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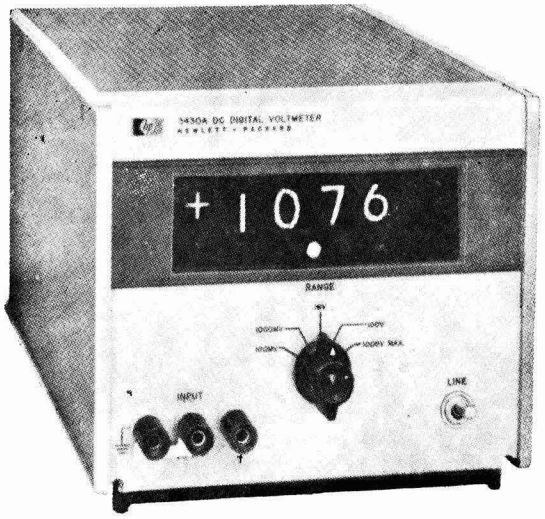

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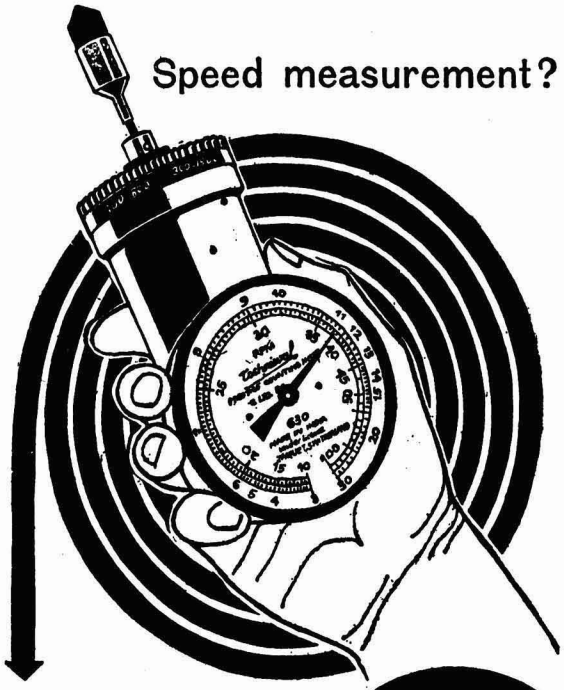

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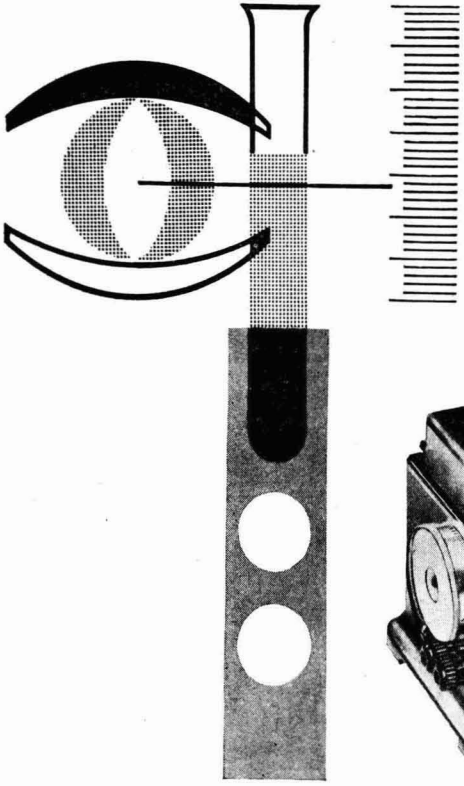

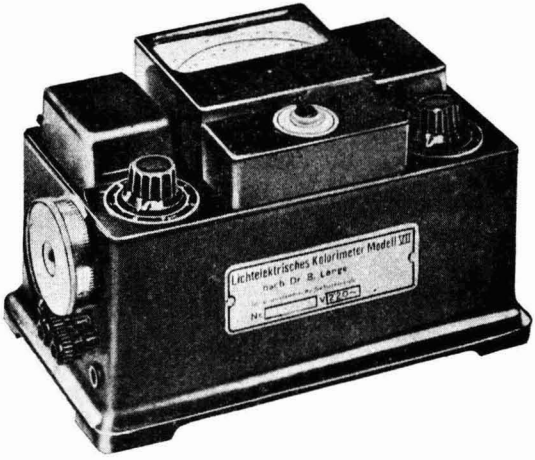

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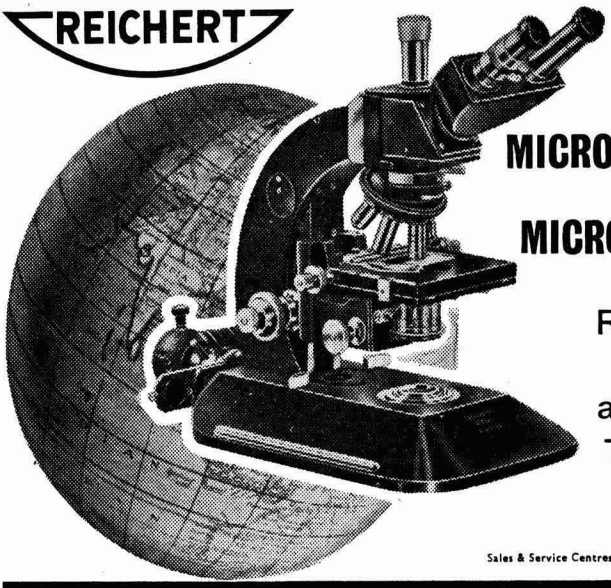

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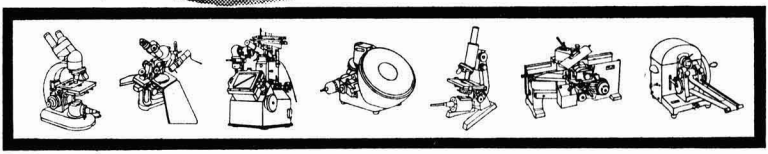

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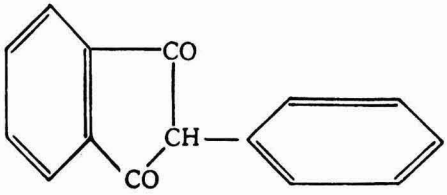

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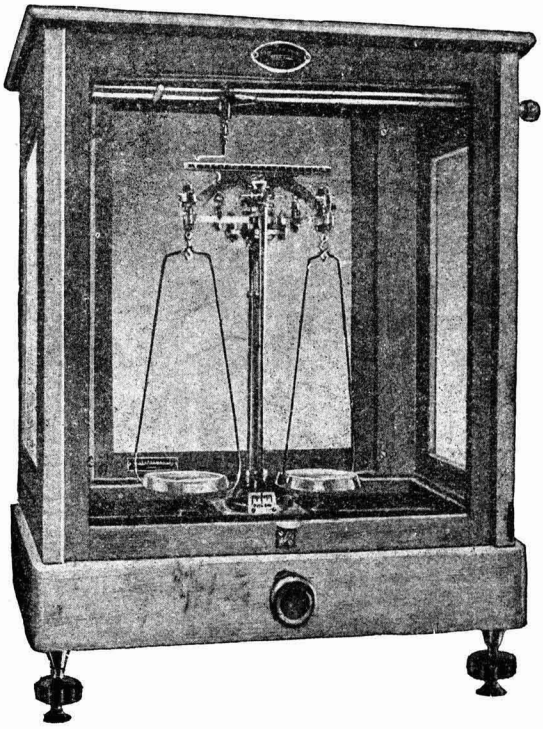

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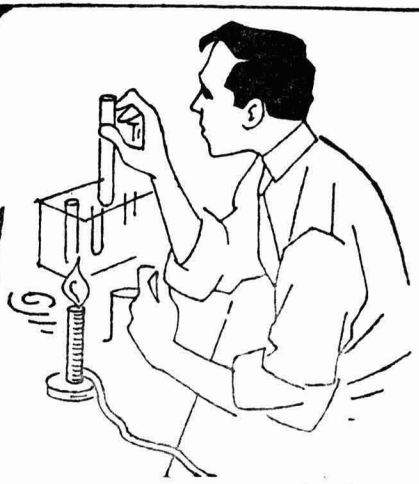

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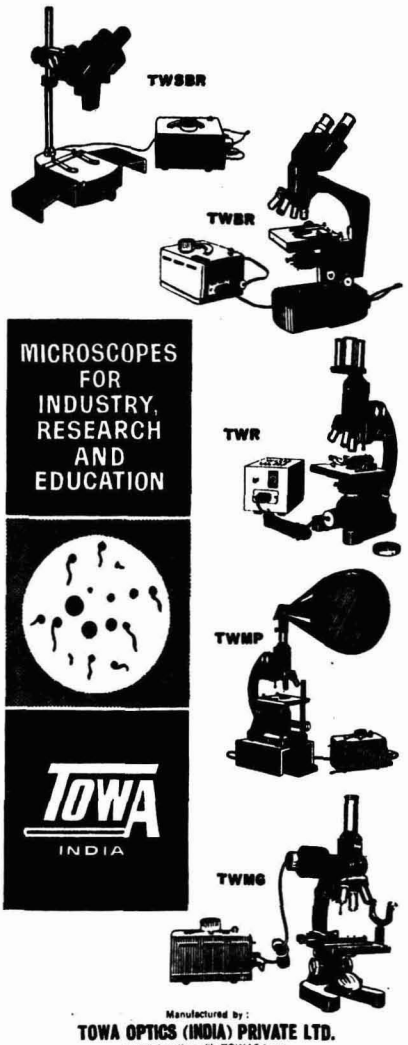

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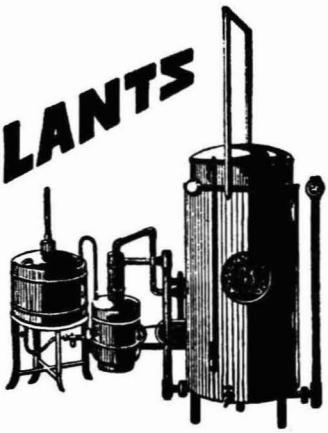

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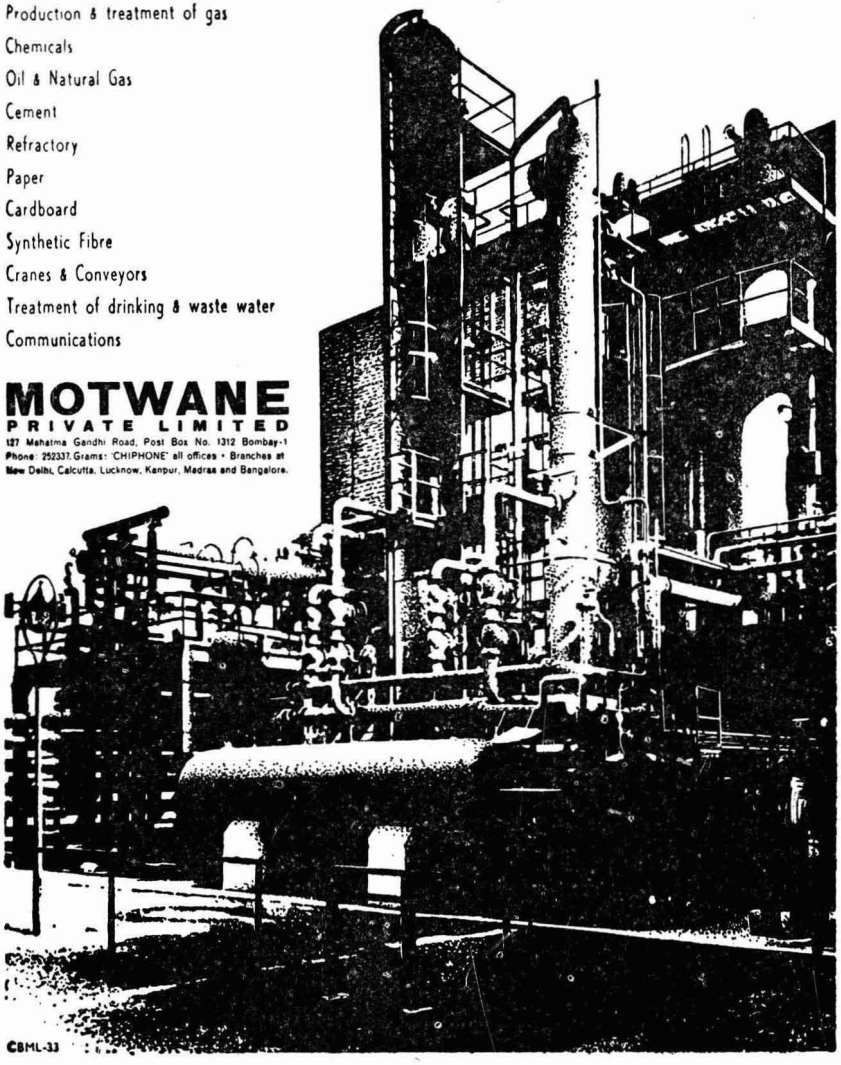

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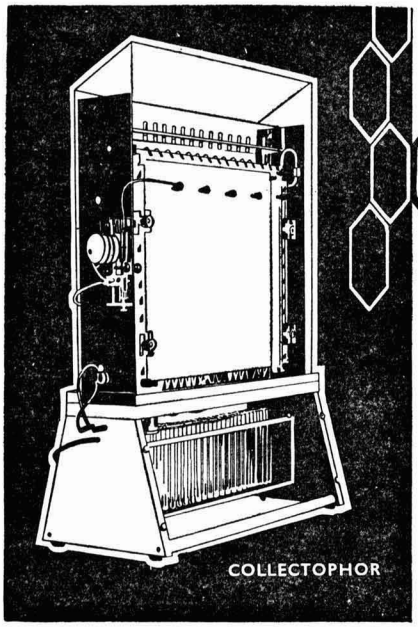

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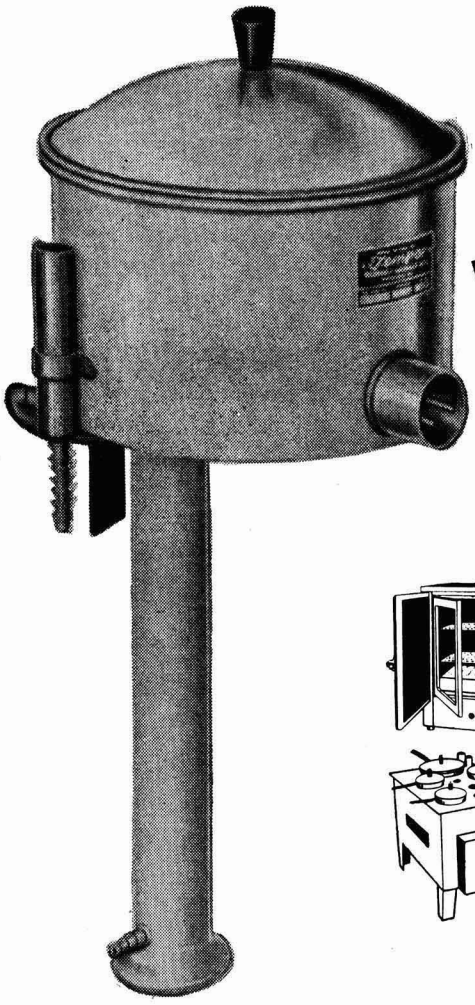

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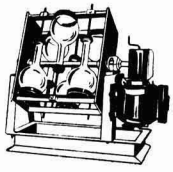

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Current Topics

Plans for Increasing Rice Output in Developing Countries : Need for Fixing Priorities

THE deliberations of the International Rice Commission (IRC), which met in New Delhi during October for its tenth session, are of considerable significance to rice producing countries, particularly to developing countries like India where rice shortage is chronic and the gap between demand and supply is continually widening. The launching of the International Rice Year, whose objectives formed the subject of discussion at the meeting of IRC, coincided with the session. A special symposium on factors promoting high yields of rice was also organized during the session. The meeting served to recapitulate and restate the problems involved in increasing rice production and the measures to be taken to achieve the targets.

Reports by various agencies and individuals presented at the meeting of IRC show that the problems concerned with increasing rice output have received serious thought and the basic ones have been spelt out. Also, the solutions for most of the problems are known. The main requirement, therefore, appears to be a question of choosing the most feasible of the solutions, and assign them top priority.

Basic problems to be considered can be grouped under three heads: (i) Agricultural, (ii) Social, and (iii) Technological. The implementation of programmes coming under these categories has to be viewed from three considerations: (i) magnitude of the programmes, (ii) resources available, and (iii) feasibility of the programmes in a given time period. The last consideration is important and should receive special attention as one is confronted with a race between rapidly increasing population and production of adequate quantities of foodgrains. Agricultural programmes are long-range ones requiring a good deal of spade-work, much capital investment and tremendous extension work. Much investigation has to be done to evolve high yielding varieties of rice, try them out under a variety of soil and climatic conditions, and popularize them. Many snags have to be overcome, particularly combating diseases to which the high yielding varieties are susceptible and fertilizer production has to be increased many times. Also, modernization of agriculture is closely interwoven with many social problems involving large sections of the population. A high percentage of literacy is a prerequisite for any country-wide programme to be put into operation. This is a slow process and it may be 2 or 3 decades before literacy percentage can be raised to the required level. Introduction of land reforms and new patterns of land use, raising the income of the farmer and facilitating agricultural credit, mechanization of agriculture and expansion of irrigation facilities are all long-range socio-economic problems, and quick solutions are not possible.

While modernization of agriculture is an important measure and must be pursued vigorously, technological aspects of rice industry are equally important in augmenting our rice supplies. Modernization of rice industry — storage, preservation and processing — alone is estimated to cut down losses and augment our rice resources by 10-20 per cent, and if the programmes are properly implemented, the present deficit will be more than wiped out.

An attractive feature of the above viewpoint is that the steps to be taken could perhaps be implemented more quickly and with less capital expenditure than other measures. The question of educating the masses about the advantages of adopting modern technological methods may not be insurmountable. Well designed and simple community storage silos for paddy and rice have to be developed to suit an average rice producing village employing locally available construction materials. This would be the first step in minimizing the heavy losses due to rodents. Effective preservation of stored grain from insect pests with insecticides, preferably with activated clays which have been found effective, should be taken up side by side. As a first step in rice processing, the existing mills may be modernized so that parboiling, drying, shelling and other operations are effectively carried out. New mills should be set up only when a rice producing area has not adequate number of mills. All the know-how for processing rice, developed within the country, is available for exploitation, and equipment needed can also be produced within the country.

From all these considerations, the important point emerging is that a careful review of all the feasible measures for increasing our rice production is called for, and a crash programme should be undertaken to implement the selected ones. It appears that the technological aspects of rice industry set out are more promising and offer a quicker solution to the problem of increasing rice output. The technological measures have something else to recommend their immediate implementation. Increased rice production as a result of modern agricultural practices will not represent a real net increase unless the technological aspects are introduced well in advance and the capacity of storage and processing units is expanded gradually. If not, it will result only in greater losses due to defective storage, preservation and processing, and a *status quo* as far as the gap between the supply and demand of rice is concerned.

