measure of the rate of the chemical reaction. Due to the great 'purity' attainable in a laser beam, it is possible to detect a wavelength spread of even one part in 10 million Å.

This laser technique can be applied to a variety of ultra-fast chemical reactions, such as those involving proton transfer. It should enable biochemists, for example, to gain insight into the mechanistic details of enzyme-initiated processes [*Chem. Engng News*, 46 (No. 29) (1968), 14].

Melting point determination by NMR

A new method for observing melting and determining melting points, utilizing the motional narrowing of the NMR line at the phase transition, has been developed. Impurity premelting takes place during the melting process in any real substance. This premelting helps the observation of a spin echo signal immediately below the melting point.

Impurity premelting, also known as two-phase premelting or Raoult

premelting, arises from the presence of impurity centres distributed throughout the solid sample. In the immediate vicinity of these impurity centres, the melting temperature is lower than that for the pure substance. As the temperature is increased, more of the substance melts until the apparent melting point $(T_m)_A$ is attained. At this point, the entire sample is liquid.

According to the Raoult theory of impurity premelting, the fraction of the sample which is liquid, $A(T)$, at any temperature T below the apparent melting point, is given by the equation

$$A(T) = \left[\frac{1 + (T_m)_A - T}{\Delta} \right]^{-1}$$

where Δ is the difference between the true melting point T_m and the apparent melting point $(T_m)_A$.

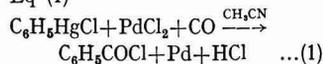
With the aid of pulsed NMR, the liquid fraction of a sample is measured as a function of temperature in the premelting region. This new method of melting point determination is applicable, in principle, to any substance containing nuclei with spin greater than zero. To measure this liquid fraction, a spin echo is produced by placing the sample in a magnetic field and irradiating it with two radio frequency pulses a time τ apart at the Larmor frequency. The echo, of amplitude A , appears at a time 2τ after the first impulse. The echo amplitude depends on n , the number of spins contributing to the signal. It can be shown that the spin echo amplitude in most cases depends on n , the number of spins in the liquid environment, so that A is directly proportional to the liquid fraction $A(T)$.

Two samples of ethane were used to demonstrate the above method. The first sample had a purity of not less than 99.96%, and the second not less than 99%. The echo spin amplitude was observed for both cases as a function of temperature between 88.8° and 90.2°K. It was found, after making the necessary calculations, that the estimation of

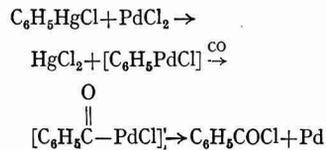
the true melting point in the two cases agreed very well. The temperature control in this experiment was done using a liquid oxygen bath, the melting point of ethane being close to the boiling point of oxygen. A platinum resistance thermometer calibrated by the National Bureau of Standards was put next to the sample tube and both were placed in a copper case to minimize temperature gradients. The vapour pressure over the bath was controlled to ± 0.1 mm of mercury by a Cartesian manostat. The two commonly quoted values for the melting point of ethane are 89.89° and 90.35°K. The above method yielded the value 89.82°K, consistent with the first one [*Nature, Lond.*, 219 (1968), 59].

A new synthesis of aromatic and aliphatic carboxylic acids

A new synthesis of carboxylic acids by a Pd(II)-catalysed carbon monoxide insertion, which takes place under mild conditions with a variety of metal aryls, has been reported. These metal aryls react with Pd(II) under carbon monoxide atmosphere to give palladium metal and acid chlorides. Thus, phenyl mercuric chloride reacts with Pd(II) chloride in CH_3CN under atmospheric carbon monoxide pressure at 25°C to give benzoyl chloride according to Eq (1)



This reaction probably proceeds by way of an unstable aryl palladium compound which inserts carbon monoxide followed by decomposition of aryl compound to give acid chloride.



Esters are obtained using alcohols as solvent. Pd(II) catalyst may be regenerated by reaction with CuCl_2 . In some cases diaryl ketones and coupled products are obtained as side products. The corresponding alkyl metal compounds react to give aliphatic

carboxylic acids. The reaction is applicable in those cases also where mercury aryls have been generated *in situ* [*Tetrahedron Lett.*, (No. 19) (1968), 2285].