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Integrated Survey of Natural Environment

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WITH the assistance of Unesco, the Netherlands Government have set up an International Training Centre for Aerial Survey (ITC) where trainees from African, Asian, Middle-Eastern and Latin American countries are undergoing training. In terms of the agreement with Unesco, the first Annual Seminar on ' Integrated Surveys of Natural Environment ' was organized from 22 to 29 April 1966 at Delft. The members of the Unesco Natural Resources Research Advisory Committee were invited to take part in the symposium. Besides, the staff members of the ITC participated and presented papers on various aspects of natural environment.

In his opening address, Mr M. Batisse, Chief, Natural Resources Research Division, Unesco, discussed the broad concept of natural resources which includes resource products and resource situations. The fundamental factor which complicates the concept is the dynamic changes taking place in survey and development of resources with changing technology, availability of large volume of energy from thermal, hydro and nuclear sources. In this concept the economic aspect cannot be overemphasized. In the study of the potential of natural environment several physical sciences are involved in which human beings take part. It is a living environment and biotic factor is the synthesizing agent. In the ecosystem circulation of energy, fixation of energy by photosynthesis, and rational use of biosphere and its conservation and optimum utilization must receive due attention. In development of the potential of resources ecological attitude is essential.

Dr I. S. Zonneveld stressed the importance of plant ecology in integrated surveys. The factors such as climate, parent rock, relief, hydrology and biotic activity which are associated in the formation of soils also influence vegetation and both remain in dynamic equilibrium. The tasks assigned to ecology are inventory of vegetation resources in mappable units, indication of other natural resources, interrelation of climate and vegetation and development of resources of crop production, rangeland and forestry.

Mr R. Oluwole Coker spoke on the need of aerial photographs of required specifications for land development. Mapping is a prerequisite for it. Larger the scale of map, earlier will it be out of date. Therefore, mapping programme should meet not only the immediate but future needs as well.

According to Prof. C. Troll, landscape ecology denotes the analysis of a physico-biological complex of interrelations which govern the different area units of a region. Consequently, in a natural landscape this ecological complex is the only active one and in a humanized landscape it is but in interaction with the social-cultural functions. It involves large-scale analysis of natural regions and study of the problems of population, society, rural settlement, land use, transport, etc. It aims directly at integrated survey of natural environment. According to its physiographic nature, a natural region consists of a certain set of ecotopic types. Energy balance determines productivity of landscape in terms of microorganisms, vegetation, animals and human beings.

Dr J. C. J. Mohrmann stated that the problems of operational design of integrated surveys are often underestimated with the result that many surveys become costly, inefficient and ineffective. This is of value both in scientific and practical surveys. There should be clear phase limit between survey and development planning for which careful planning of the different phases of survey and design is of prime importance. Ripening process of ideas and the psychological penetration of teamwork are essential prerequisites for such surveys. The agronomist in the team has a central position in coordinating the activities and integrating the results of surveys. The development planning surveys and projects should have local participation well integrated with aims of local policy, to the skill of local technical services and to the way of living and thinking of the population.

Prof. E. W. Russell stressed the study of soil characteristics which are of major importance for crop production under irrigation, particularly the microphysical structure consisting of pore space, size distribution of pores, distribution of pores containing air, soil structure and degree of cracking, soil depth, exchangeable sodium percentage, infiltration rate, nutrient status of soil, salt content and available soil water range. Besides, the surveyor must obtain the maximum amount of ancillary information that is relevant to salinity prediction under irrigation.

Dr I. Szabolcs stated that the main task of soil survey is to promote the satisfactory utilization of soil for agricultural purposes. Genetic soil survey is the proper system to solve the problems of the soils for their rational and economical utilization. The soils should be classified into types, subtypes and variants to indicate the problems of soil technology and reclamation. The data should be represented in various cartograms, viz. (i) *p*H and lime status, (ii) mechanical composition, (iii) level of ground water, (iv) hydraulic properties, (v) humus content, (vi) NPK status, (vii) degree of erosion, (viii) land use, (ix) irrigation capability rating, (x) conservation of land, and (xi) soil amelioration. The use of these cartograms helps the agronomist in the field in recommending management practices to the farmers.

Prof. V. Kovda's paper on ' Soil surveys in land development ' covered the survey of (a) new lands, (b) ancient irrigated lands, (c) sequence of surveys, and (d) landscapes of arid and semi-arid group of soils. In all these surveys internal and external

geomorphology plays an important role in crop production. Besides, study of hydrogeology and investigations on hydrochemistry, microbiology and physical structure of soil are of great importance. The sequence of surveys should consist of reconnaissance surveys by aerial photo-interpretation, economic evaluation of the project, budgetary estimates of the technical project, working project, process of construction and development, and special amelioration plan. For irrigation projects the prediction of secondary water logging and salinity and the possibility of drainage should be investigated as it has several social and political consequences. The landscape of arid and semi-arid regions consists of four groups. The first group consists of those areas having no natural drainage but possessing residual salinity associated with marine deltas of internal and external seas, continental deltas, sea coast banks, and lowlands. The second group of landscapes possesses natural drainage not fully adequate and substantial residual salinity. Such a situation exists in secondary and tertiary terraces with limited degree of drainage. Secondary salinity and water logging can be avoided by provision of drainage in irrigated lands. The third landscape group possesses perfect natural drainage and consists of high plateau land underlain with pebbles, loose deposits on terraces, mountain plateau with excellent ground water circulation, and lower terraces which have high permeability and are joined to natural drainage. For each irrigation project inter-disciplinary surveys should be conducted and computer models should be built up such as has been developed for the Mekong River Development Project.

Dr M. M. Elgabaly addressed the seminar on the subject of ' Resource surveys for land reclamation '. A description of Marut Project covering 50,000 acres from Alexandria to Al Amin along the Egyptian coast was given. The area has undulating topography but interfluves are suitable for good crops of almond, olive and grapes. In the shallow phase barley is cultivated. Furrow system of irrigation has been adopted. Irrigation is spread to meet the needs of potential evapotranspiration and infiltration in addition to rainfall. This works out to 1400 $m.^3$/acre which is applied by basin system of irrigation of which 10 per cent infiltrates into the soil. In the area to be reclaimed and irrigated under Aswan High Dam Project, the soils have been classified according to their irrigable capability. The settlement projects are being initiated to provide new community services after proper assessment of the potential of land, water, vegetation, livestock and human resources. In these surveys inter-disciplinary aspect is given special importance. The specialists brought together are field surveyors, civil and hydraulic engineers, architects, sociologists, economists and administrators. For execution of the projects the Desert Development Authority assumes full responsibility.

On behalf of FAO, Dr V. Ignatieff presented a paper on ' Value of resource surveys to developing countries '. The surveys have been conducted in the field of agriculture, forestry and fisheries. FAO has helped in conducting soil surveys in 48 countries. Soil survey services have been strengthened in 31 countries. In addition, surveys of irrigation project areas, suitable settlement areas, surface water resources, ground water including geohydrological studies, topographical mapping, engineering geology investigations and quality of water have been conducted in various developing countries. About one-fifth of FAO budget is devoted for such surveys. Besides, crop ecological surveys have been organized in a very large number of countries. These surveys are based on crop ecologic classification of world climates which take into consideration soil conditions, topography, available water for irrigation and its seasonal distribution, farming systems, etc. Other surveys by FAO, WMO and Unesco comprise agri-climatology, pasture and fodder crops, livestock resources, dairy projects, forest resources, fisheries resources, etc. It is a very comprehensive paper detailing general approach to resource appraisal, organization of resource surveys, problems of natural resource appraisal in developing countries, importance of adequate survey interpretation, and follow up of survey and project development.

Dr Vink, now Professor of Soils at the Amsterdam University, presented a paper on ' Integrated surveys and land classification '. The burden of his thesis was that for practical purposes the soil suitability classification is a more rational basis of quantitatively expressing land use, land capability classification, etc., as it takes into consideration such factors as productivity, inputs, risks involved in land use, soil pattern, etc., than land classification based upon inherent characteristics of the soil, crop response, use capabilities, or in terms of effectiveness of the programme.

A paper entitled ' Economics and the natural environment ' was presented by Mr J. A. Tosi (Jr). The substance of his paper was that the success or failure of any rational effort to achieve sustained economic growth through planned action depends upon the orientation of the project, well-defined goals and the priorities for which integrated surveys provide a rational and systematic means of devising both the broad and the locally detailed outlines of policy and action. For this, socio-economic, technological and environmental data must be brought together for which inter-disciplinary synthesis, based on wholly ecological methodology, is essential.

Prof. Dusseldorp gave an extempore talk on ' Social surveys and the natural environment '. For harmonious development of natural resources, a dynamic view of social changes involved in groups or tribes, social and geographic mobility, production factors and social development, efficient use of leisure time, social value of money, social relations with land, economic infra-structure, means of communications and transport, ambitions and expectations, is very essential. The sociologist's work should be fully integrated in the surveys of natural environment, otherwise most projects will not yield social and economic benefits expected of them.

The Plenary Session was presided over by Prof. R. A. J. Van Lier and the author of this report as the rapporteur of the seminar. Prof. Lier in his opening remarks stressed the importance of keeping

in view the cost-benefit ratios in integrated survey of natural resources, problems of the integration of natural sciences and social sciences in surveys, and administrative planning and execution of plans based upon integrated surveys.

As a member of the Unesco Advisory Committee on Natural Resources Research, the author of this report conveyed the thanks of Unesco and the foreign participants to the Netherlands Government, to Prof. A. J. V. Wheele and staff members of the ITC for organizing the seminar and making excellent arrangements for the comfort of the participants. On 27 April 1966, ITC arranged an excursion to a Reallotment Project in one of the Zuider Zee Polders. This area has been reclaimed from the sea during the past couple of years and is being turned into land for growing crops, vegetables and fruit orchards. This is surrounded by sweet water sea on all sides and is becoming a prosperous community organization.

On the final day, a meeting of the Board of Courses of ITC was held in which the author of this report participated and put forward suggestions to expand the scope of courses in aerial photo-interpretation, planning for land transformation, extension education and project evaluation.

Council of Scientific & Industrial Research: Meetings of the Board & Governing Body

At their meetings held in New Delhi on 18 and 19 December respectively under the chairmanship of the Prime Minister, Shrimati Indira Gandhi, the Board of Scientific & Industrial Research and the Governing Body of the Council of Scientific & Industrial Research sanctioned 75 new research schemes subject to availability of funds. Approval was also accorded to the following projects: (1) Pilot plant studies on the manufacture of citric acid (50 kg. per batch) by submerged fermentation at the Regional Research Laboratory, Jammu; and (2) an All-India Directory of Instruments and Manufacturers and Dealers of Instruments to be brought out by the Central Scientific Instruments Organization, Chandigarh, in collaboration with the Directorate-General of Technical Development. Financial assistance to the Wool Research Association, Bombay, and the Cement Research Institute of India, Bombay, on the same pattern as for other cooperative research associations, i.e. 50 per cent of the annual recurring and capital expenditure, was approved. A sum of Rs 25,000 towards publication costs of the National Report for the Upper Mantle Project by the Geophysics Research Board was sanctioned.

The holding of the following events during 1967 was approved: (1) A convention of research workers to be held under the auspices of the Physical Research Committee; (2) Symposium on Upper Mantle Project (National Geophysical Research Institute, Hyderabad, in collaboration with the Geological Survey of India, the Geophysics Research Board, the Indian Geophysical Union and the Geological Society of India); (3) Symposium-cum-lecture programme on Millimetre and Submillimetre Wave Spectroscopy (National Physical Laboratory, New Delhi, in collaboration with the University of Delhi); (4) Symposium on Site Investigations for Foundations (Central Building Research Institute, Roorkee); (5) Symposium on Scientific Results of the International Indian Ocean Expedition (New Delhi; under the joint auspices of the Indian National Committee on Oceanic Research and National Institute of Sciences of India); (6) Symposium on Quality Control in the Construction of Roads and Runways, along with a technical conference of the Chief Engineers of the Centre and the States dealing with highways, representatives of Engineer-in-Chief of Border Roads Organization and Chief Engineers of Municipal Corporations (Central Road Research Institute, New Delhi); and (7) Seventh Seminar on Electrochemistry (Central Electrochemical Research Institute, Karaikudi).

Fourth International Symposium on the Chemistry of Natural Products

M. M. DHAR

Central Drug Research Institute, Lucknow

THE Fourth International Symposium on the Chemistry of Natural Products, sponsored by the International Union of Pure & Applied Chemistry (IUPAC), was held at the Royal College of Technology, Stockholm, from 26 June to 2 July 1966. This symposium, like its predecessors, proved to be, both scientifically and socially, a completely successful event. It was attended by some 900 chemists from all over the world and around 300 papers were read.

The two plenary lectures of the symposium were given by Prof. Karl Folkers and Prof. E. J. Corey and signalled the start and conclusion of the symposium. Apart from Prof. Folkers' lecture, the opening ceremony included addresses by the President of the Symposium, Prof. Holger Erdtman, and by the past President of IUPAC, Lord Todd. The music of the eminent Scandinavian composers, Grieg and Larsson, admirably performed by a chamber music ensemble of the Stockholm Philharmonic Orchestra, added aesthetic pleasure to this memorable occasion.

In his lecture on the impact of natural products chemistry on medicine, Prof. Folkers expressed his appreciation to the host country by listing important contributions from Sweden in the field of drugs, namely xylocaine, *p*-aminosalicylic acid, heparin, streptokinase and dextran. He then touched on the impact of Rauwolfia and Vinca alkaloids, on the usefulness of quinine in drug-resistant malaria, on α-methyl dopa in the treatment of hypertension and on the use of 19-norsteroids in the control of fertility. From his own studies, the recently discovered coenzyme Q has been assigned an important function in mitochondrial electron transfer.

Prof. Corey's lecture on new methods for the construction of complex molecules attempted at emphasizing fundamental concepts, strategies and methods that are routinely employed in valid synthesis though often subconsciously. In the process of this synthetic dissection, he pointed out the significance of the symmetry of structures and gave as examples of the importance of symmetry considerations in synthesis, the syntheses of usnic acid and of pentacyclosqualene. The term synthon was introduced to mean interconvertible functional groups and the importance of new reagents and procedures to bring about chemical operations more efficiently was emphasized by examples from his own studies.

The other papers were read at the symposium in one of the following four sections: (1) Polysaccharides, (2) Structural elucidation of natural products other than macromolecular compounds, (3) Biosynthesis, and (4) Chemical taxonomy. There were two principal speakers in each section.

The two main lectures in the polysaccharide section were given by Prof. G. O. Aspinall and Prof. R. W. Jeanloz. In his lecture on the exudate gums, Prof. Aspinall indicated that these polysaccharides could be divided into three groups. The first group contained interior chains of D-galactose exemplified by the arabinogalactans from coniferous woods and gum arabic from *Acacia* species. The second group was characterized by interior chains of D-galacturonic acid and L-rhamnose and included the pectins, gum tragacanth and the gums from the *Khaya* and *Sterculia* genera. The exudate gums of the *Anogeissus* genus provided the only authentic example encountered so far of the third group whose inner chain contained alternating sequences of D-glucuronic acid and D-mannose.

Dr Jeanloz summarized his studies on the chemical structure of the cell wall of *Micrococcus lysodeikticus*. Peptide chains are linked to a glycan backbone composed of alternating units of D-glucosamine and muramic acid. To this structure is covalently linked a branched polysaccharide containing D-glucose, D-glucosamine and 2-acetamido-2-deoxy-D-manuronic acid.

The other papers in the section included the report on a new synthesis of polysaccharides using orthoesters, e.g. an araban was obtained by polymerizing 1,2,5-orthobenzoyl-β-L-arabinofuranose. The majority of papers expectedly were concerned with the chemistry of various polysaccharides from both plant and animal sources. The isolation of a carbohydrate serine compound was reported as also more studies on the position polysaccharide chains in fibrinogen, biological properties of ovalbumin and on the serological properties of various polysaccharides.

The main lectures in the section dealing with the structural elucidation of natural products were given by Prof. P. Karlson and Prof. K. Nakanishi. Prof. Karlson described the elucidation of the structure of Ecdysone, the insect molting hormone. This pentahydroxy ketosterol has now been synthesized by two other groups of workers.

Prof. Nakanishi discussed the chemistry of the bitter principles from the root bark of *Ginkgo biloba*, a plant directly traceable to the Palaeozoic fossil Cordaites. Substances designated as ginkgolides, A ($C_{20}H_{24}O_9$), B and M ($C_{20}H_{24}O_{10}$) and C ($C_{20}H_{24}O_{11}$) are remarkably acid stable, have no measurable UV absorption and are unusual hydroxylated lactones having a *tert*-butyl group.

This section contained more than half of the papers read at the symposium and structural studies on new natural products of all the usual types were well represented. The most significant point about these papers was the obvious impact the relatively new physical aids, mass and NMR spectrometry, have made on structural elucidations. Even in the cases of larger molecules such as oligopeptides, the mass spectrometry of the acylated esters has provided good information about the amino acid sequence.

The main lectures in the section on biogenesis were delivered by Prof. A. R. Battersby and Prof. F. Lynen. In a brilliantly delivered lecture, Prof. Battersby described studies which had identified a C_9 or C_{10} unit, originating from mavalonate, that got incorporated into the catharanthine, aspidosperma and corynanthane type of alkaloids.

Prof. Lynen delved into the biosynthetic pathway leading to polyacetyl chains. This process involves the enzymatic carboxylation of CoA to form malonyl CoA, which in turn is used in condensation reactions involving decarboxylation. Branched chain structures originate from acetyl CoA via Δ^3-isopentenyl pyrophosphate. Enzyme specificity in determining stereochemistry and chain lengths was discussed.

A large number of papers reported on the origin of various atoms and groups of atoms in a wide variety of structures. The location of labelled atoms in structures obtained in biosynthetic experiments with labelled precursors has in the past involved a large amount of degradative work. It is likely that mass spectrometry may be used to an increasing extent for this purpose since comparison of the spectra of the labelled and unlabelled compound can permit the location of the labelled atom.

It has been the practice in the past to use plant taxonomy as a guide to sources of useful plant materials. Chemotaxonomy is the reverse of this process where chemical constituents are used for the classification of taxa and no one has contributed more significantly to bring about this state of affairs than Prof. R. Hegnauer. In his talk, he surveyed the development of this subject and outlined the advantages a botanist could derive from a knowledge of the chemical constituents in a plant.

The other main lecture of the section on chemical taxonomy was given by Prof. B. L. Turner who indicated that micromolecular data were especially significant in the solution of systematic problems in which hybridizations and introgressions had been involved. As an example he discussed the solution of such problems in the genus *Baptisia* (Leguminosae) by a study of the flavanoids present in 16 species. These studies also provided new insight into the phyletic relationships of the species studied.

This symposium was arranged and run most efficiently. All participants left Stockholm deeply grateful to the Swedish National Committee for Chemistry, which organized the symposium and particularly to Dr (Mrs) G. Aulin-Erdtman, the General Secretary of the Organizing Committee, without whose untiring efforts the symposium could not have been the success it proved to be.

Nobel Prize Awards

The Nobel Prize in Physics for 1966 has been awarded to the French physicist, Alfred Kastler, in recognition of his work on optical resonance in atoms. First published in 1950, his work explains the technique for irradiating an atom to make it emit characteristic radiations revealing the nature of its structure. His work paved the way for the subsequent development of masers and lasers.

The chemistry prize has been awarded to Prof. Robert Mulliken of the University of Chicago for his molecular orbital theory, first reported in 1928. Repudiating the earlier established concept that atoms retain their identity when they form molecules, he put forth the concept that the balance of particles in atoms changes when they become part of molecules, and the electrons may take up orbital paths around the entire molecule instead of remaining in orbit around the atomic nuclei. Most of the significant work done on molecular structure since then is based on Mulliken's theory.

Prof. Francis Peyton Rous of Rockefeller University and Prof. Charles Brenton Huggins of the University of Chicago share the award in physiology and medicine for their pioneering research in cancer treatment. Dr Rous isolated for the first time a filterable virus from sarcoma and showed that the filtrate can cause identical cancer. Based on his research, virus-induced tumour has been produced in several mammalian species for experimental purposes. Dr Huggins pioneered operations for castration in the treatment of prostate cancer, and established that the benefit achieved is the result of hormonal change caused and not surgery itself. He has also successfully treated prostate patients without surgery using hormones. He has helped in the synthesis of variants of natural hormones for use in cancer treatment.

Measurement of Pressure in High & Ultrahigh Vacuum Regions Using Ionization Gauges: Part II—Cold Cathode Gauges

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SINCE ultrahigh vacuum techniques are finding applications in a number of new fields in recent years, it was considered worth while to critically review the recent developments in the design of ionization gauges for vacuum measurements. In Part I of this paper published[1] earlier, the author confined the attention mainly to hot cathode gauges. The present Part II of the paper reviews the recent developments in the field of the design and operation of cold cathode type gauges.

It has been pointed out in Part I of this paper that in the case of hot cathode type of gauges, apart from the other defects experienced in the accurate measurement of pressure in the high and ultrahigh vacuum regions, the lower limit of the pressure measurements in the conventional and inverted gauges has been set by the X-ray photocurrent and the inherent dark current respectively. It was also pointed out that some of the defects found in the case of hot cathode gauges such as production of photocurrent, vapour pressure of hot filament, etc., could be completely avoided by the use of cold cathode gauges. Even though the conventional cold cathode gauges could be used till some time back only for measuring pressures down to 10^{-5} mm. of mercury, because of the new designs developed by Hobson and Redhead[2-4], pressures down to 10^{-13} mm. of mercury could now be measured.

Defects of the Conventional Cold Cathode Gauges

A cold cathode ionization gauge comprises a discharge tube in which positive ions are produced by the phenomenon of discharge between the two electrodes. The whole assembly is placed in a magnetic field to increase the path of the electrons from cathode to anode so that the ionization current may be of measurable range. The first practical design of the gauge to measure the pressure in the region below 10^{-3} mm. of mercury was given by Penning[5,6] and hence it is also known as the Penning gauge (also sometimes called the Philips gauge). A sketch of the Penning gauge is shown in Fig. 1. The gauge is normally operated with a d.c. potential of 1 kV. applied between the anode and the cathode. The cathode plate is chosen to have low work function and very low sputtering rate and is usually made of zirconium or aluminium. The assembly of ring-shaped anode (R) and cathode plates (P) is placed between the poles of the magnet (H). The electrons which would otherwise move straight from cathode to anode are made to follow a spiral path by putting the gauge in a magnetic field of 250 oersteds. The gauge designed by Penning can be safely used for measuring pressures in the region of 10^{-3} to 10^{-5} mm. of mercury. At pressures above 10^{-3} mm. of mercury the ion current has been found to vary abruptly and does not bear any exact relation with pressure, and hence this gauge cannot be used at pressures above 10^{-3} mm. of mercury. It has also been reported that it is difficult to start a discharge below 10^{-5} mm. of mercury. For these low pressures, there may be a little delay or time lag for starting a discharge or the current may fail completely to build up, unless some external source is provided to start the discharge. Accordingly, McIlwraith[7] and Evans and Burmaster[8] have modified the gauge by introducing an external source to start the discharge, and pressures as low as 10^{-3} mm. of mercury could be measured with these modified gauges.

Thus the conventional cold cathode ionization gauges suffered from the following two main defects which prevented their use for the measurement of extremely low pressures: (i) most of the designs of the gauge failed to strike below a pressure of 10^{-5} mm. of mercury, and (ii) the ion collector electrode is exposed to a very high electric field causing field emission current which is indistinguishable from the ionic current and sets the limit of the measurable lower pressure.

Measurement of High Vacuum

It has been suggested by Beck and Brisbane[9] and Haefer[10,11] that the first defect in the conventional gauge could be overcome by an adequate design of the gauge in which electrons may be trapped in the discharge region. The second defect was removed by Hobson and Redhead[2] by introducing an auxiliary cathode which shields the ion collector from high electric fields. This auxiliary cathode also gives electrons by field emission and thus helps to start the discharge. The modifications

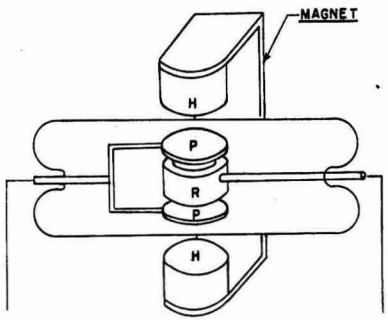

Fig. 1 — Cold cathode ionization gauge designed by Penning[5,6] [P, cathode plates; R, ring-shaped anodes; and H, magnetic poles]

suggested by them enabled the gauges to measure the pressure in the high and ultrahigh vacuum regions.

Measurement of Ultrahigh Vacuum

An extensive study of the cold cathode ionization gauges has been made by Hobson and Redhead[2-4,12,13] with particular attention to their behaviour at very low pressures. They have developed new designs of cold cathode type of gauges which are known as inverted magnetron and magnetron gauges. These gauges suggested for the high and ultrahigh vacuum measurements have the same basic principle of working as the Penning gauge. Their sensitivities are also of the same order of magnitude as that of the Penning gauge. The details of the electrode arrangement and the operation require special attention.

Inverted Magnetron Gauge

The basic arrangement of the electrodes in the inverted magnetron gauge[2,4] is shown in Fig. 2. The electrode system, a modification of the design of the cold cathode ionization gauges, has been suggested by Beck and Brisbane[9] and Haefer[10,11]. The ion collector is a closed cylinder with holes in the centre through which the anode and the shielding tubes pass. One of the main modifications of considerable importance at very low pressures in the introduction of the auxiliary cathode which is held at cathode potential to prevent field emission from the cathode. The typical values of the voltages and magnetic field applied in this gauge are 6 kV. and 2000 oersteds respectively.

The inverted magnetron gauge has been found to obey a relationship between collector current (i_p) and pressure (P) over a wide pressure range and is given by

$$i_p = CP^n$$

where C is a constant, and n lies between 1·1 and 1·4 for various gauges. The value of the exponent n is essentially independent of the anode voltage and approaches unity as the magnetic field is increased. If the guard ring had not been introduced, the field emission would be comparable with the main discharge current at very low pressures and would have set up a lower limit of pressure measurement in a manner analogous to the role of X-ray photocurrent in the hot cathode type gauges[14,15].

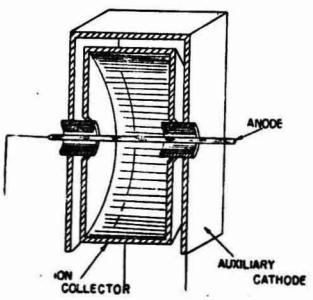

Fig. 2 — Cutway diagram of the inverted magnetron gauge[2]

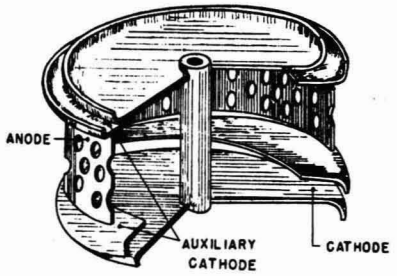

Fig. 3 — Design and electrode arrangement of the magnetron gauge[3]

Inverted magnetron gauge is most suitable for measuring pressures down to 10^{-13} mm. of mercury. For measuring pressures below 10^{-10} mm. of mercury, these gauges must be completely shielded to prevent them from the ambient light to reduce the photo effects and must be operated from a well-stabilized voltage supply. The sensitivity of this gauge has been reported to be of an order of magnitude higher than the Bayard-Alpert gauge[16].

Magnetron Gauge

The magnetron gauge[3,4] is based on similar principles as those of the conventional cold cathode ionization gauge and has a geometry similar to that of a normal magnetron. The sketch of this gauge is shown in Fig. 3. The cathode consists of a cylindrical spool to which are welded two circular disks. The anode is a short, open-ended cylinder which is perforated to improve gas flow through the gauge. The annular electrodes, called the auxiliary cathodes, are introduced between the anode ring and the cathode and are maintained at cathode potential for shielding the cathode assembly from the high electric field. The gauge is operated at a potential difference of 5-6 kV. between the anode and the cathode and using a magnetic field of 1000 oersteds applied along the axis of the spool. The gauge operates satisfactorily in a pressure range of 10^{-3} to 10^{-12} mm. of mercury.

Comparative performance of the magnetron and the inverted magnetron gauges — The magnetron gauge has a linear variation of ion current with pressure down to 10^{-10} mm. of mercury, while below it the variation is no longer linear. The magnetron gauge has a higher sensitivity and needs a smaller magnetic field and hence a smaller magnet size compared to the inverted magnetron gauge. On the other hand, the inverted magnetron gauge is more convenient because the same calibration curve can be used over the entire range of measurement of pressures below 10^{-10} mm. of mercury.

Radioactive Ionization Gauges

There are also some ionization gauges in which positive ions are produced by a stream of particles from a radioactive source. The rate of production of positive ions in any gas through which radioactive particles pass is directly proportional to molecular concentration or gas pressure. This novel

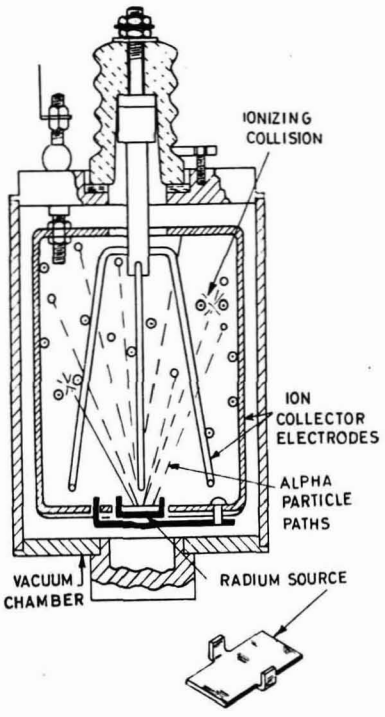

Fig. 4 — Alphatron gauge designed by Downing and Mellen[17]

idea was suggested by Downing and Mellen[17,18] who developed the radioactive ionization gauge (Fig. 4) which was designated as alphatron gauge because it used α-particles (from a radium source) as the source of ionization.

Because of the relatively low sensitivity of the gauge, the limiting pressure is of the order of 10^{-5} mm. of mercury. Beynon and Nicholson[19] have described a radioactive ionization gauge which could be used for measuring pressures over the range from 10 to 10^{-5} mm. of mercury. It is worth mentioning that Vacca[20], Roehrig and Vanderschmidt[21] and Spencer and Boggers[22] have used β-particles instead of α-particles because the former have lesser energy and are safer to work with. Radioactive gauge, though having the advantages of being a cold cathode gauge, has the disadvantage that precautions have to be taken to avoid radiation hazards.

Scintillation Gauge

Barnes[23,24] has suggested a cold cathode gauge which employs a field ion emitter source for the production of positive ions and a phosphor screen or scintillation crystal for detecting them. The light output is proportional to the number of ions and hence to the gas pressure. He has also recommended the use of a scintillation screen photomultiplier for detecting the small ion currents which would be found at pressures below 10^{-10} mm. of mercury. It has been pointed out by Nottingham[25], however, that the scintillation gauge suggested by Barnes cannot be used as a pressure measuring device because it cannot measure with reliability the pressure of such inert gases as nitrogen, argon, helium, etc.

Photomultiplier Ionization Gauge

A possible way of producing positive ions, which can be used for the pressure measurement, has been suggested by Riemersma *et al.*[26]. The sketch of the photomultiplier ionization gauge is shown in Fig. 5. A photomultiplier is used as a source of electrons and ultraviolet light is focused on to the first dynode through a quartz window. Photocurrent is multiplied at various stages and the electrons leaving the last dynode are accelerated by 100 V. to the electron collector. Collisions made in transit produce ions which are accelerated towards and collected at the ion collector. The variation of ion current with pressure is linear and the departure from linearity occurs only at pressures at which the electron mean free path becomes comparable with the gauge dimensions. The gauge has been reported to show a linear response down to a pressure of 10^{-10} mm. of mercury and a sensitivity lower than the Bayard-Alpert gauge.

Cold Cathode Orbitron Gauge

Very recently, Mourad *et al.*[27] have suggested a design of the cold cathode ionization gauge in which the electron path length has been increased just as in the magnetron gauge but without using a magnetic field. In this gauge, called orbitron ionization gauge, the electrons are ejected by a pointed cathode into the electrostatic field between

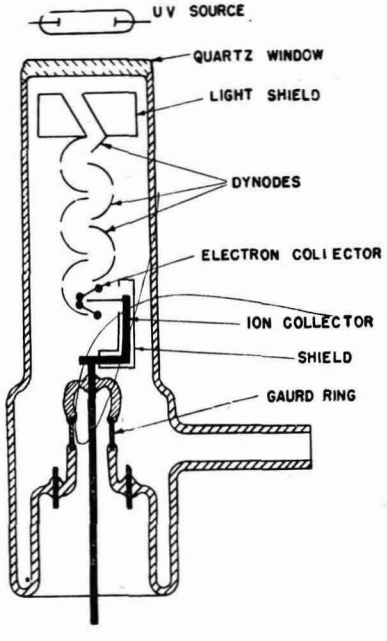

Fig. 5 — Sketch of photomultiplier ionization gauge[26]

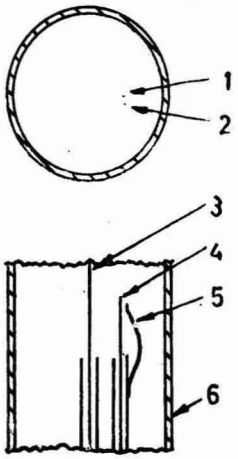

Fig. 6 — Sketch of the orbitron ionization gauge[27] [1, extractor; 2, point; 3, tantalum central wire; 4, tantalum extractor; 5, tungsten point (estimated radius, 0·25 μ); and 6, collector cylinder]

two concentric cylinders in such a way that they miss the anode and follow a very extended path[28] before they are collected by the anode. The arrangements of the various electrodes in the interior of the gauge are shown in Fig. 6. Pressure down to 5×10^{-11} mm. of mercury are measured with the help of this gauge.

Comparison between the Hot and Cold Cathode Gauges

The cold cathode gauges have the following advantages: (i) the X-ray photocurrent does not limit lower range of pressure measurement as in the case of the hot cathode gauges; (ii) the sensitivity is higher than that of the hot cathode type; and (iii) these gauges do not have hot filament to burn and so the photocurrent and the chemical changes caused in hot cathode type are commonly absent.

On the other hand, they also suffer from the following disadvantages: (i) stable operation can only be achieved in a limited range of voltage and magnetic field; (ii) the ion current versus pressure characteristics are in general nonlinear, and thus the gauges require a calibration over a wide range of pressures; (iii) at very low pressures (below 5×10^{-10} mm. of mercury) the gauge takes some time to strike; (iv) oscillations occur in the discharge at all pressures and care must be taken to see that their presence does not cause errors in the pressure measurement; and (v) pumping action of these gauges is greater than the hot cathode type under normal conditions of operation. This pumping is caused by a number of reactions taking place between the ionization gauge and the gas molecules in the system causing removal of the gases. The pumping action of the gauge would disturb the pressure distribution in the system and cause error in measurement.

Summary

The general techniques adopted in the measurement of high vacuum using cold cathode gauges have been reviewed. The principles involved in the construction and operation of various cold cathode gauges like the magnetron and the inverted magnetron gauges, radioactive ionization gauge, scintillation gauge, photomultiplier ionization gauge, and the cold cathode orbitron gauge are described and their limitations indicated. A comparison of the relative performance of the hot and the cold cathode gauges is made. Substantial improvements in the design and fabrication of the cold cathode gauges are necessary before they can be used to measure the same lower limits of pressure as the hot cathode gauges.

Acknowledgement

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Advances in Insect Population Control by the Sterile-male Technique

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WITH the successful eradication of the screw-worm, *Cochliomyia hominivorax* Coquarel, from the island of Curacao and South Florida by the sterile-male technique[1], the emphasis has shifted from chemical control of insects to eradication programmes using sterile-male technique. The technique involves, in short, the rearing, sterilizing either by radiation or by chemosterilants and release of sterile, sexually vigorous, long-lived and field-adapted males, in large numbers to exceed the natural population. The released sterile males compete with the indigenous males for females with the result that most of the females lay sterile eggs. By repeating this process for a few generations, complete eradication of the pest is achieved. The interest which the sterile-male technique has evoked can be judged from the fact that International Atomic Energy Agency convened a panel of experts, twice, once in 1962 and again in 1964, to discuss the various aspects and application of sterile-male technique and also to suggest future lines of research. These discussions led to the publication of two technical reports[2,3] which are the subject of this review.

Early Development in the Sterile-male Technique and Eradication of Screw-worm

The remarkable story of early development of sterile-male technique which proved highly successful in the eradication of screw-worm from Curacao[1] has been admirably reviewed by Bushland[4] and Weidhaas *et al.*[5]. The validity of such an approach in the control of insect species has again been demonstrated by the recent eradication of the melon fly[6], *Dacus cucurbitae* Coq., and oriental fruit fly[7], *Dacus dorsalis* Hendel, from the island of Rota, Marina Islands. It would be pertinent, however, to give a brief history of the development of the concept of sterile-male technique.

The use of sterile males for the control or eradication of screw-worm fly was first proposed by Knipling[8] in 1938. This led to the beginning of laboratory studies which showed screw-worm could be successfully sterilized by X-rays or gamma rays at 3000 rads applied to late pupae and that sterile males emerging from irradiated pupae could effectively compete with normal males[9,10]. In 1954-55 screw-worms were eliminated permanently from the islands of Curacao by releasing 400 irradiated males per square mile per week on 170 sq. miles area. Subsequently, in the south-eastern screw-worm eradication programme, the mass rearing[11], irradiation and packing techniques[12] were perfected and the screw-worm was eradicated successfully from Florida[13,14]. The highlight of the programme was setting up of a fly rearing factory to produce up to 50 million screw-worm flies per week. This required gigantic efforts by way of facilities and food for rearing flies: 40 tons of meat a week, 4500 gallons of blood, 9600 gallons of water, 65 gallons of plasma and 35 gallons of honey. Total eradication of the pest, however, was achieved in 2 years' time, at a cost of $ 8 million. Since the control of screw-worm led to an annual saving of approximately $ 20 million to livestock industry, the savings paid for the programme. Latest reports[3] about a still bigger attempt to eradicate the screw-worm from Texas and South-western United States (south-western screw-worm eradication programme) not only provide some staggering statistics but also confirm the absolute validity of the sterile-male technique in field trials.