A new oxytocic substance from ox hypothalamus

A new oxytocic substance exhibiting properties different from those of any of the hitherto known oxytocic peptides has been isolated from ox hypothalamus [*Biochim. biophys. Acta*, **160** (1968), 286]. It has similar threshold doses when tested on rat uterus, guinea-pig ileum and hen rectal caecum. The smooth-muscle stimulating action of the new substance is not destroyed by incubation with thioglycollate, nor antagonized by $1.44 \times 10^{-6} M$ atropin or $3.29 \times 10^{-7} M$ dibenzylamine. The elution volume of the active material on Sephadex G-25 was similar to that of oxytocin at pH 2.9, but the substance is separable from oxytocin at pH 5.3.

Acetone-dried powders of ox hypothalamus were extracted at 80°C in 10 vol of dilute acetic acid (pH 2.9). The cooled mixture was shaken for 2 hr and spun at 1200 g for 10 min at room temperature. The precipitate was washed with half the original volume of acetic acid and recentrifuged. The supernatants were combined and concentrated *in vacuo*. The residue was taken up in 0.05M ammonium acetate buffer (pH 5.3) and spun at 1200 g for 10 min at room temperature. The supernatants were subjected to gel filtration on Sephadex G-25 columns (1.0 × 100 cm) under the pressure of a hydrostatic head of acetate at pH 5.3 and 4°C. Fractions (2.0 ml) were collected and pooled in the basis of absorbance at 280 m μ into a number of peaks (AB, C & D). After concentration *in vacuo*, the peak fractions were taken up in modified Krebs solution and tested for their biological activity against synthetic oxytocin on the isolated, isotonic uterus of the oestrus-induced virgin rats.

Quantitative analysis of S-adenosylhomocysteine in liver

Estimation of S-adenosylhomocysteine in biological materials

has, so far, been hampered by technical difficulties, such as acid instability and the lack of a quantitative method for the separation of adenosylmethionine and adenosylhomocysteine. These difficulties have now been overcome in a recently developed procedure that combines isotope dilution with two consecutive separations by ion-exchange chromatography [*Biochim. biophys. Acta*, **158** (1968), 461].

Freshly excised livers (30-50 g) from various vertebrates are deproteinized by homogenization in 1.5M perchloric acid (1:4, wt/vol) and filtered. To the filtrate, labelled adenosylhomocysteine is added and the mixture is chromatographed through a Dowex-50 (H⁺ form) column (resin bed: 5 × 1 cm) previously equilibrated with 0.5M H₂SO₄. Elution is carried out with 0.5M, 1M and 3M H₂SO₄ (0.5 ml of thiodiglycol per litre of eluant is used to prevent oxidation of adenosylhomocysteine) until the absorbance at 256 m μ is <0.05. From the 3M acid eluate, adenosylmethionine and adenosylhomocysteine are precipitated by the addition of 20% phosphotungstic acid. The precipitate is dissolved in acetone-water (1:1, vol/vol) followed by extraction of the phosphotungstate with isoamyl ether according to the procedure of Schlenk and Depalma [*J. biol. Chem.*, **229** (1957), 1051]. From the aqueous solution, adenosylmethionine is removed by decomposition with alkali [0.05M Ba(OH)₂ solution] and heating (100°C for 8 min). The solution is then adjusted to pH 4 with 0.5M H₂SO₄ and the BaSO₄ is discarded after centrifugation. The supernatant is chromatographed on a small column (3 × 0.3 cm) of Dowex-50 previously equilibrated with 0.5M H₂SO₄ to separate the degradation products of adenosylmethionine (eluted with 1M H₂SO₄) from adenosylhomocysteine (eluted with 3M H₂SO₄). The quantitative estimation of the thioether in the fractions (3M acid) is made on the basis of the molar absorbance (14200 at 256 m μ in acid) and the radioactivity is determined with a Packard Tri-Carb liquid scintillation spectrometer using a scintillation solution containing 4 g of

2,5-diphenyloxazole in 500 ml of toluene and 500 ml of ethanol. On the basis of the isotope dilution formula, the initial amount of adenosylhomocysteine in the tissue can be evaluated with accuracy.

A new method for the determination of blood volume

A new and rapid method for the determination of blood volume using indocyanine green dye (cardio green) has been developed. The blood volume determinations are made in patients by using radioactive iodinated albumin (Albumotope-RISA, Squibb) and/or chromium 51 (Chromotope, Squibb) tagged red blood cells. Indocyanine green dye (2.5, 5.0 or 10.0 mg) is injected through the same centrally placed polyethylene catheter (id 1.57 mm, od 2.08 mm; length, 33 in.) as the isotopic indicator(s). Prior to the injection of isotopes and indocyanine green dye, blood is withdrawn for calibration purposes. A separate three-point calibration is plotted for each indocyanine green dye-dilution curve. The blood collected prior to the injection of the bolus in indicators likewise is used for base counting of isotopic activity. The femoral artery is characterized precutaneously and serves as the blood sampling site. Constant withdrawal of arterial blood through a dichromatic densitometer is accomplished by a Harvard infusion-withdrawal pump. The analogue signal of the densitometer system resulting from the wave fronts of changing optical density at 805 m μ due to indocyanine green dye concentrations is continuously recorded on one channel of a multichannel recorder.

After the inscription of primary wave of the dye dilution curve, used for calculation of cardiac output, the withdrawal speed of the pump is changed from 15.3 to 7.6 ml/min and the disappearance of the indocyanine green dye from circulation is marked at the interval of 5 sec referenced to the injection of dye as time zero. A single exponential function of the decay slope is computed mathematically and extrapolated to give the theoretical optical density due to the dye at zero time. The optical density expressed as

millimetres deflection on the record was taken as variable and transcribed to a semilogarithmic plot. At 15, 25, 35, 45 min after injection blood samples are collected for counting radioactivity due to the isotopic indicators. The values of these samples are plotted in the usual fashion and plasma volume and red cell mass derived.

Besides the non-toxic nature of the dye used the other merits of this indocyanine green method are the simplicity of the instruments required for plotting the changes in its concentration in blood; the process with which this dye is removed from the circulatory system is also very rapid [*Life Sci.*, 7 (1968), 1001].

Progress in leprosy research

Although *Mycobacterium leprae*, the causative organism of leprosy, was identified in 1874, even long before the identification of many other bacteria causing disease in human, the research on leprosy progressed slowly, since *M. leprae* was not cultured till 1960. The Ninth International Leprosy Congress, held at the Imperial College, London, during September 1968 was dominated by the results obtained from fundamental and applied studies based on experimental model infection in animals. Though only localized and limited multiplication of *M. leprae* resulted from mouse footpad inoculation of bacilli from man, this model infection was used for the determination of generation time (12-15 days) to screen drugs for anti-leprosy activity and to demonstrate, for the first time, the emergence of drug resistance in patients on standard treatment with dapsone (diaminodiphenyl sulphone).

Workers at the National Institute of Medical Research, London, reported that more extensive and generalized infection can be obtained in mice made immunologically incompetent by thymectomy followed by total body irradiation (900 r). The model infection obtained by both local and intravenous inoculation of *M. leprae* reproduced a disease mimicking lepromatous leprosy seen in man, including more involvement and heavy infection of skin and nose. After a year or so, these nearly

infected mice develop intermediate type, also seen in man. Similar changes can be brought about in thymectomized irradiated mice with established lepromatous leprosy by donating back syngeneic lymphoid cells from normal mice, indicating the importance of immunological capacity of the host in determining the type of disease.

Dr Rees and his collaborators at the National Institute of Medical Research, London, showed that several of the cases of relapse during treatment with dapsone or thiambutosine carry drug-resistant strains of *M. leprae*. They have recorded 18 dapsone-resistant and 5 thiambutosine-resistant strains isolated from patients in Malaysia, India and Africa.

One of the important reports at the congress was on the effectiveness of small doses of dapsone. Before the footpad technique became available, clinicians chose doses of leprosy drug empirically. A trial in which patients were treated with only 1 mg dapsone/day, compared with the previous standard dose of 600 mg weekly, has yielded encouraging clinical and bacteriological results [*Nature, Lond.*, 219 (1968), 1310].

Regional Research Laboratory, Jammu

The annual report of the Laboratory for the year 1967 records the production, for the first time in India, on a commercial scale of ergot sclerotia of rye using the fungus strain [*Claviceps purpurea* Fr. (Tul.)]. Work on large-scale production of ergot is in progress. Of the several high alkaloid yielding strains, two (R-57 and R-56) producing mostly ergotamine and one (R-57) producing ergometrine have been tested.

A simple method for the recovery of xanthoxine from the seeds of *Ammi majus* has been developed.