Requirement for the Sterile-male Technique

The possibilities of controlling or eradication of insects by releasing sterile males have very exacting requirements[3,8,15-20] and require a thorough scientific study of various aspects of insect physiology, ecology and population dynamics. Before any large-scale eradication programme is undertaken, it is essential to have information on the following biological aspects of the insect pest: (i) method for large-scale rearing of the insect pest in laboratory must be available; (ii) the insect species must be such that it can readily be dispersed and that the males are able to effectively search the opposite sex and are able to mate in competition with native population; (iii) method for sterilizing the pest either by radiation or by chemosterilants must be worked out keeping in view that it should not affect the mating behaviour and vigour of the irradiated insects. It would be better if some sort of test for the sexual aggressiveness of the irradiated insect be made along the lines suggested by Baumhover[21]; (iv) the species to be controlled must have a low population, otherwise the pest population should be brought to a low level by insecticide treatments; (v) the area from which the insect is to be eradicated must be isolated and reasonably protected against reinfestation; (vi) the males to be released must not be harmful to man, animal or plants. Easy method of separation of sexes must be available in cases where the female is harmful. For example, the release of female mosquitoes in large numbers would create very unhealthy condition for man and animals within the area; (vii) a thorough knowledge of the habits and ecology of the insect pest such as number of annual generations, the length of various stages under different conditions and population per unit area is essential.

Methods of Inducing Sterility in Insects and the Factors Affecting it

One of the basic tenets for the successful use of sterile-male technique is that the insects should be

sterile but normal in every other respect. Although sterilization in screw-worm eradication programme was done by irradiation, there are reports in literature that chemosterilants[22-25] and genetic manipulations[26,27] can also be successfully utilized for this purpose. Irradiation is usually done either by X-rays or more economically by ^{60}Co and the insects are irradiated either in pupal or adult stage when the imaginal tissues have differentiated and the gonads are in a state of active cell division. This is the ideal time for irradiation because at this stage there are the greatest differences in sensitivity between the somatic and gametic tissues.

The radiation dose required to produce sterility varies with insect species and sex. A comparison of radiation dosages required by twenty different insect species for complete sterilization show that it varies anywhere between 5000 and 75,000 rads[28-35]; highest dose (75,000 rads) was required to sterilize human body louse, whereas for most of the insect species studied the range varied between 5000 and 10,000 rads. Generally males of the species are more susceptible to irradiation; however, in case of codling moth (*Carpocapsa pomonella* L.)[33], leaf roller (*Platynosa stultana* Walsingham)[35] and potato tuber moth (*Gnorimoschema operculella* Zeller)[36] the females were observed to be more susceptible to irradiation than males. It has also been observed that anoxic conditions and the presence of nitrogen and carbon dioxide at the time of irradiation have distinct radioprotective effect, whereas oxygen has very little effect[37]. Thus it was observed that a dose of 11,000 rads was required to sterilize females of the screw-worm in carbon dioxide atmosphere, whereas only 5500-6250 rads were sufficient under oxygen or air under forced ventilation[38].

Besides irradiation, sterility could also be produced in insect pest species using chemosterilants[22-25]. The advantage of this alternative method of producing sterility in the pest population is that it is more flexible and economical. Irradiation methods require a costly permanently fixed ^{60}Co source and sterility can be induced only in the laboratory reared or field collected insects which must be brought to the laboratory for irradiation. The advantage of using chemosterilants is that they can be used in the field as baits or residual films, thus sterilizing the population in their natural habitat which may preclude the necessity of rearing large number of insects[19]. Most commonly used chemosterilants are alkylating agents and their radio-mimetic effect is considered to be due to their reaction with deoxyribonucleic acids in chromosomes. Although a large number of compounds possessing sterilant activity are known[23-25], a few which have been successfully utilized in the laboratory and under field conditions are Tepa [tris-(1-aziridinyl)-phosphine oxide], Metepa [tris-(2-methyl-1-aziridinyl)-phosphine oxide] and Apholate [2,2,4,4,6,6-hexakis-(1-aziridinyl)-2,2,4,4,6,6-hexahydro-1,3,5,2,4,6-triazatriphosphorine][39-41]. Preliminary field studies for eradicating bollworm, *Anthonomas grandis* Bohmann, by releasing Apholate sterilized males have given very encouraging results[42]. It may, however, be pointed out that at least in some species of insects resistance to chemosterilants can develop and may thus force the use of irradiation as the method of producing sterility[43].

The possibility of manipulating genetic mechanisms already present in the natural population for producing hybrid sterility and propagation of deleterious genes has been discussed[3,26,27]. This method of population control, however, is still in the experimental stage.

Aspects of Insect Nutrition Concerned with Mass Culture of Insects

The successful application of sterile-male technique for the eradication of insect pests is dependent on the continuous supply of large number of insects of the pest species for irradiation purpose. Since the irradiation of the insects is generally done in pupal stage, the insects are normally reared in the laboratory. For this a very thorough knowledge of various aspects of insect nutrition is required. A survey of the various reviews on insect nutrition[44-51] reveals that although the basic nutritional requirements of various insects are quite similar, it may differ in the requirements of certain 'odd' chemicals which are not nutritional requirements in strict sense but must be present in the diet of some insects in order to obtain a good feeding response. Further, differences which are important from the point of view of mass culturing of insects are ratio of one nutrient to another in the culture medium and its physical texture. The large-scale rearing of insects is possible only if a good and continuous supply of fertile eggs can be obtained. And since the nutritional requirement of insects in adult stage varies significantly, a thorough understanding of it is essential. Many insects, e.g. moths and warble flies, do not require a complex diet for proper maturation of ovaries and egg laying. Others may require only water or water plus a carbohydrate which lengthens longevity and hence greater yield of eggs from the culture. Still in some others the nutrient transfer from the larval stage may be practically nil and, therefore, may have a complex requirement for proper egg laying.

Insect Species being Considered or Used in the Programme of Sterile-male Releases

Fruit Flies

The trypetids occur as the most destructive fruit and vegetable pests present in most tropical, subtropical and some temperate areas of the world. Earlier work in the United States has indicated that three species of fruit flies, melon fly (*Dacus cucurbitae* Coq.), oriental fruit fly (*D. dorsalis* Hendel) and the Mediterranean fruit fly (*Ceratitis capitata* Weidemann), could be eradicated by the sterile-male technique[52,53]. Doses between 6700 and 10,000 rads of gamma rays from ^{60}Co administered to pupae gave good sterilization of both sexes without undue loss in effectiveness of insemination by the male. Recent indications are that chemosterilants, viz. Tepa, Apholate and Tretamine, sterilize these three species of fruit flies as well as Mexican fruit flies (*Anastrepha ludens* Loew) as effectively and efficiently as ionizing radiations[41,54]. Melon fly[6] and

oriental fruit fly[7] have been eradicated from the island of Rota, Marina Islands, and the work has been extended to include islands of Guam and Saipan-Tinian in the Pacific for the eradication of these pests[3,55]. It may be mentioned here that the eradication of oriental fruit fly[7,56] from the island of Rota was achieved not by sterile-male technique but by what is more commonly known as 'male-annihilation' technique. The technique, in short, involves air dropping small baits containing attractants for males with some toxicants, thus completely annihilating the male population. The difference between the sterile-male technique and the male-annihilation technique is simply that in former the females are allowed to mate with sterile males and in the latter the females are denied the luxury of male company with the result that the species is not able to reproduce and in time is completely eradicated. Oriental fruit fly, however, was eradicated from Guam Island by the use of irradiated flies[55]. Preliminary studies have also been initiated in Israel, Tunisia, France and Costa Rica with a view to eradicating Mediterranean fruit fly. Work in Israel and Tunisia pertained to mass rearing of Mediterranean fruit fly under local condition, and at both places, by a slight modification of Hagan's formula[2] for rearing fruit flies, it has been possible to produce up to 1 million flies per week at reasonable cost.

Studies on irradiation dosages and mating behaviour carried out in Costa Rica and France with Mediterranean fruit fly indicated that 7-9 days old pupae when irradiated with a dose of 10,000 rads produce 100 per cent sterility in males. The irradiated males, however, did not compete equally in mating with normal males. In laboratory, the population of 1 irradiated male : 1 normal male, the egg fertility was found to be 74 per cent instead of theoretical 50 per cent and that female fertility could be reduced by 89 per cent only when the ratio of irradiated males to normal males reached 39 per cent. These studies thus suggested that in any programme to eradicate Mediterranean fruit fly field population will have to be flooded with irradiated males at the rate of 39 to 1 or higher.

Other species of fruit flies on which the work is going on with a view to eradicating the species are olive fly (*Dacus oleae* Gmelin) and Queensland fruit fly (*Dacus tryoni*). Studies on olive fly, which is a serious pest in Greece, Israel and Italy, were mostly confined to mass rearing techniques[3]. Similarly, mass rearing techniques were perfected so as to produce anywhere between 5 and 10 million Queensland fruit flies per week on an improved carrot-yeast medium of Christenson *et al.*[57].

Tsetse Flies

Sleeping sickness of man and Nagana of domestic animals is transmitted by the tsetse fly, *Glossina* sp., in Africa. Although there are 25 species of *Glossina* in Africa infesting approximately 25 million square miles area, only a few species are efficient vectors of trypanosomiasis, *G. morsitans* West. and *G. pallidipes* Aust. are active in east and *G. palpalis* R-D. in West Africa[58].

Since it is rather difficult to control the fly by insecticide, the possibility of using sterile-male technique for its eradication was indicated by Ray[59] as early as 1957. Preliminary investigations on the possibility of sterilizing the fly by gamma radiation showed that the flies could be sterilized with a dosage of 6000-12,000 rads though it reduced their longevity[69]. Similarly, sterilization of flies with chemosterilants Metepa, Apholate and Tepa indicated that Tepa was very promising in inducing sterility in *Glossina morsitans* and that there was only a moderate decrease in the survival rate of the adult fly. It may, however, be pointed out that the greatest bottleneck in the tsetse fly eradication programme is that as yet no satisfactory method of large-scale rearing of vigorous flies is available and most of the studies done so far in this direction have been made on field collected adult flies and pupae collected from the breed.

Mosquitoes

Laboratory studies to show the possibility of using sterile-male technique for the eradication of mosquito, *Anopheles quadrimaculatus*, gave promising results and it was shown that various stages of adults could be sterilized by 8865-13,000 rads of ^{60}Co gamma rays and that the irradiated males competed successfully with normal males for mating with the available females[61]. However, when the technique was applied in field the results were disappointing because of the lack of competitiveness of the sterilized males[62] and also because the behaviour of laboratory reared males was found to be different from that of the wild population. The laboratory reared males were found to be incapable of mixing themselves with the wild population[63]. Similarly, the work of Morlan *et al.*[64] and Fay *et al.*[65] with yellow fever mosquitoes, *Aedes aegypti* L., indicated that sterilized males were less competitive than the normal wild population and also pointed out the desirability of having additional information on male dispersal under field conditions. Small-scale field trials carried out in India by Krishnamurty *et al.*[66] for the control of the mosquito, *Culex fatigans*, showed that it may be possible to utilize the sterile-male technique for the control of this species. This study is of importance in that it not only emphasizes the desirability of gathering additional knowledge on mosquito biology but also reveals the difficulties that will be encountered in utilizing this technique in a developing country with limited resources.

Other Species

With the success of sterile-male technique in the screw-worm and fruit flies, work has been initiated to produce sterility in various species of insects by irradiation so that this information could be utilized eventually in the sterile-male technique eradication programme. Lewis and Eddy[31] showed that both sexes of horn fly, *Haematobia irritans* L., could be sterilized by 5000 rads by irradiating pupae with a ^{60}Co source and that the longevity of adults was not affected by this treatment though the males produced from the irradiated pupae were not fully competitive with normal males for females.

Adult males (< 24 hr old) of European corn borer, *Ostrinia nubilalis* Hubner, could be sterilized by X-rays at a dose of 32,000 rads without reducing either the longevity or their ability to compete with normal males for mating[28]. Pink bollworm, *Pectinophora gossypiella* Saunders, has also been the subject of a number of studies. The effect of gamma radiation from a ^{60}Co source indicated that the males are more resistant to irradiation than females[67] and that the longevity of irradiated males was nearly 50 per cent of the normal. Because of this adverse effect of gamma irradiation on males chemosterilants were tried to induce sterility and the results indicated that Metepa could be successfully used for sterilizing the males and that the Metepa sterilized males were fully competitive with normal males in mating with females[68,69]. As the technique for mass rearing for pink bollworm has already been perfected[70], it would appear that the stage is now well set for the total eradication of this pest from USA. Since mass rearing is a must for the success of any sterile-male technique eradication programme, the large-scale rearing procedures for a number of insects have also been worked out recently. Shorey and Hale[71] have perfected methods for mass rearing of nine noctuid species, the cabbage looper, *Trichoplusia ni* Hubner, the alfalfa looper, *Autographa californica* Speyer, the yellow-spotted army-worm, *Prodenia ornithogalli* Guenee, the beet army-worm, *Spodoptera exigua* Hubner, the bollworm, *Heliothis zea* Boddie, tobacco bud-worm, *H. virescens* F., *H. phloxiphaga* G. & R., the army-worm, *Pseudaletia unipunctata* Haworth, and the variegated cut-worm, *Peridromia saucia* Hubner, on a simple artificial diet economically. Similarly, a simple large-scale rearing technique has been developed for the rearing of tobacco horn-worm, *Protoparce sexta* Johansson, at a cost of approximately $ 1.00 for 1000 horn-worms[72].

Future of Sterile-male Technique

Although the sterile-male technique has been successfully used to eradicate screw-worm flies and fruit flies in the USA, the technique was a total failure in eradicating blow fly, *Lucilia sericata*, from Holy Islands in UK, in spite of an estimated 5 : 1 preponderance created by releasing sterile males[73]. The probable cause of this failure might have been either the reduced vigour of the irradiated males or inadequate sterilization.

Similarly, the efforts to control the Mediterranean flour moth, *Anagasta kuhinella* Zeller, by sterile-male technique in UK had to be abandoned primarily because of the poor dispersal of the released males and reinfestation. Since the sterile-male technique is species specific, other pests would not be eliminated and would in any way require occasional fumigation or insecticide treatment. It was, therefore, concluded that this technique is unsuited for the economic control of the Mediterranean flour moth[74]. Since these arguments can also be applied for other stored grain pests it would appear that sterile-male technique is unlikely to be effective for the control of these insects[2]. However, P adhan *et al.*[75] suggest that this technique can still be used in the control of stored grain pests.

Despite the failure of the irradiated blow flies and the Mediterranean flour moth in UK, the sterile-male technique presents greater possibilities for pest control. It should, however, be pointed out that the technique cannot be applied as a general method for insect control. Moreover, it should also be recognized that an immense amount of research in behaviour, ecology and mass rearing of the insect species to be controlled is necessary before it can be ascertained if the method has any practical potentialities. Keeping this in view the method will probably be used on only a few species of insects and under certain type of situation and also when it is economical in the long run. Nevertheless, the method is promising and is another useful weapon which could be used either alone or in conjunction with other methods for the control of insect pests of medical, veterinary and agricultural importance.

In spite of heavy initial outlay, the sterile-male technique may prove useful in controlling some of the most difficult insect pests in India which seriously affect agricultural production. Efforts directed towards controlling 'Jowar' stem borer, *Chilo zonellus* Swinhoe, which seriously affects sorghum and maize production in the country, may perhaps pay handsome dividends, as it has been practically impossible to control this pest by conventional insecticidal treatments. For this a method of mass rearing of *Chilo* in laboratory and an efficient method of sterilizing insects either by radiation or by chemosterilant will have to be worked out before taking up the control work. Work already done by Pant *et al.*[76,77] may prove useful in formulating a diet for mass rearing of this insect. Similarly, the technique may prove useful in the control of goat warble-fly, *Hypoderma crossii* Patton, a serious pest of goats in this country. Although no reliable estimates of the monetary loss due to the damage of goat-skins by this insect has been made, the loss must be extensive because India produces nearly one-third of the total world production of hides and skins[78]. Because of the non-availability of any suitable method of rearing goat warble-fly in the laboratory, the work done on the control of tsetse fly by sterile-male technique may provide some useful information for initiating research on this insect.

Summary

The history and basic principles of the sterile-male technique for the eradication of insect pest species have been described. A critical evaluation of various methods of inducing sterility in insects and the aspects of insect nutrition concerned with mass rearing of insects have been discussed. A brief account of the researches going on in different parts of the world on various aspects of the technique has been given and future lines of work indicated.

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ห้องสมุด กรมวิทยาศาสตร์

Naturally Occurring Terpenoid Furanolactones

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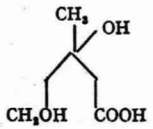
FURAN derivatives are widely distributed in nature particularly among the non-nitrogenous active principles[1], some of which being extremely bitter. They fall into a number of classes which differ fundamentally in structure and probably have different biogenetic origin. In some natural products the furan moiety is fused to an aromatic ring giving rise to the heterocyclic benzofuran. Furan ring may be attached to a terpenoid at the α- and β-positions as found in linderane[2-5], cafestol[6-9], etc. Another class consists of simple α-substituted furans, e.g. α-methyl furan, and one is inclined to suggest that they may be related to carbohydrates. In the fourth major class of naturally occurring furan derivatives, the furan ring is β-substituted. This class contains a large number of compounds of varying molecular complexity. Unlike the α-substituted furan system, the β-substituted furan group has an isoprene carbon skeleton (I) and it is, therefore, possible that in nature the β-substituted furan unit arises from mevalonic acid (II), the biogenetic precursor of the terpenes and in fact examination of the structure of natural products containing a β-substituted furan ring shows that most of them are typical terpenes.

(I)



(II)

Of particular interest in more recent years have been the compounds involving a C_{26} carbon skeleton with a 17-3′-furylsubstituent. The realization of the potentialities of these new members, which are in large numbers being isolated from plant kingdom, has recently stimulated much chemical work. Detailed structural investigation of these compounds which has been facilitated by NMR spectroscopy, mass spectrometry and X-ray spectoscopy has shown that various genera of meliaceous and rutaceous plants (Table 1) can give rise to an interesting group of active principles containing the same characteristic pattern derived from butyrospermol. It may be due to their descent from common ancestor and in fact Meliaceae and Rutaceae family are included under the same order[10], Rutales. Even at the risk of criticism for wishful thinking one is tempted to believe at this stage that the presence of the basic skeleton of 26-carbon atoms has some taxonomic significance in the dicotyledonous trees, like the phenolic components[11-13] present in *Pinus* genus. As there is a real harmony between chemical and botanical character, the knowledge of the biosynthesis of these skeletally related natural products, which are enjoying a consistency in stereochemical control, may be of great value to build up gradually a system of chemical relation contributing to the course of plant evolution.