Cultivation of *Datura innoxia* has been carried out in order to have the seeds as a source of the alkaloid, scopolamine (hyoscyne). One lot of 50 kg of seeds yielded 50 g of hyoscyne hydrobromide besides 9 litres of a fixed oil as a byproduct. A method has been developed for inducing tetraploids in *D. innoxia* and *D. metel*. The tetraploids thus obtained have

high alkaloid contents. Plants with as high as 0.50% alkaloid content have been produced against the normal alkaloid content of 0.2-0.3%. Cultivation of *Fagopyrum esculentum* and *F. tataricum* (buck wheat), good sources of rutin has been successful in Jammu. The leaves of *F. tataricum* gave a rutin yield of 5.7% (moisture-free basis).

The total alkaloids of *Gloriosa superba* showed a superior biological activity compared to pure colchicine. Experimental trials on the cultivation of a superior strain of *Eucalyptus citriodora* giving essential oil rich in citronellal have given encouraging results. Oil produced from these plants is of a high quality in terms of its citronellal content and odour evaluation. A method has been developed for obtaining pure hydroxy-citronellal from citronellal.

The alkaloid usaramine isolated from *Crotalaria brevifolia* was found to possess marked hypotensive property in anaesthetized dogs at a dose level of 1.5 mg/kg. It also exhibited spasmolytic and antispasmodic activities in smooth muscles of different animals. Another alkaloid, integramine, isolated from the *Crotalaria* plant showed hypotensive action. The volatile oil isolated from *Tagetes minuta* showed tranquillizing, hypotensive, anti-inflammatory, spasmolytic and bronchodilatory activities.

Methods for the extraction of active principles from senna (*Cassia angustifolia*) leaves and pods have been standardized. A process has been worked out for recovering the residual menthol from the spent oil of *Mentha arvensis* after the separation of menthol by chilling. A process for the preparation of a stable, dewaxed and refined extract of pyrethrins from the crude pyrethrum oleoresin for use in the aerosols has been standardized.

Many of the plant materials available locally have been surveyed for their suitability for the preparation of paper and rayon grade pulps. Crude pine resin after maleinization has given a satisfactory surface coating material. A blend of the extracts of pomegranate and *polygonum* when used for tanning hides gave leather of satisfactory quality.

A process for converting the locally available gypsum into surgical grade plaster of paris and surgical bandages of standard acceptable grade has been worked out. A laboratory method to recover elemental sulphur of good quality from sulphur ore deposits in the Ladakh region has been developed.

Pasteur Institute, Coonoor

Among the main achievements of the Institute recorded in its annual report for 1967 are those relating to the development of Semple vaccine for the treatment of rabies, development and improvement of methods for the cultivation of influenza and other respiratory viruses and their concentration.

Extensive treatment trials have established the superiority of 5% Semple vaccine over the 1% vaccine. The comparative efficacies of β -propiolactone (BPL) and phenol in inactivating 20% sheep brain vaccine has been studied. It was found that in one-fourth the dose of phenol-inactivated vaccine, the vaccine inactivated with BPL conferred the same degree of protection. The antigenic values of sheep and suckling mouse brain vaccines inactivated with phenol and BPL have been compared. The antigenicity of suckling mouse brain vaccine inactivated with phenol was found almost the same as that of BPL-inactivated vaccine. The brain and salivary glands of guinea-pigs which had survived on treatment after infection with street virus have been studied by the fluorescent antibody technique. The brain and salivary gland of some of the guinea-pigs showed the presence of rabies virus antigen. The finding lends support to the view that the fluorescent antibody technique reveals non-infective antigen material in treated animals surviving an infection. There was no significant difference when the conjugates employing rhodamine B₂₀₀ and fluorescein isothiocyanate were used.

The occurrence of hydrophobia among persons bitten by apparently healthy dogs has been explained by a sub-clinical infection in the animals with transient salivary excretion of virus. The

possibility of reactivating the rabies virus in the tissues of a host by the administration of a cortisone has been examined. It was found that following the administration of prednisolone, the dog excreted rabies virus in the saliva could be lyophilized.

Studies have been undertaken to determine the presence of neutralizing antibodies in natural and experimental rabies infection. It was found that neutralizing antibody was present in only 17% of sera from animals which developed rabies in spite of the treatment, compared to 59% in the sera of treated animals which survived. This finding suggests that the chances of survival are better among animal containing neutralizing antibody than those without it.

The incubation period of the rabies street virus was found to be significantly prolonged in animals which were treated with β -phenylserine post-infection.

The epidemiology and biological characteristics of 2 strains of Asian influenza virus and 7 strains of type B virus isolated earlier have been studied. The properties studied were (i) reaction to erythrocytes from different species of animals, (ii) effect of ether, and (iii) effect of heat. Antigenic analysis of strains revealed that while the Asian strain isolated during 1967 resembled A₂/Taiwan/64 strain, the B strain was closely related to the influenza B variants.

Considerable progress has been achieved towards the improvement of the medium used for the cultivation of influenza virus *in vitro*. The effect of adding certain compounds, such as cephalin, glycoyamyl, lecithin (egg) and 5-methyl creatinine sulphate to the medium was studied. The addition of egg albumin in a concentration of 2 mg/ml appeared to have the maximum beneficial effect on the culture.

Methods have been developed for the preparation and maintenance of primary monkey renal monolayer culture, primary rabbit monolayer culture, primary human amnion cell culture and BSc-1 cell culture. By serial passage of throat swab specimens in primary human amnion cell cultures one strain each of adenovirus types 1

and 2 have been isolated. Both these types are known to be latent in tonsils and lymphoid tissue of pharynx. One strain of papin-fluenza type I virus and two strains of influenza A₂ virus and influenza type B virus have been isolated.

Three monovalent seed viruses containing attenuated (Sabin) poliomyelitis virus strain, types 1, 2 and 3, have been prepared. These strains were stored at -70°C and used for the preparation of live poliomyelitis vaccine (Sabin). The effect of incorporating different compounds into nutritive agar medium at pH 7.6 on the growth of *V. cholerae* has been studied. Potassium nitrate (0.5%) suppressed the growth of *V. cholerae*. Tartaric acid at 0.5% concentration inhibited the growth of *V. cholerae*, *S. typhi*, *Sh. boydii* and *Alc. faecalis*, while *Esch. coli*, *B. proteus* and *Ps. aeruginosa* grew well in the medium. EDTA, salicylaldehyde, α,α -dipyridyl and 8-hydroxy quinoline, at 0.3% concentration, have been found to inhibit the growth of all vibrios. The action of some dyes generally used to render culture media selective was tested on *V. cholerae*. Crystal violet at a concentration of 10 μ g/ml caused complete inhibition of the growth of all the vibrios.

International Journal of Soil Biology and Biochemistry

To be published from early 1969 by Pergamon Press Ltd, Oxford, this journal will act as a medium for papers on soil organisms, their biochemical activities and their influence on the soil environment and plant growth. The journal will also publish papers dealing with biological transformation of nitrogen and of other plant nutrients in soil; soil-borne phases of plant parasites; the ecological control of soil-borne pathogens; the influence of pesticides on soil organisms; the biochemistry of pesticide decomposition in soil; microbial aspects of soil pollution; the composition of soil populations; the biochemical activities of soil organisms; and the interactions of soil organisms with one another and with plants. To be published quarterly, the annual subscription for the journal will be £ 12 or \$ 30.00.

Technological Forecasting

Elsevier Publishing Co Inc, New York, will start this new journal from early 1969. The journal will publish papers dealing directly with the methodology and practice of technological forecasting as a planning tool, including the interrelationship of social, environmental and physical factors affecting such forecasts.

Journal of Noncrystalline Solids

This new bimonthly journal has been started by North-Holland Publishing Co, Amsterdam. The journal devotes itself to original research papers and critical and authoritative review articles on glasses and amorphous materials. The annual subscription for the journal is \$ 20.00.

New Periodicals from Pergamon Press Ltd

Geo Forum — This new journal will publish papers on various aspects of physical geography, eg geology, oceanography and meteorology. The subscription rates for the journal are yet to be announced.

Forma et Functio — This journal to be published quarterly will carry papers dealing with morphology, physiology, ecology and evolution in relation to functional biology. The annual subscription for the journal is £ 10.

Archives of Anthropology International Journal of Comparative Human Biology — This journal, which was started in 1866 and lapsed in 1943, is now being revived as a quarterly. It will deal with palaeanthropology, physical anthropology, population genetics, ethnology and social anthropology. The annual subscription for the journal is £ 10.

Contribution to Atmospheric Physics — This new quarterly journal reports the results of experimental and theoretical research work in all branches of meteorology. Published in English, French or German, all articles have summaries in other two languages. The annual subscription is £ 10.