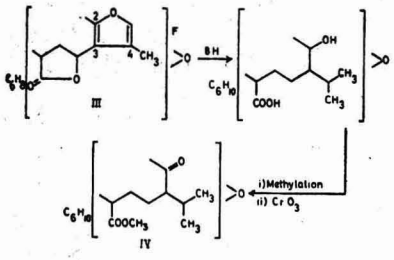
The present review is devoted to the chemistry of the terpenoid furanolactones and in more detail the advances of the last decade. It is more convenient to treat this group of compounds under the general headings: sesqui-, di- and tetranortriterpenoids, according to their carbon skeleton.

Sesquiterpenoid Furanolactones

The root of *Lindera strychnifolia* Vill (Fam.: Lauraceae) contains three closely related furanolactones: linderane[3], linderolactone[4] and isolinderolactone[4]. Takeda and coworkers have established the constitution of these compounds.

Linderane

Linderane, $C_{15}H_{16}O_4$, m.p. 190-91°, $[\alpha]_D$+180°, $\lambda_{max.}$ 214 mμ, ε 6770, is an interesting monocyclic sesquiterpenic lactone. The nature of four oxygen atoms as one γ-lactone, one furan (1556, 1616, 3060 and 3130 $cm.^{-1}$) and one epoxide functions together with an isolated double bond has been settled from IR and NMR spectra coupled with suitable preparative methods. It also shows the presence of a methyl signal on a furan ring (doublet at τ=8·00, J=1·1 *cps*) and a tertiary C-methyl group (τ=8·43). The reductive degradation products of linderane on catalytic dehydrogenation afford vetivazulene together with an unidentified naphthalene derivative. All these facts are in keeping with the presence of a ten-membered monocyclic system in the molecule.



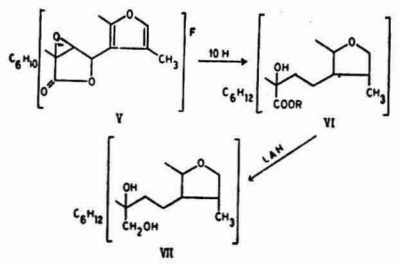
Linderane on hydrogenation gives three important acids due to hydrogenolysis of the lactone ring of which two have been formulated as decahydrolinderane, $C_{15}H_{26}O_4$, and octahydrolinderane, $C_{15}H_{24}O_4$. The third product being an oil was isolated as a ketomethyl ester, $C_{16}H_{26}O_4$, (IV) after methylation followed by oxidation with chromic acid. The formation of this compound, having

TABLE 1 — ACTIVE PRINCIPLES ISOLATED FROM MELIACEAE AND RUTACEAE FAMILY HAVING CHARACTERISTIC PATTERN DERIVED FROM BUTYROSPERMOL

Compound	Source	Parts	Family	Ref. to isolation
Aglaiol	*Aglaia odorata* Lour	Leaves	Meliaceae	129
Andirobin	*Carapa guianensis* Aubl	Seeds	do	108
Anthothecol	*Khaya anthothecac* DC.	Timber	do	106
Carapin	*Carapa procera*	do	do	141
Cedrelone	*Cedrela toona* Roxb.	do	do	126, 127
7-Deacetoxy-7-ketogedunin	*Carapa guianensis* Aubl	Seeds	do	108
	Cedrela odorata Linn.	Timber	do	114
7-Deacetoxy-7-ketokhivorin	*Khaya senegalenis*	do	do	106
Deacetylnomilin and deoxylimonin	Citrus fruits	Seeds	Rutaceae	130
Dihydrogedunin	*Guarea thompsonii*	Timber	Meliaceae	109
Entandrophargmin	*Entandrophragma cylindricum* Sprague	do	do	99
Flindissol	*Flindersia maculosa* Lindl; *Flindersia dissosperma* Domin	Bark Bark and leaves	Rutaceae do	125 125
Fraxinifolin	*Evodia fraxinifolia* Hook. f	Bark	do	131
Gedunin	*Entandrophragma angolense*	Timber	Meliaceae	99
Indicic acid and katonic acid	*Sandoricum indicum*	do	do	139
Khivorin	*Khaya grandifolia*; *Khaya ivorensis*	do	do	105
Limonin	Citrus fruits	Seeds	Rutaceae	74, 87, 88, 132
Maxicanolide	*Cedrela maxicana*; *Cedrela odorata*; *Carapa procera*	Timber	Meliaceae	106, 140
Methyl angolensate	*Entandrophragma angolense*; *Entandrophragma utile*; *Guarea thompsonii*; *Cedrela odorata*	do	do	99, 106, 109, 114
Mahogenin	*Swietenia mahogany* Jacq	Seeds	do	133
Nimbin	*Azadirachta indica* Juss. Syn. *Melia azadirachta* Linn.	Seed oil	do	134, 135
Nomilin	Citrus fruits	Seeds	Rutaceae	88
Obacunone and obacunoic acid	do	do	do	88, 100
7-Oxodeacetoxydihydro-α-gedunol	*Guarea thompsonii*	Timber	Meliaceae	109
Salanin	*Azadirachta indica* Juss. Syn. *Melia azadirachta* Linn.	Seed oil	do	136
Swietenine and swietenolide	*Swietenia macrophylla* King	Seeds	do	137
Turraeanthin	*Turraeanthus africanus*	Timber	do	138
Utilin	*Entandrophragma utile*	do	do	99

an isopropyl group (NMR spectrum) in it but without any loss of carbon atoms, can be explained in terms of hydrogenetic cleavage of the 2,3-substituted-4-methyl furan ring in the molecule of linderane which is allylic to the lactonic oxygen atom.

Decahydrolinderane, $C_{15}H_{26}O_4$ (VI; R=H), is a saturated monohydroxy acid having no ethereal oxygen or isopropyl grouping in it, which points out that during the hydrogenolysis of the lactone ring, the reductive cleavage of the 1,2-epoxy group occurs liberating thereby the hydroxyl group which is α- to the carboxyl since the methyl ester (VI; R=Me) by lithium-aluminium hydride reduction is converted to an α-glycol (VII).

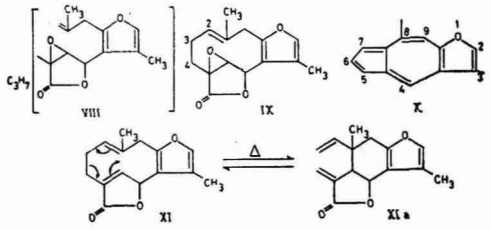


Linderane contains one ethylenic linkage which has been formulated as —CH=CMe— from the oxidative degradation studies. Moreover, dihydroxylinderane obtained by treating linderane with osmium tetroxide gives a monoacetate which still contains a hydroxyl group. This monoacetyl derivative (τ=5·02) undergoes dehydration yielding thereby a linderane monoacetate having a conjugated double bond ($\lambda_{max.}$ 249 mμ, ε 7290) lacking OH-band in its IR spectrum. This obviously suggests the elimination of the tertiary hydroxyl group in dihydroxylinderane with the formation of a double bond in the linderane, the double bond being in conjugation with the furan ring. Thus (V) may be expanded to (VIII).

Linderane gives succinic acid when it is subjected to ozonolysis followed by oxidation with permanganate which indicates the presence of $-CH_2-CH_2-$ system in the molecule and this unit constituting the molecule of linderane has been placed at the remaining C_3 and C_4 positions. All these experimental results establish linderane unequivocally as (IX).

The formation of ujacazulene[8] from linderane on palladium-charcoal dehydrogenation, which is

shown to be identical with 3,8-dimethylazuleno-(6,5-b) furan (X) adds further evidence for the above structure.



Linderolactone and Isolinderolactone

In a recent communication[4] the chemistry of linderolactone, $C_{15}H_{16}O_3$, and isolinderolactone has been studied. From a consideration of the available chemical evidence and spectroscopic analysis as well as of the formation of ujacuzulene (X) on dehydrogenation, linderolactone has been shown to have the structure (XI) and isolinderolactone being a valence tautomer[5] of the former has been represented by (XIa).

The ring structure of linderane is of biogenetic significance. The carbon skeleton of linderane, like those of pyrethrosin[14] and costunolide[15], can be constructed from a farnesol chain through a ten-membered ring intermediate (XII) and it suggests that linderane belongs to eudesmane type sesquiterpenoid (Chart 1)

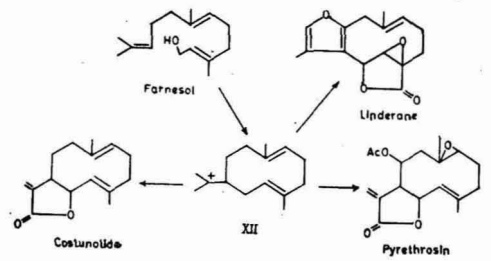


Chart 1 — Reactions suggesting that linderane belongs to eudesmane type sesquiterpenoids

Marrubiin

Marrubiin[16-25], $C_{20}H_{28}O_4$, m.p. 160°, $[\alpha]_D$+35·8°, $\lambda_{max.}$ 212 mμ (ϵ 5623), the bitter principle of horehound (Fam.: Labiatae), is the first diterpenoid lactone in which the presence of the β-substituted furan system was recognized and it has been shown to have the constitution (XIII) by Cocker and coworkers[26]. This formulation was substantiated by further investigations. The C_{14} ketolactone (XIV) from marrubiin by ozonolysis is converted into the cisoid dienone (XV) by the oxidation of the derived hydroxy acid with chromium trioxide followed by selenium dioxide (Chart 2). This eventually confirms the relative position of the two oxygen atoms of the free hydroxyl and the hydroxyl of the lactone. The carbon skeleton of marrubiin has been settled (Chart 2) by the transformation of the enol lactone (XVI) into the compound[27] (XVII) which has been obtained by degradation of ambrein[28,29] (XVIII).

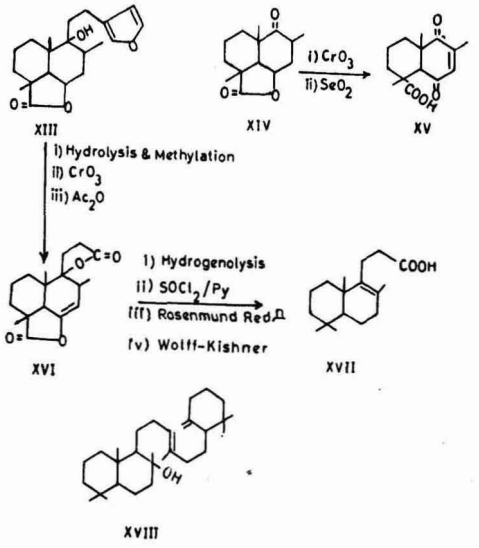
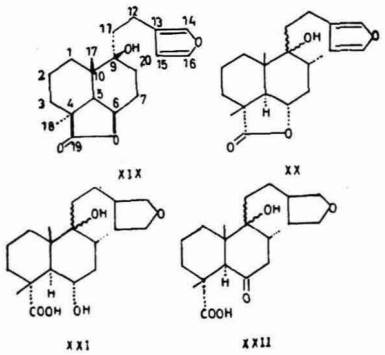


Chart 2 — Reactions confirming the carbon skeleton of marrubiin (XIII)

Diterpenoid Furanolactones

Marrubiin, columbin, chasmanthin, jateorin, tinophyllone, sciadin and sciadinone are the members of this group. There are three main classes of diterpenoids based on their dehydrogenation products which are (i) retene, (ii) pimanthrene and (iii) 1,7,8-trimethylphenanthrene. But the diterpenoid furanolactones where the furan rings are β-substituted yield a different product, viz. 1,2,5-trimethylnaphthalene either on direct dehydrogenation or on dehydrogenation of suitable derivatives. So they constitute a class by themselves. This growing family of diterpenoid compounds, in addition to their considerable chemical interest, obey the Isoprene Rule with the exception of columbin, jateorin, chasmanthin and tinophyllone.

In ambrein, rings A and B have *trans* configuration which led Cocker and coworkers[26,30] to suggest similar A/B *trans*-fusion in marrubiin and thus demonstrate the *trans*-relation between the methyl group and the hydrogen atom at C_5 and C_{10}. This along with the application of Hudson's Lactone Rule, as developed by Klyne[31], would indicate the configuration of marrubiin as shown in (XIX). However, later workers[27,32,33] did not accept the configurational assignments particularly at positions 4, 6, 8 and 9. The formation of tetrahydromarrubic acid (XXI) by the reduction of ketoacid[34] (XXII) with lithium-ammonia in methanol shows that the hydroxyl group at C_6 has α-configuration[35]. (XXI) is also obtained by the sodium or potassium borohydride reduction of



XIX XX

XXI XXII

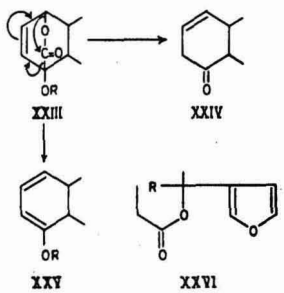
(XXII), a fact which can be explained by assigning an α-configuration to the carboxyl group at C_4. Marrubiin on this basis can then be formulated as in (XX). But recently Fulke and McCrindle[36] have advanced reasons in favour of the steric structure (XIX), based mainly on the studies of NMR spectra of anhydrotetrahydromarrubiin and its derivatives.

Columbin

Colombo root (Fam.: Menispermaceae) contains four bitter principles: columbin, chasmanthin, jateorin and the isomeric palmarin. These compounds have been investigated in many laboratories[37-41]. However, the work by English school[42] has elucidated the structures of these compounds of which columbin is the main bitter principle. Mention may be made here that tinosporine[43], recently isolated from *Tinospora cordifolia* Miers (Fam.: Menispermaceae), has been found to be identical with columbin from the comparison of X-ray diffraction patterns besides other physical and chemical data.

Columbin, $C_{20}H_{22}O_6$ (m.p. 192-5°, $[\alpha]_D$ +31·4°, $\lambda_{max.}$ 209 mμ, ε 6026) is easily isomerized by base to isocolumbin. Both the substances behave as dilactones and give the same monoacetyl derivative. They, therefore, contain one hydroxyl group which is rather acidic but neither enolic nor phenolic. Columbin and isocolumbin both lose one molecule of carbon dioxide on melting giving the monolactones decarboxycolumbin and decarboxyisocolumbin respectively. These cannot be acetylated or methylated and, therefore, decarboxylation involves the removal of the hydroxyl group as well. However, isocolumbin acetate and O-methylisocolumbin both decarboxylate to products which still contain an acetyl and a methoxyl group respectively. On the other hand, dihydrocolumbin does not decarboxylate. These reactions can be explained in terms of the partial structure for columbin (XXIII; R=H). The spectra of decarboxycolumbin (XXIV), decarboxyisocolumbin acetate (XXV; R=Ac) and decarboxy-O-methylisocolumbin (XXV; R=Me) are in agreement with the structure indicated. Further confirmation of the partial structure (XXIII) was obtained by oxidation of isocolumbin with osmium tetroxide.

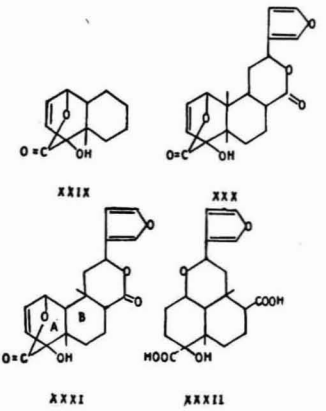
Columbin contains a β-substituted furan system as confirmed by proton magnetic resonance[44,45] and by Alder-Rickert decomposition[46]. The furan ring is allylic to the lactone ring since columbin during hydrogenation brings about the hydrogenolysis of the lactone ring and also saturates the furan moiety yielding thereby octahydrocolumbinic acid. Furthermore, decarboxycolumbin also undergoes hydrogenolysis giving decarboxyoctahydrocolumbinic acid. So the lactone ring which undergoes hydrogenolysis is, therefore, not the one represented on the partial structure (XXIII). These results suggest that the remaining lactone ring is attached as indicated in the partial structure (XXVI).



XXIII XXIV XXV XXVI

The molecular formula of columbin requires ten double bond equivalents. Eight of these are accounted for by the above experimental evidences. The remaining two are presumed for the bicyclic skeleton. The formation of 1,2,5-trimethylnaphthalene from decarboxyoctahydrocolumbinic acid on LAH reduction followed by Se-dehydrogenation is of interest in this connection. Octahydrocolumbinic acid, when submitted to the same treatment, gave the same trimethylnaphthalene and not a tetramethylnaphthalene as expected. This clearly indicated that the formation of the trimethylnaphthalene from decarboxyoctahydrocolumbinic acid must have involved migration of a methyl group from a quaternary position. This was subsequently confirmed by dehydrogenation of decarboxyoctahydrocolumbinic acid after removal of the carbonyl group by Wolff-Kishner reduction; the product was 1-methyl-2-naphthoic acid (XXVII). On the other hand, reduction of the carbonyl to hydroxyl, giving decarboxydecahydrocolumbinic acid and dehydrogenation gave 1,5-dimethyl-2-naphthoic acid (XXVIII) in which the methyl migration has taken place because of the presence of an oxygen function which provides a carbonium ion by elimination. This gives for columbin the partial formula (XXIX).

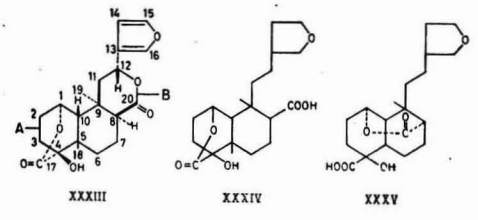
The formation of (XXVII) determines the position of the side chain bearing the furan ring at C-1 and the carboxyl group of the second lactone ring at C-2. This together with the fact that columbin contains two C-methyl groups allows two possible structures for columbin, namely (XXX) and (XXXI). (XXXI) is preferred as it explains the degradation of columbin to *o*-cresol and 2,4-dimethylbenzoic acid better than (XXX) which might be expected



XXIX XXX XXXI XXXII

to give 2,3-dimethylphenol and 3,4-dimethylbenzoic acid.

Stereochemistry of columbin—The stereochemistry of columbin was first investigated by Cava *et al.*[47] who suggested *trans*-fusion of the rings A and B from biogenetic considerations. Later on Overton *et al.*[48] have discussed the stereochemistry of columbin on the basis of the evidence secured from alkaline hydrolysis followed by oxidation experiments on the derivatives of columbin. Thus the formation of dicarboxylic acid (XXXII) from isodihydrocolumbin (XXXIII; 8β-H) coupled with the fact that octahydroisocolumbinic acid (XXXIV) yields the isolactone (XXXV) under mild alkaline conditions in high yield favours the unusual *cis*-fusion of the rings A and B as well as the *trans*-relation between the methyl groups at C-19 and C-18. Of the two lactones involved in rings A and B, the latter can be easily hydrolysed, a fact which can explain the β-configuration of the hydrogen atom at 12-position in the molecule and the transformation of columbin to isocolumbin involves the epimerization at C-8 as well as the change from a *cis*- to a *trans*-fused lactone. All these facts taken in conjunction support the stereochemistry of columbin presented by (XXXIII; double bond at positions 2 and 3).



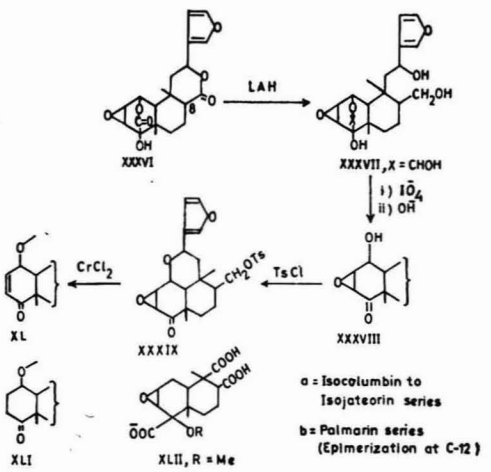
XXXIII XXXIV XXXV

Palmarin, Isojateorin, Chasmanthin and Jateorin

Chasmanthin, $C_{20}H_{22}O_7$, and jateorin, $C_{20}H_{22}O_7$, the congeners of columbin, can be easily isomerized in the presence of alkali to palmarin and isojateorin[49-52]. The alkali induced isomerization involves only the epimerization at C-8 as evidenced from the conversion of columbin to isocolumbin under the same identical condition. Palmarin and isojateorin contain all the functional groupings occurring in columbin.

Palmarin (XXXVIb) which is a companion bitter principle of columbin upon LAH reduction yields a hemiacetal (XXXVIIb) which on periodate oxidation and alkaline hydrolysis is converted to a trihydroxy ketone (XXXVIIb) (Chart 3). Treatment of the latter with *p*-toluene-sulphonyl chloride furnishes anhydromonotoluene-*p*-sulphonate (XXXIXb) which on reduction with chromous chloride gives an α,β-unsaturated ketone (XLb), thus indicating the presence of the remaining oxygen atom at 1,2-epoxide ring in β,γ-relationship to the 'A-lactone' of palmarin. Isocolumbin on similar processing is converted to the unsaturated ketone (XIa) which corresponds to that obtained from isojateorin. Furthermore, dihydroisocolumbin can be degraded under identical condition to a saturated ketone (XLIa) obtained by hydrogenation of (XLa) thus proving that isojateorin is the 1,2-epoxide of isocolumbin.

Oxidation of methylpalmarin and methylisojateorin leads to merochasmanthinic acid (XLII) retaining lactone-A but lactone-B as well as the furan ring having been replaced by two carboxyl groups. The identity of this product shows that the stereochemistry at C-12 is the only difference between palmarin and isojateorin.



XXXVI XXXVII, X = CHOH XXXVIII XXXIX XL XLI XLII, R = Me

Chart 3 — Experimental evidence to arrive at the structures of palmarin and isojateorin

A consideration of the above reactions coupled with other degradative experiments leads to a structure (XXXVI) for palmarin, chasmanthin, jateorin and isojateorin which are epoxycolumbins[50] differing in configuration at C-8 and C-12.

Tinophyllone

Tinophyllone[51], $C_{21}H_{24}O_8$, m.p. 175°, $[\alpha]_D$ −98°, has been isolated from the root and bark of a plant (*Tinomiscium philippinense* Diels) of the same family as that of *Jatrorrhiza palmata* Miers from which columbin has been isolated. It contains

one α,β-unsaturated carbomethoxy group, one α,β-unsaturated ketonic system ($\lambda_{max.}$ 238 mμ, ε 7500) and two tertiary methyl groups. On reduction with zinc-acetic acid tinophyllone furnishes dihydrotinophyllone, $C_{21}H_{26}O_6$ ($\lambda_{max.}$ 283 mμ, ε 35), which indicates the presence of

$$-C-C=C-COOCH_3$$
$$\quad\quad\quad

Furthermore, oxidation of the hydroxyketolactone (L) with chromic acid gives a saturated dilactone, $C_{16}H_{20}O_5$ (LI). IR spectrum shows the presence of γ- and δ-lactone rings in the molecule and their formation can be best explained by the oxidative destruction of the furan ring. On the basis of the above evidence the partial structure of sciadin can be elaborated to the expression (XLIVa).

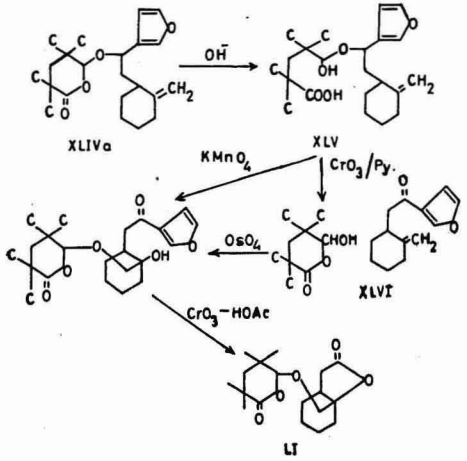


Chart 6 — Partial formulation of sciadin (XLIVa) on the basis of oxidative degradation studies on sciadinic acid (XLV)

The NMR spectrum of sciadin discloses the presence of (i) a β-monosubstituted furan ring (τ=2·60 and 3·53)[44], (ii) a vinylidene group (around 5·17-4·99 τ), (iii) one tertiary methyl group (τ=8·77) similar to that present in resin acids and other diterpenoids[54-57] and of a proton attached to C-17 (τ=4·50) carrying two oxygen atoms like the C-16 proton of clerodin[58]. The evidence thus accumulated favours the structure (XLIVb) for sciadin which receives confirmation by further experimentations discussed in the sequel.

Sciadin with hydrogen chloride gas yields anhydrosciadin, $C_{20}H_{22}O_3$ (LII). Its spectroscopic analysis shows the presence of a benzofuran nucleus ($\lambda_{max.}$ 250, 257, 279 and 288 mμ) and of a γ-lactone (1759 cm.$^{-1}$) with one tertiary C-methyl at C-4 (sharp singlet: τ=8·77) and the other secondary C-methyl (doublet: τ=8·63 and 8·52) corresponding to the grouping (LIII) in the molecule. The anhydrosciadin (LII) on treatment with ozone is converted into a norphenol, $C_{19}H_{22}O_4$ (LIV). Its IR and UV spectra[59] also define the presence of salicylaldehyde moiety in the molecule, thus giving further evidence for the presence of a benzofuran nucleus[60] in the molecule of anhydrosciadin which affords salicylaldehyde derivative[61] on ozonolysis. Since dehydration of the lactonic hydroxyl of anhydrosciadin takes place easily during the alkaline hydrolysis yielding thereby bisanhydrosciadinic acid, $C_{20}H_{22}O_3$ (LV; R=H), the lactonic hydroxyl group must be tertiary and attached to a carbon atom adjacent to the aromatic nucleus (Chart 7).

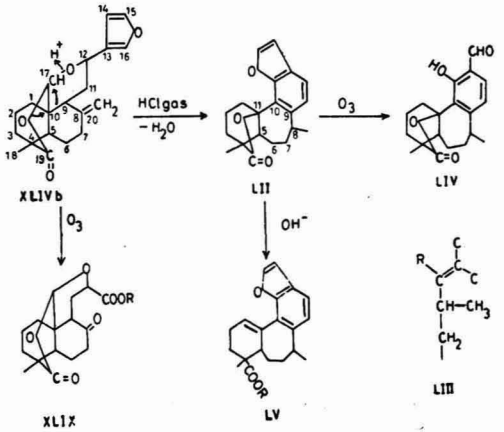
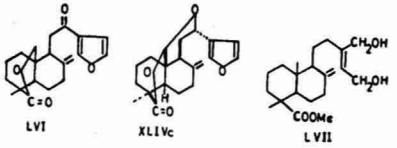


Chart 7 — Experimental evidence confirming the structure of sciadin as shown in (XLIVb)

The knowledge regarding the conformation of sciadin is obtained from the negative Cotton effect of the ORD curve of the methyl ester of the C-16 ketoacid (XLIX; R=Me) obtained by the ozonolysis of sciadin, which suggests that the position of the lactol ether grouping in the molecule of ketoacid as well as in aciadin is on the β-side and not on the α-side[56,62] and the conformation of sciadin may be represented in (XLIVc).



Sciadinone

Sciadinone[63], isolated from the leaves of *S. verticillata* Sieb et Zucc, is closely related to sciadin but differs only in containing a ketonic function instead of an oxide ring in the molecule. Its structure (LVI) has been elucidated and its relation to sciadin is made apparent. It would be interesting to mention that methyl sciadopate[64,65], $C_{21}H_{34}O_4$ (LVII), which occurs along with sciadin in *S. verticillata*, represents the precursor of these two oxygenated derivatives of enantio-polyalthic acid[57].

Biogenetic Relationship of the Diterpenoid Furanolactones

The key substance in the biogenesis (Chart 8) proposed for this group of substances, namely marrubiin, columbin, sciadin and the related compounds, is geranylgeraniol from which biogenetic transformation[66] leads to these furanolactones.

Tetranortriterpenoid Furanolactones

Considerable progress has been made since about 1957 in our knowledge of these natural compounds which are degraded tetracyclic triterpenoids with butyrospermol as a progenitor. Apart from minor differences arising from the presence of substituents and the degree of unsaturation, this interesting

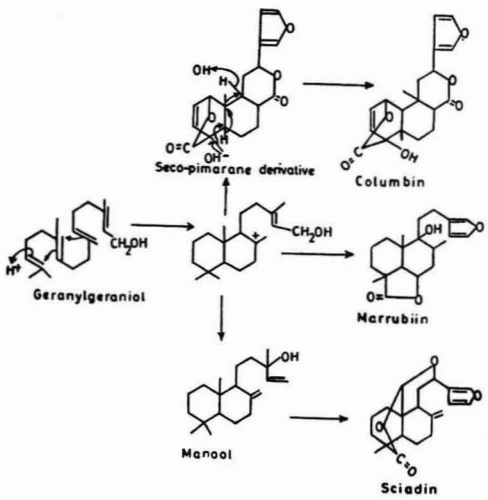
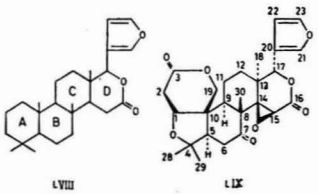


Chart 8 — Biogenesis of diterpenoid furanolactones

group of compounds is associated with the oxidative cleavage of ring D (gedunin anthothecol and khivorin), cleavage of rings D and A (limonin, nomilin and obacunone), and cleavage of rings D and B



(andirobin, swietenolide, methyl angolensate and swietenine). They may be derived from a hypothetical C_{26} carbon skeleton (LVIII) having a β-furyl-δ-lactone grouping in it. The most striking features of them are the reductive elimination, alkaline cleavage followed by rearrangement and stereochemical requirements of reactions and of the relationship of physical properties. In this connection the structures of nimbin and salanin containing a cleaved ring C need mention. Nimbin like limonin, swietenine and swietenolide give alkyl naphthalenes on dehydrogenation. The production of C_{26} acid, a characteristic feature of this group, is also obtained by the hydrolysis of nimbin and salanin. The brilliant achievement to their chemistry is concerned with the recent structural work on limonin, which has become a significant milestone in the progress of this growing class of natural products. Table 2 lists the more important

TABLE 2 — NATURALLY OCCURRING FURANOID TETRANORTRITERPENES AND THEIR PHYSICAL CONSTANTS

Compound	Mol. formula	m.p. °C.	$[\alpha]_D$ deg.	$\lambda_{max.}$ mμ	Ref.
Andirobin	$C_{26}H_{32}O_6$ ($-OCH_3$)	195-7	—	209 (9100) (andirobindiol diacetate)	108
Anthothecol	$C_{26}H_{32}O_6$ ($-COCH_3$)	225	−63	219 (13000); 281 (11000)	106
Carapin	$C_{26}H_{32}O_6$ ($-OCH_3$)	175-8	+64	213 (16000)	141
Cedrelone	$C_{26}H_{30}O_5$	209-14	−64·5	217 (11800); 279 (9100) shifting in alkali to 327 (5550)	126, 127
7-Deacetoxy-7-ketokhivorin	$C_{30}H_{32}O_7$ ($-COCH_3$)$_2$	228-30	−106	213 (7762); 282 (66)	106
Dihydrogedunin	$C_{28}H_{32}O_6$ ($-COCH_3$)	237-8	+3·7	207 (6600); 289 (25)	109
Gedunin	$C_{28}H_{32}O_6$ ($-COCH_3$)	218	−44	215 (15850); 335 (63)	99, 100
Khivorin	$C_{30}H_{32}O_7$ ($-COCH_3$)$_2$	256-63	−42	209 (6285)	105
Limonin	$C_{26}H_{30}O_8$	298	−120	207 (7000); 285 (85)	74
Mahogenin	$C_{28}H_{32}O_7$ ($-OCH_3$)	225-8	−72	207 (9120); 285 (29)	133
Maxicanolide	$C_{28}H_{32}O_6$ ($-OCH_3$)	222-7	−90	209 (10850) shifting in alkali to 287 (31700) and in acid to 264 (20800)	140
Methyl angolensate	$C_{28}H_{32}O_6$ ($-OCH_3$)	199-201	−48	206 (3100); 287 (26)	109, 114
Nimbin	$C_{28}H_{32}O_6$ {($-COCH_3$), ($-OCH_3$)$_2$}	205	+170	214-215 (17780); 335 (54)	134, 135
Nomilin	$C_{28}H_{32}O_8$ ($-COCH_3$)	298-9	−95·7	218 (18620)	88
Obacunone	$C_{26}H_{30}O_7$	229-30	−50·2	213 (14000)	91, 92
7-Oxodeacetoxydihydro-α-gedunol	$C_{26}H_{34}O_6$	300-1	−126	207 (6600); 291 (48)	109
Swietenine	$C_{28}H_{32}O_7$ {($-OCH_3$), ($-COC_4H_7$)}	260	−168	215 (18620); 279-282 (62) (shoulder)	115
Swietenolide	$C_{26}H_{32}O_7$ ($-OCH_3$)	220 (decomp.)	−126	215 (10960); 288 (52)	121
Salanin	$C_{28}H_{32}O_6$ {($-COCH_3$), ($-OCH_3$), ($-COC_4H_7$)}	167-70	+167	—	136

Values in parentheses refer to ε values.

naturally occurring furanoid tetranortriterpenes with characteristic physical constants.

Limonin

Limonin (LIX) was first isolated from citrus fruits by Bernay[67]. Since then it has been investigated in many laboratories[68-71]. The constitution of limonin has recently been elucidated by a number of workers[72-74], and independently from its X-ray crystallography by Robertson and coworkers[75,76].

Limonin, $C_{26}H_{30}O_8$, contains two δ-lactones (1755-1760 cm.$^{-1}$) and a six-membered ketonic function. The absence of hydroxyl and alkoxy groups in the molecule suggests the inert character of the three remaining oxygen atoms which are in keeping with the presence of oxide rings. The spectroscopic evidences[70,72], including NMR[44] spectrum, however, point out that one of the oxide rings is a mono-β-substituted furan system which has been confirmed also by photooxidation[72], and by the application of the Alder-Rickert reaction[70]. On hydrogenation limonin gives tetrahydrolimonin, $C_{26}H_{34}O_8$ (furan ring has been saturated), as well as hexahydrolimoninic acid, $C_{26}H_{36}O_8$, by hydrogenolysis of one of the lactone rings. This behaviour is reminiscent of columbin[42] and the high acidity of hexahydro compound[69] (pK 2·7) indicates that the carbon atom α- to the lactonic carbonyl probably carries an electron attracting oxygen function.

Reduction of limonin with hydroiodic acid[77] or chromous chloride yields deoxylimonin, $C_{26}H_{30}O_7$, containing an α,β-unsaturated lactone ring (1714 cm.$^{-1}$) as also a saturated δ-lactone (1747 cm.$^{-1}$). Other derivatives of limonin also behave similarly. The reductive elimination of 1,2-epoxide conjugated with the lactonic carbonyl[78] can explain the formation of α,β-unsaturated lactone in deoxy compounds of limonin which are in agreement with the spectral feature of deoxylimonin and tetrahydrodeoxylimonin (sharp peak at τ=11·42).

Tetrahydrolimonin (LX) with hydrochloric acid-acetic acid mixture under controlled conditions gives an enolized α-ketolactone (LXI; R=H) characterized by spectroscopic examinations as well as by ozonolysis followed by mild hydrolysis which affords oxalic acid. It is found that hexahydrolimoninic acid (LXII) by the action of hydrochloric acid-acetic acid rearranges to a γ-lactone (LXIIa) in agreement with the observations recorded above. The latter upon oxidation with pyridine-chromium trioxide is converted to a non-enolizable α-keto-γ-lactone (LXIII). These results suggest that one of the lactone ring occurs as that shown in the partial structure (LXIV) (Chart 9).

Limonin and its derivatives are rapidly autoxidized in dry *tert*-butanol containing potassium *tert*-butoxide[74], with uptake of one mole of O_2 yielding products having diosphenol chromophore (LXV) ($\lambda_{max.}$ 207 and 278 mμ shifted reversibly in alkali to $\lambda_{max.}$ 336 mμ; 1689 and 1661 cm.$^{-1}$), thus indicating the environment of ketonic function as indicated below:

$$-\underset{|}{CH}-CH_2-\overset{|}{C}=O$$

The relationship between the ketonic group of limonin and the lactone ring D has been explored

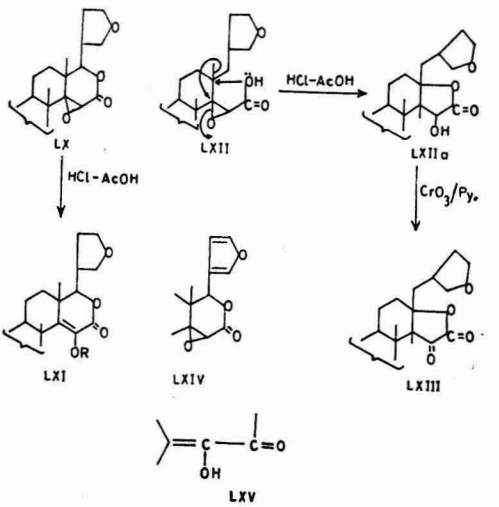


Chart 9 — Various reaction products of limonin suggestive of the type of one of the lactone rings as shown in the partial structure (LXIV) for limonin

in the following way. Deoxylimonin (LXVI) on alkaline hydrolysis gives deoxylimonic acid[74] (LXVII; R=H) which contains two lactone residues but no ketonic function. The ester (LXVII; R=Me) shows four methyl peaks of which one is at 8·37τ arising from the methyl group attached to the tetrasubstituted double bond at C-8. On the other hand, deoxylimonin oxime does not undergo similar alkaline degradation which proves that the ketonic group of deoxylimonin is involved in the formation of deoxylimonic acid according to the mechanism shown in Chart 10.

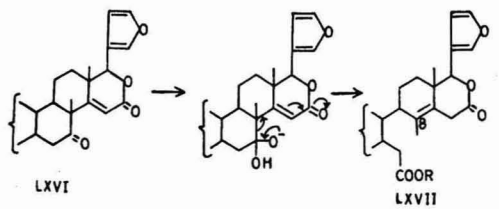


Chart 10 — Relationship between the ketone group of limonin and the lactone ring D

Similarly, deoxytetrahydrolimonin (LXVIII) gives deoxytetrahydrolimonic acid, $C_{26}H_{36}O_8$ (LXIX), on alkaline degradation. The acid reacts with one molecule of chlorine to give an unstable adduct (LXX) which on heating *in vacuo* gives two crystalline diene acids. The major product is a cisoid diene (LXXI; $\lambda_{max.}$ 255 mμ, ε 7800) which gives formaldehyde on ozonolysis and has three vinyl type protons (τ=4·60, 4·38 and 3·97). The minor product is a transoid diene (LXXII) as shown by its UV spectrum ($\lambda_{max.}$ 230 and 284 mμ, ε 6300 and 16400 respectively). These experiments (Chart 11) add further evidence in support of the above facts as well as establish the location of a methyl substituent at C-8 and hydrogen atom at C-9 (LXXIII).