Energy Conversion — Papers concerned with primary energy sources, both direct and conversion process, and the problems associated with regulation and control will

FORTHCOMING INTERNATIONAL SCIENTIFIC CONFERENCES, 1969

| Date | Conference | Place |
|------------|---|---------------------|
| 5-8 May | International Symposium on Microwave Theory and Techniques | Dallas |
| 19-24 May | International Colloquium on the Organic Chemistry of Phosphorus | Paris |
| 2-6 June | 3rd International Symposium on Yeasts | Delft and The Hague |
| 3-13 June | International Conference on Arid Lands in a Changing World | Tucson |
| 9-11 June | International Communications Conference | Boulder |
| 9-12 June | 7th International Food Congress and Exhibition | Madrid |
| 15-20 June | International Data Processing Conference | Montreal |
| 16-20 June | International Colloquium on Classical and Relativistic Magnetohydrodynamics | Lille |
| 16-21 June | 4th International Federation of Automatic Control Congress | Warsaw |
| Summer | 8th Congress of the International Commission for Optics | Great Britain |
| 22-29 June | 5th International Congress on the Application of Mathematics in Engineering | Weimar |
| 23-25 June | 7th International Shock Tube Symposium | Toronto |
| June | 16th International Astrophysical Symposium | Liège |
| June | 7th International Aerosol Congress | Paris |
| 7-11 July | International Colloquium on Structure and Properties of Solid Surfaces | Paris |
| 13-19 July | 7th International Congress of Clinical Pathology | Montreal |
| 14-18 July | 4th International Congress for Pharmacology | Basel |
| 14-18 July | International Conference on Atomic Absorption Spectroscopy | Sheffield |
| 20-25 July | 8th International Conference on Medical and Biological Engineering | Chicago |
| 21-25 July | International Symposium on Analytical Chemistry | Birmingham |
| July | 25th International Union of Pure and Applied Chemistry Conference | Cortina D'Ampezzo |

appear in this new quarterly. The annual subscription for the journal is £ 16.

Announcements

■ *The Sixth International Congress of Precast Concrete Industry* will be held in Amsterdam during 17-23 May 1969. The congress is being organized by the Association of Concrete Products Manufacturers in Holland, the Netherlands number association of the Bureau International du Béton Manufacturé. About 1700 delegates representing 50 countries will attend the congress.

The congress will have five working sessions covering (i) quality control; (ii) organization, mechanization and automation; (iii) structural lightweight concrete; (iv) outstanding aspects of the manufacture and application of precast concrete; and (v) construction of bridges with precast elements. There is also an extensive programme of technical study visits.

Further details can be had from the Congress Organizer,

Organisatie Bureau Amsterdam, P.O. Box 7205, Amsterdam, the Netherlands.

■ *The Twentieth Meeting of the International Committee on Thermodynamic and Electrochemical Systems (CITCE)* will be held at Strasbourg (France) during 14-20 September 1969. The discussions at the meeting will be on 'Transport phenomena in electrochemical systems'. The main aspects to be covered include (1) theoretical and experimental basis of the phenomena; (2) mass and charge transport in solution (in particular in diffusion layer); (3) mass transport in the pores of porous electrodes (particularly in fuel cells); and (4) mass transport towards and through membranes (in particular in biological electrochemical systems).

The last date for the submission of forms by intending participants is 15 April 1969. The forms can be obtained from Dr H. Tannenberger, Institut Battelle, 7 route de Drize, 1227 Carouge-Geneve, Switzerland.

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This well-illustrated supplement provides information in an easy-to-grasp form on: (i) zoological names of 376 fishes of economic value, found in Indian water, along with their English names; (ii) description and distribution of the fishes; (iii) coastal, deep sea and fresh water fisheries; (iv) ingenious devices for catching and preserving fish; (v) fisheries in various States; (vi) manufacture of fish oil and manure; (vii) analytical values of fish-foods and their byproducts; and (viii) marketing practices and data concerning fish trade. An annotated bibliography of 220 references and an exhaustive index are provided.