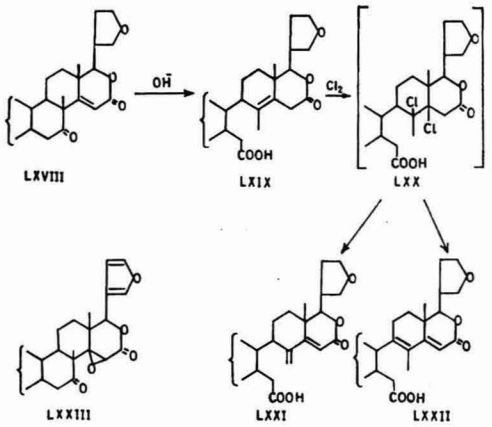
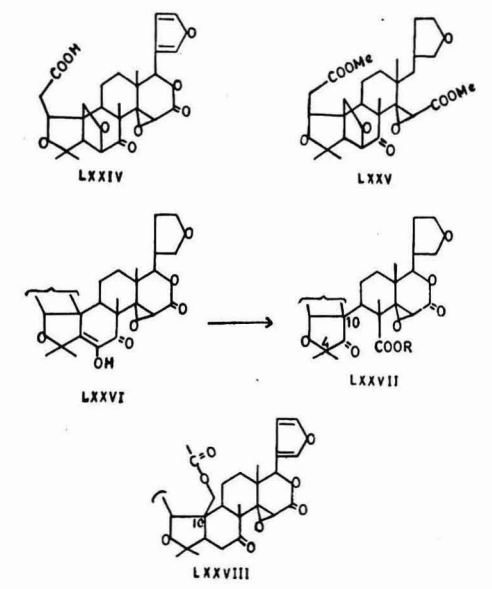


Chart 11 — Experiments supporting the view that the ketonic group of deoxylimonin is involved in the formation of deoxylimonic acid (Chart 10) as well as establishing the location of a methyl substituent at C-8 and hydrogen atom at C-9 as shown in (LXXIII)

Limonin with hypoiodite oxidation[69] gives limonilic acid, $C_{26}H_{30}O_9$, which with chromous chloride gives deoxylimonilic acid, $C_{26}H_{30}O_8$, in which the lactone ring D has been converted to an α,β-unsaturated lactone. Furthermore, limonilic acid on hydrogenation followed by esterification gives a diester (LXXV) identical with that obtained from hexahydrolimoninic acid on hypoiodite oxidation. Thus the formation of limonilic acid involves the lactone ring A. Limonilic acid shows the absence of hydroxyl and aldehyde functions, but it contains the ketonic group which does not undergo diosphenol reaction. Eventually, the environment of the ketonic function has been modified. Limonilic acid furnishes limonin on reduction with aluminium amalgam. All these reactions can explain the presence of an axially oriented ether[79,80] substituent in α-position with respect to the keto group of limonilic acid represented by (LXXIV), the formation of ether linkage being facilitated by a favourable steric situation.



On ozonolysis the diosphenol (LXXVI) obtained from tetrahydrolimonin affords a crystalline nor-acid, $C_{25}H_{32}O_{10}$ (LXXVII; R=H), which with alkali undergoes a reversed aldol-type elimination of formaldehyde. This shows the grouping (—CH_2—O—CO—) either at C-10 or C-4. The methyl ester of the nor-acid (LXXVII; R=Me) and its trisodium salt obtained by opening the two lactone rings show a carbonyl band near 1760 $cm.^{-1}$. This enhanced frequency for the ketonic group indicates the presence of a cyclopentanone with an α-ethereal substituent thus favouring the position C-10 for the system (—CH_2—O—CO—) in the partial formula of limonin (LXXVIII).

In the next series of experiments reduction of the ketonic group in limonin by the Meerwein-Ponndorf method furnishes the axial alcohol, limonol[81,82] (LXXIX; R=OH, R′=H) and with sodium borohydride[72] or sodium amalgam[72,83], affords the epimeric equatorial alcohol, epilimonol (LXXIX; R=H, R′=OH). Limonol on alkaline degradation gives merolimonol[72], $C_{21}H_{28}O_6$ (LXXX), with the elimination of furan-3-aldehyde which is associated with the rings C and D, the furan moiety and the 7α-hydroxyl group[84]. The NMR spectrum of limonin indicates the presence of 4C—CH_3 groups but that of merolimonol shows 3C—CH_3 groups in addition to a methyl group attached to a double bond (τ=6·6). Merolimonol characterized as a monoacetate can be dehydrated to an unsaturated conjugated-lactone, anhydromerolimonol (LXXXI). On ozonolysis it furnishes a keto-acid (LXXXII) responding to iodoform test (methyl ketone peak at τ=6·78). On the other hand, on oxidation with manganese dioxide, merolimonol gives the decarbonylated derivative (LXXXIII) in which the presence of δ-lactone and the α,β-unsaturated-γ-lactone groupings can be clearly discerned. The formation of an unenolized α-ketolactone (LXXXIV) by the ozonization of (LXXXIII) favours also the placing of the methyl group at the C_8-position. As a whole the reactions (Chart 12) of merolimonol establish the relationship of C-12, C-13, C-14, C-15, C-16 and C-18 with that of C-7 and C-8.

Treatment of merolimonol with barium hydroxide affords an acid (LXXXV; R=H) in which the lactone ring A has opened irreversibly, unlike limonin and its derivatives. The methyl ester of this acid on oxidation with manganese dioxide furnishes the γ-lactone (LXXXVI) which can yield the aldehyde (LXXXVII) on further oxidation with chromium trioxide. Limonin on more vigorous alkaline degradation gives limoclastic acid, $C_{20}H_{28}O_6$, with a total loss of six carbon atoms. From the knowledge of its functional groups coupled with its same peculiar reactivity as merolimonol it has been assigned the structure (LXXXVIII; R=H). It is interesting enough that the aldehyde (LXXXVII) can be converted to the lactone ester

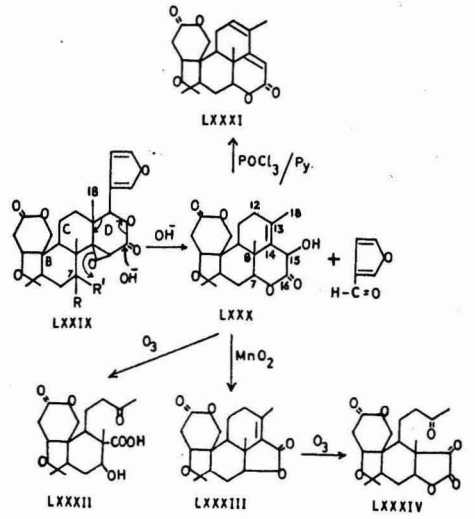


Chart 12 — Formulation of merolimonol and its reactions to establish the relation of C-12 to C-16 and C-18 with that of C-7 and C-8

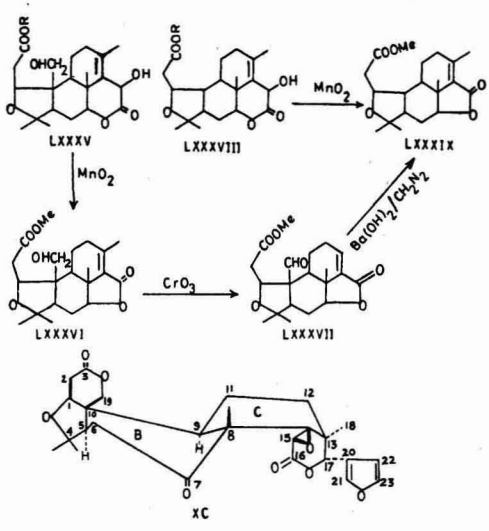


Chart 13 — Action of barium hydroxide on merolimonol and vigorous alkaline degradation of limonin to give limoclastic acid

(LXXXIX) which is derived from limoclastic acid by oxidation with manganese dioxide.

All these experimental facts eminently signify that under the influence of alkali, a deep-seated hydrolytic change occurs in limonin involving the splitting of a β-substituted furan nucleus followed by the opening of the δ-lactone ring A which remains attached to C_{19} or other equivalent methyl carbon. Furthermore, the δ-lactone ring A is provided with at least one hydrogen to allow reversible elimination of β-placed ethereal oxygen for the limonin → limoclastic acid change.

These results together with the isolation of 1,2,5-trimethylnaphthalene on degradation[88,85] and the formation of acetone from the alkali fusion of limonin[69] have established its structure as (LIX) which has been elegantly confirmed in every detail by the excellent X-ray work on epilimonol iodoacetate.

The absolute stereochemistry[86] has also been deduced from the crystal structure of epilimonol iodoacetate[75,76], $C_{28}H_{33}O_9I$, and hence to the derivation of limonin as shown in (XC) in which ring B has a chair and ring C a boat conformation.

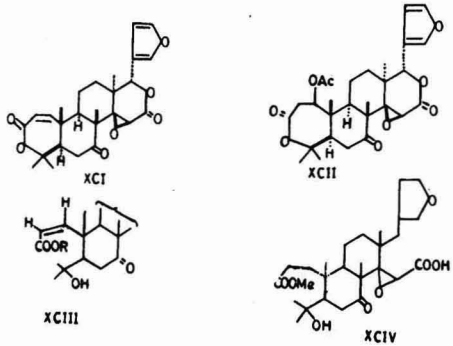
Nomilin and Obacunone

Nomilin[87], $C_{28}H_{34}O_9$, and obacunone[88,89], $C_{26}H_{30}O_7$, are two bitter principles which occur in citrus fruits. Obacunone was also isolated from *Phellodendron amuranse* Rupr[90,91].

Both nomilin and obacunone are keto-dilactones. Of the two lactone rings one can be opened reversibly and the other irreversibly. On alkaline hydrolysis nomilin and obacunone give obacunoic acid[74], $C_{26}H_{32}O_8$, $\lambda_{max.}$ 210 mμ, ε 14000, the former yielding acetic acid at the same time. Obacunone and obacunoic acid show high intensity of absorption at about 209 mμ while nomilin absorbs much less intensely. These results suggest that one of the lactone rings in nomilin carries a β-acetoxy group, obacunone being the corresponding α,β-unsaturated lactone. Further, nomilin is convertible to obacunone by boiling with γ-picoline[92]. The absence of hydroxyl and alkoxy groups in both obacunone and nomilin suggests that the remaining two oxygen atoms not accounted for by the above functional groups are present in oxide rings. Spectroscopic analysis[92] and also the Alder-Rickert decomposition[70] indicate that one of these oxide rings is a mono-β-substituted furan.

Obacunoic acid (XCIII; R=H), which contains an alcoholic hydroxy group and isomerizes to isoobacunoic acid, is formed by the irreversible and facile fission of the seven-membered lactone ring A. It has also been confirmed from the reactions of etio-obacunoic acid[93,94], $C_{23}H_{28}O_8$, obtained by the degradation of furan ring occurring in obacunone. Furthermore, obacunoic acid gives positive iodoform test which also indicates the presence of hydroxyisopropyl group separated by two carbon atoms from the ketonic function[92].

Methyl obacunoate (XCIII; R=Me) on catalytic reduction undergoes hydrogenolysis of the lactone



ring D due to the activation of the furan system yielding thereby (XCIV; $C_{27}H_{42}O_8$; pK 2·9 in water), which can also be obtained from obacunone by hydrogenation followed by methanolysis of the saturated 7-membered ring. Obacunoic acid is reduced with chromous chloride to a deoxyobacunoic acid, $C_{26}H_{32}O_7$, which contains α,β-unsaturated-δ-lactone ring (1715 and 1695 cm.$^{-1}$). The above facts together with the conversion of the dilactone, $C_{24}H_{28}O_7$, obtained by potassium permanganate oxidation of obacunone[95], to a deoxy compound, indicate the presence of α,β-epoxy-δ-lactone moiety which is allylic to the β-substituted furan ring as in limonin. The formation of a diosphenol chromophore by the alkaline autoxidation of obacunoic acid also defines the environment of the ketonic group.

The NMR spectrum[74] of methyl obacunoate indicates 4 tertiary C—CH_3 groups, one C—CH_3 group (τ=8·618) close to the furan ring as in limonin and a *cis*-β-substituted acrylic ester (A-B quartet at τ=4·24 and 4·49 and at 3·798 and 4·08) as well as a β-substituted furan ring[44]. The peak at 4·63 τ can be assigned to >CH—O— at 17-position like that of limonin.

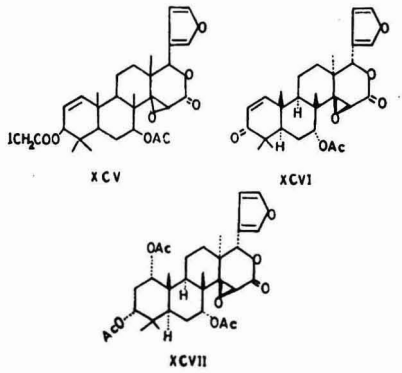
The correlation of obacunone and limonin has been established from their degradative experiments. Thus methyl deoxytetrahydroanhydromerolimonolate[97,98] derived from limonin corresponds with tetrahydroderivative of methylanhydroepimeroobacunolate[96] in IR, m.m.p. and $[\alpha]_D$ showing thereby the same configuration of carbon atoms at C-5, C-8, C-9 and C-10 like limonin. These results together with biogenetic considerations lead to the steric structures (XCI) and (XCII) for obacunone and nomilin respectively.

Gedunin

Gedunin, $C_{28}H_{34}O_7$, and its derivatives are distributed in the various species belonging to Meliaceae family (Table 1). Gedunin[99,100] is a ketolactone having a double bond in the α,β-position with respect to the ketonic function ($\lambda_{max.}$ 215 and 335 mμ; 1668 cm.$^{-1}$). On hydrogenation it gives dihydrogedunin showing a new band at 1709 cm.$^{-1}$ (saturated ketone). Gedunin on mild alkaline hydrolysis gives an alcohol, deacetylgedunin, $C_{26}H_{32}O_6$, which resists acetylation under ordinary condition, but on treatment with acetic anhydride and *p*-toluenesulphonic acid furnishes gedunin. On alkaline degradation gedunin produces furan-3-aldehyde like that of limonol[72] which discloses the presence of same structural moiety responsible for this reaction in gedunin. Further the formation of deoxygedunin, $C_{28}H_{34}O_6$, and deoxydihydrogedunin, $C_{28}H_{36}O_6$, in the same way as those obtained from limonin[74] indicates the presence of an α,β-spoxy-δ-lactone ring in the molecule. So all these experimental facts are able to support the characterization of all the oxygen functions in the molecule of gedunin.

$NaBH_4$ reduction of gedunin furnishes a saturated alcohol, $C_{28}H_{38}O_7$, which on oxidation with chromium trioxide[101] regenerates dihydrogedunin. This unusual reduction of an unsaturated ketone to the corresponding saturated alcohol has been observed in cholest-1-en-3-one[102]; as such gedunin is a 3-oxo-Δ′-triterpene derivative. On ozonolysis, dihydrogedunin gives an acid $C_{25}H_{34}O_8$; the methyl ester of this acid on reduction with sodium borohydride furnishes an amorphous alcohol which produces acetone on treatment with phosphorus pentachloride followed by ozonolysis. This reaction sequence is characteristic of 4,4-dimethyl-3-oxo-di- and triterpenes with rings A and B *trans*-fused[100]. It seems interesting to point out that dihydrogedunin on reduction followed by selenium dehydrogenation yields 1,2,7,8-tetramethylphenanthrene unlike limonin and other tetracyclic triterpenes[103].

A consideration of these experiments along with the X-ray analysis of dihydrogedunin-3β-yl-iodoacetate[104], $C_{30}H_{39}O_8I$ (XCV), has established the constitution and stereochemistry of gedunin as shown in (XCVI).



Khivorin

Another related compound, khivorin, and its derivative, 7-deacetoxy-7-ketokhivorin, occur in the timber of *Khaya* species[105,106] growing in Africa. Khivorin, $C_{32}H_{42}O_{10}$, has been shown to have an α,β-epoxy-δ-lactone moiety by its smooth reduction with chromous chloride to deoxykhivorin. On alkaline hydrolysis deoxykhivorin yields acetic acid and trisdeacetyl compound, $C_{26}H_{36}O_6$, which on oxidation affords an 1,3-enolic triketone[107] ($\lambda_{max.}$ 253 mμ, ε 16100 and in ethanol-3*N* KOH $\lambda_{max.}$ 285 mμ, ε 24100). Khivorin itself on alkaline hydrolysis is converted to khivol, $C_{21}H_{32}O_5$, liberating furan-3-aldehyde[72] and acetic acid. Khivol contains hydroxyl groups (3300 cm.$^{-1}$). These reactions can account for all its carbon atoms on the basis of three acetoxy groupings present in the molecule of khivorin[105].

The presence of a C_{26} carbon skeleton in association with a β-substituted furan ring along with other reactions as those observed in this new class of natural products suggest the structure (XCVII) for khivorin[105].

Comparison of the molecular rotations[105] of khivorin, gedunin and their derivatives with those of limonin and its reaction products suggests that absolute configurations of khivorin and gedunin are the same as that of limonin. Since the ring A remains cleaved in limonin, the absolute configuration of 1 and 3 hydroxyl groups cannot be assessed

from these data; but from biogenetic analogy with 7-ketodihydro-α-gedunol the hydroxyl group at C-3 appears to have β-configuration.

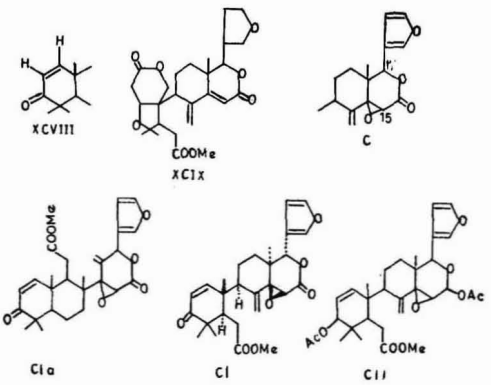
Andirobin

Andirobin[108], $C_{27}H_{32}O_7$, isolated from *Carapa guianensis* Aubl, contains one α,β-unsaturated ketonic function (1680 cm.$^{-1}$) and a δ-lacton ring (1760 cm.$^{-1}$) but no hydroxyl group. On reduction with $NaBH_4$, andirobin yields a tetrahydrodiol characterized as a diacetate ($\lambda_{max.}$ 209 mμ, ε 9100). This peak is in good agreement with the presence of a β-substituted furan ring in the molecule which is supported by the NMR spectrum of andirobin, which also discloses the presence of $4C-CH_3$ groups and one exocyclic methylene group (two singlets at τ=4·55 and 4·67). On alkaline hydrolysis it gives the corresponding carboxylic acid, $C_{26}H_{30}O_7$, which by reaction with diazomethane regenerates andirobin; so it is a methyl ester of a C_{26} acid. Subtraction of the UV spectrum of andirobindiol diacetate from that of andirobin shows a maximum at 235 mμ (ε 9500) corresponding with the α,β-unsaturated-3-ketochromophore of triterpenoids and steroids[108]. This fact together with the structural features (τ=2·80 and 3·88; J=11 *cps*) suggests the partial formula of the andirobin as (XCVIII) with a *cis*-disubstituted double bond and a tertiary γ-carbon atom in the molecule.