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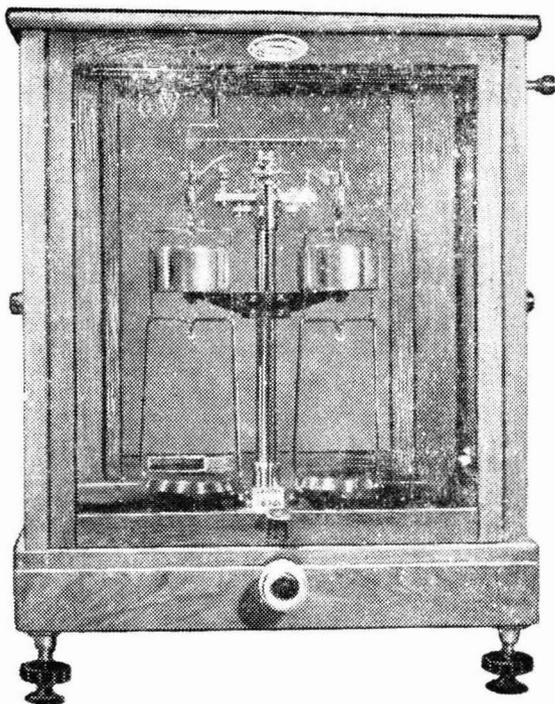
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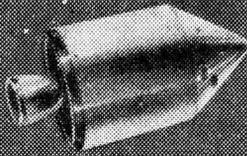
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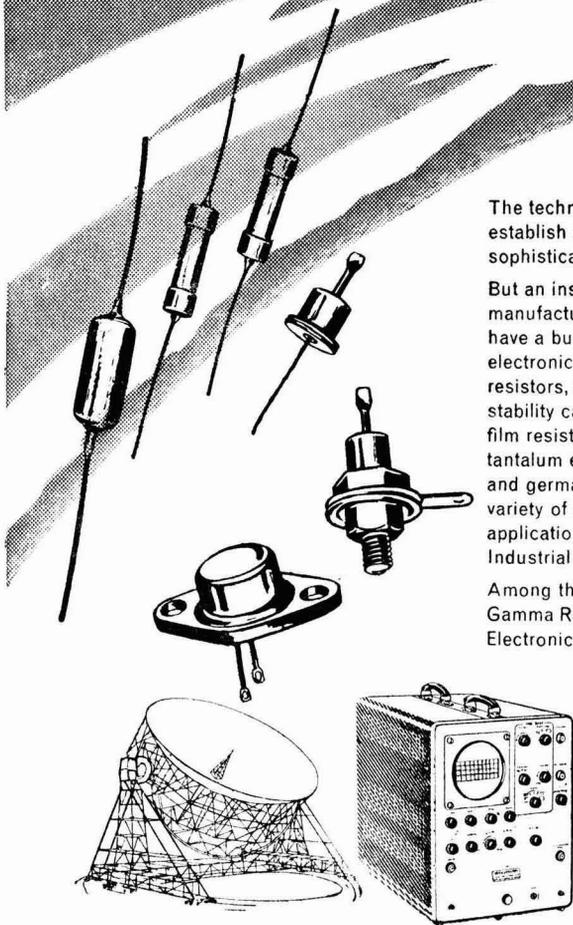
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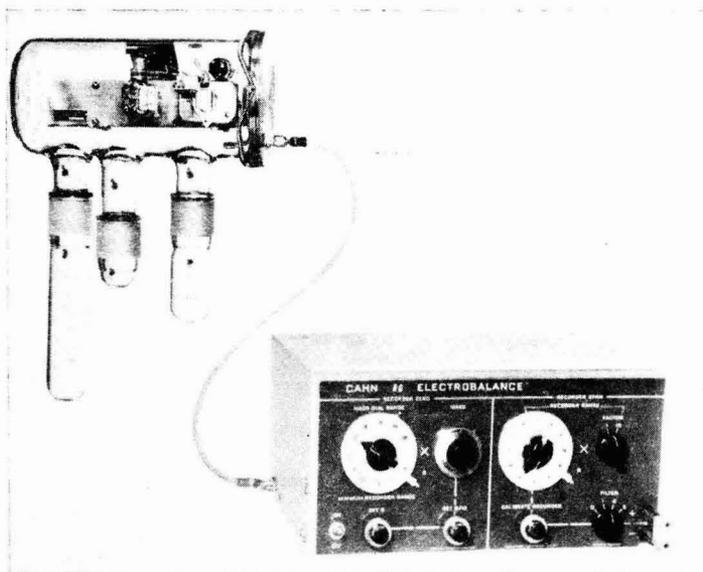
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