Andirobin can be smoothly converted to deoxyandirobin, $C_{27}H_{30}O_6$. Moreover, the comparison of the NMR data of andirobin (τ=4·47 and 5·92) with that of dihydrogedunin[109] (τ=4·40 and 6·48) indicates the similarity in the location of protons at C-17 and C-15 and as such the presence of an α,β-epoxy-δ-lactone structure has been established.

Deoxyandirobin does not isomerize to an α-pyrone in the presence of acid. The subtractive UV curve of andirobin from that of deoxyandirobin has $\lambda_{max.}$ 257 mμ (ε 8300) which is very similar to that of cisoid diene (XCIX) obtained from limonin[74]. This close spectral similarity defines the presence of same chromophoric system in deoxyandirobin. The above knowledge may be summed up in the fragment (C) of andirobin.

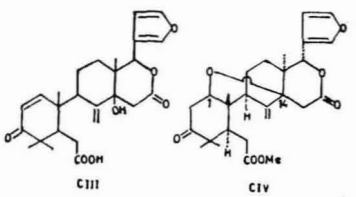
This evidence together with the presence of $-CH_2-COOCH_3$ and $>C=CH_2$ groupings, the latter being explained by the biochemical equivalent of oxidation of ring B[73,75,76,110-113] at the 7-8 bond



in the molecule of 7-deacetoxy-7-ketogedunin[108], favour the structure (CI) for andirobin rather than the alternative (CIa). It conforms well with the NMR data of andirobindiol diacetate (CII) as well as with those of other derivatives of andirobin[108].

Methyl Angolensate

Methyl angolensate[114], $C_{27}H_{34}O_7$, is isolable from the various species of Meliaceae[99,100,109] family. It is closely related to andirobin as defined by the spectroscopic and chemical data but differs in containing a 3-oxo group without any unsaturation in the α,β-position and in having no epoxide function involving C-14 and C-15. It undergoes hydrogenolysis giving an octahydro acid, $C_{27}H_{42}O_7$ (pK 7·2), which also suggests that the carbon atom α to the carboxyl group does not carry any oxygen atom like hexahydrolimoninic acid (pK 2·7)[69] but the NMR spectrum shows band at τ=3·47 (quartet) which is indicative of $>CH-O-$. This together with the acid-catalysed cyclization of the unsaturated methyl ester (CIII) derived from angolensic acid, to methyl angolensate, determines the position of the ether linkage in which one terminus is at C-1 and the other terminus has been settled at C-14 (steric and biogenetic considerations). All these facts have led to the formulation of methyl angolensate[114] as (CIV).



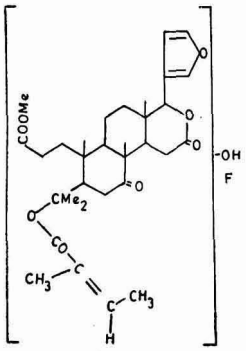
Swietenine and Swietenolide

Swietenine, a non-bitter principle, and swietenolide[115], a bitter principle, have been isolated from the seeds of *Swietenia macrophylla* King. Swietenine[116] has the formula $C_{32}H_{42}O_9$ (proton count of the NMR spectrum and X-ray method). With alkali it behaves as a lactone which can be opened reversibly. It contains one hydroxyl group. The presence of a mono-β-substituted furan system in the molecule has been shown by the IR (3160, 1506, 1030, 877 and 815 cm.$^{-1}$) and NMR spectra of swietenine, which is allylic to the δ-lactone ring as it undergoes hydrogenolysis yielding thereby a decahydro acid[116], $C_{32}H_{52}O_9$. This behaviour is reminiscent of columbin[42] and limonin[72].

The IR and UV spectra of swietenine also disclose the presence of an unconjugated six-membered ketonic function in the molecule. On mild hydrolysis with γ-picoline swietenine yields an acid which on treatment with diazomethane regenerates original compound. Swietenine has been further shown to be an ester of tiglic acid. Thus it appears to be the tiglate of a methyl ester derived from C_{26} skeleton as found in the β-substituted tetranortriterpenoid furanoid lactones. On selenium dehydrogenation swietenine yields 1,2,5-trimethylnaphthalene

and the unidentified polyalkylnaphthalene derivative.

All these findings in association with its relation to limonin can be summarized in the expression (CV) for swietenine.

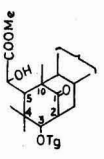


CV

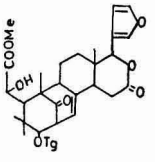
Recently, a modified steric structure (CVI) for swietenine has been advanced by the English school[117-120] based on the crystallographic measurements of the *p*-iodobenzoate of destigloylswietenine (CVII) which conform well with the chemical investigations.

Alkaline hydrolysis of swietenine (CVI) gives the α-hydroxy acid, $C_{26}H_{34}O_8$ (CVIII; 3-epi), which on methylation and oxidation affords the triketone, $C_{27}H_{32}O_8$ (CIX). Oxidation of swietenine yields 6-dehydroswietenine, $C_{32}H_{40}O_9$ (CXI), which also on hydrolysis followed by methylation and oxidation leads to (CIX). Moreover, reduction of triketone gives a diketone, $C_{27}H_{34}O_7$ (CXII), which undergoes β-dicarbonyl cleavage in the presence of mild alkali to yield a γ-lactonic ester in which the double bond is not conjugated with the carbonyl function. This suggests the presence of cyclooctanone moiety and it can be formulated in (CXIII).

The relationship between the α-hydroxy ester and the tiglate ester groupings in swietenine has been disclosed by the oxidation of α-hydroxy acid (CVIII) to the noraldehyde, $C_{25}H_{32}O_6$, (CXIV) which undergoes intramolecular Cannizzaro reaction to yield the γ-lactone (CXV) which thus defines the configuration of C-3 hydroxyl in destigloyl-3-episwietenine (CX) derived from (CVIII) by methylation. Further C-4 and C-10 do not bear any hydrogen as evident from a sharp doublet at C-5 (τ=0·18; J=6·5 *cps*) in (CXIV) and (CXV), collapsing to a sharp singlet (τ=7·25) when decoupled respectively from H-6 and H-3. The presence of unconjugated six-membered carbonyl functions at C-1 and C-3 as well as the nonenolization of the β-diketones, (CIX) and (CXIVa), can explain the formation of bicyclo(3:3:1)nonenone in the molecule of swietenine. All these facts can be expressed by the part structure (CXVI) which has been elaborated to (CXVII).



CXVI



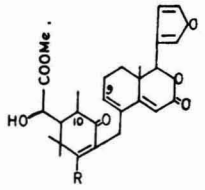
CXVII

The constitution of swietenine is the most intricate and subtle of this new class of compounds so far encountered. The presence of α-oriented 19-methyl group as well as the position of the hydroxy and ester $>C<^{Me}_{Me}$ groups reveals that it is associated with the oxidative cleavage of ring B at 7-8 positions followed by rotation of ring A about the 9-10 bond and with the addition of C_2 to C_{30}, which have been disclosed by the X-ray analysis.

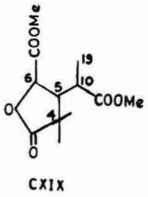
Swietenolide[121,122], $C_{27}H_{34}O_8$, contains a carbonyl function and has a δ-lactone which is allylic to the furan ring. Hydrogenation involves hydrogenolysis of the lactone ring as well as saturation of the furan and hexahydroswietenolic acid, $C_{27}H_{40}O_8$ ($\lambda_{max.}$ 208 mμ, ε 6500), is obtained which still contains one ethylenic double bond. Alkaline hydrolysis of swietenolide gives swietic acid, $C_{26}H_{32}O_8$, from which the original ester is regenerated with diazomethane. It contains two hydroxyl functions of which one is α- to the carbomethoxy group. The above results raise the interesting possibility that swietenine might be a tiglate ester of swietenolide which has been ruled out on several reasons. Firstly, tiglylation of swietenolide does not afford

swietenine. Secondly, destigloyl-3-dehydroswietenine (CXII) is not identical with 3-dehydroswietenolide. They also differ in their behaviour on dehydrogenation. Thus selenium dehydrogenation of the nonvolatile product obtained by caustic fusion of swietenolide yields trialkylnaphthalene.

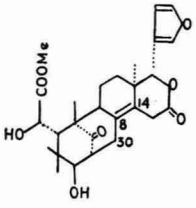
Further insight into the structure has been obtained by oxidative degradation. Swietenolide consumes one atom of 'oxygen' with 0·1*N* potassium dichromate in acetic acid yielding thereby 3-dehydroswietenolide, $C_{27}H_{32}O_8$ ($\lambda_{max.}$ 288-289 mμ, ϵ 102), whose behaviour is strongly reminiscent of enolized β-diketone[123] (in alkaline-ethanol $\lambda_{max.}$ 287 mμ, ϵ 34200 shifted on acidification to $\lambda_{max.}$ 265 mμ, ϵ 14700). On the assumption that bicyclo-(3:3:1)nonenone structure is present as in swietenine the change can be explained by the oxidation of secondary hydroxyl at C-3 and cleavage of the 9-10 bond which lead to the formation of dienelactone formulated as (CXVIII; R=OH). This on further oxidation with sodium periodate[124] followed by methylation results without any loss of carbon atom in the formation of two fragments characterized as γ-lactone dimethyl ester, $C_{12}H_{18}O_6$ (CXIX), and dienelactone methyl ester, $C_{17}H_{18}O_5$ (CXX).



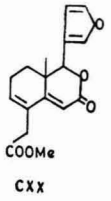
CXVIII



CXIX



CXXI



CXX

Acetylation of swietenolide (depending upon condition) affords a monoacetate, $C_{29}H_{34}O_8$ (CXVIII; R=OAc) [$\lambda_{max.}$ 236 mμ, ϵ 11600 and $\lambda_{max.}$ 278 mμ, ϵ 15000; vinyl protons at τ 3·87 (C-3), 4·02 (C-9) and 4·30 (C-15)], also provides additional support for the above formulation.

On the evidence of these reactions swietenolide is represented by (CXXI) which is a double bond isomer of destigloyl swietenine.

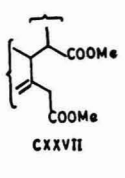
Biogenesis

Tetranortriterpenoids may be derived from butyrospermol with the formation of a furan ring by the cyclization of C-20 to C-23 followed by loss of four carbon atoms at the end of the chain (C-24—C-27) leading thereby to an intermediate (CXXII) which gives rise to cedrelone, anthothecol, nimbin and salanin. In this connection the isolation of turraeanthin[138] from Meliaceae family needs mention as it supplies the missing link in the same stereochemical compounds with a 17-3'-furyl substituent. The formation of α,β-epoxy-δ-lactone moiety may be explained by the biochemical equivalent of the Baeyer-Villiger oxidation of a C-16 ketone derived from (CXXII), to furnish the D-lactone which with the migration of a methyl group from C-14 to C-8 gives rise to a precursor (CXXIII) of these furanolactones. Such a reaction is known to occur in dihydrobutyrospermyl acetate[128] (CXXIV) which furnishes a product[73] (CXXV) of the same partial structure as gedunin and the related products. Moreover, it is notable that katonic acid[139], a pentacyclic terpenoid which occurs in Meliaceae family, can also be built up from the same branch skeleton generated from squalene. Thus a tentative biosynthetic scheme covering the skeletally related C-26 modified triterpene has been outlined in Chart 14.

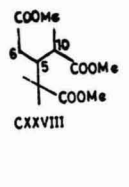
It seems worth while to discuss the plausible biosynthetic pathway to tetranortriterpenoids from mevalonic acid. The biosynthetic sequence thus involved has several implications as this might be useful for studying chemical plant taxonomy. It may so happen that in future from related botanical species having close taxonomical relationship, intermediates as shown in the scheme may be isolated as has been observed in other fields. This would help the understanding of the process of biological evolution and, therefore, to find out the missing link rather the bridge between related botanical families.

Most Recent Advances

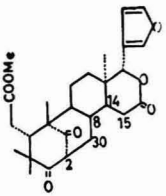
The relevant data which have come to the attention subsequent to the completion of this article have been incorporated in Table 1 and the chemistry of a few items are described below.



CXXVII



CXXVIII



(CXXIXa, double bond at C_{14}–C_{15})
(CXXIXb, double bond at C_8–C_{14})

Maxicanolide[140] and carapin[141-143] are two closely related β-substituted furanoid lactone methyl esters having the molecular formula $C_{27}H_{32}O_7$. The UV spectrum of carapin at 213 mμ (ϵ 16000) precisely matches that of deoxykhivorin which, therefore, determines the position of the isolated double bond between C-14 and C-15. On chromatography over alumina carapin is converted into isomeric maxicanolide which on treatment with methanolic sulphuric acid furnishes a product characterized as (CXXVII) not given by the former under identical condition. Both of them undergo irreversible rearrangement in the presence of alkali to yield a

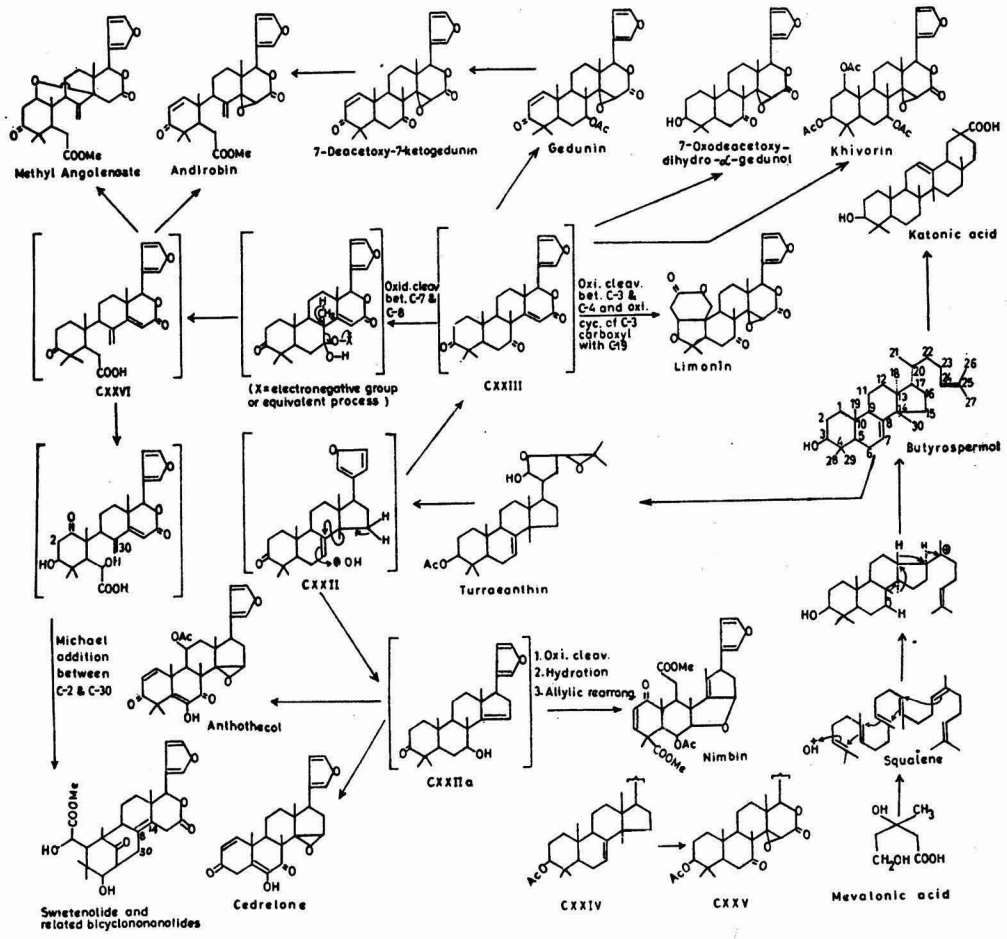


Chart 14 — A tentative biosynthetic scheme covering the skeletally related C_{26} modified triterpenes

diene-lactone which on periodic acid oxidation followed by methylation gives a diene-lactone methyl ester (CXX) and an oily trimethyl ester, $C_{13}H_{22}O_6$ (CXXVIII). These reactions are closely parallel to those exhibited by 3-dehydroswietenolide. The above data along with their NMR spectra settle the constitutions of carapin and maxicanolide as (CXXIXa) and (CXXIXb) respectively.

It is, however, interesting to note that carapin and maxicanolide like swietenine and swietenolide contain bicyclo[3:3:1]nonane moiety and all of them show similar structural characteristics, but the base promoted cleavage of 1,3-dioxo derivative of swietenine series occurs in the C-2—C-3 bond and in the case of maxicanolide and swietenolide fission occurs at the C-9—C-10 bond. Thus, the position of the isolated double bond is of supreme importance to determine the course of the above reaction. Mention may also be made that all the four bicyclononanolides can be biogenetically derived from (CXXVI), the precursor of andirobin and methyl angolensate. So it is expected that through the fission of C-2—C-30 bond (involving retro Michael reaction) in the molecule of this series, a common meeting ground can be evolved.

Summary

Advances in the chemistry of terpenoid furanolactones during the last decade have been reviewed under the following headings: (1) Sesquiterpenoid furanolactones; (2) Diterpenoid furanolactones; and (3) Tetranortriterpenoid furanolactones. As a result a tentative biosynthetic scheme has been proposed covering the skeletally related C_{26} modified triterpenes.

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REVIEWS

MECHANICS: Berkeley Physics Course, Vol. 1, by Charles Kittel, Walter D. Knight & Malvin A. Ruderman (McGraw-Hill Book Co. Inc., New York), 1965. Pp. xviii+480. Price $ 5.50

The Berkeley Physics Course is designed to reflect the tremendous revolutions in physics in the last hundred years like special relativity, quantum physics and statistical physics. The research frontier is moving at an exponential rate and the book under review is a successful attempt at bridging the gap between the physicist working in the forefront and the college student majoring in science and engineering. The book gives a lucid exposition of the following important aspects in mechanics: the principle of Galilean invariance of physical laws, Coriolis force, charged particle in uniform constant electric and magnetic fields, cyclotron accelerating principle, non-relativistic dynamics, conservation of linear and angular momentum, harmonic oscillator as a model in classical and quantum physics, Euler's dynamical equations of motion of a rigid body, gyroscopes, gravitational and electrostatic self-energy, Doppler effect, speed of light in inertial frames in relative motion, Lorentz transformations of length and time, momentum and energy in relativistic dynamics, principle of equivalence of inertial and gravitational mass, and the stable and unstable particles of modern physics. Application of the principles of mechanics to astronomy, geophysics, chemistry and biophysics are amply dealt with. Each chapter ends with a film list, a book list with commentaries on their scope and form, advanced topics consisting of important relevant material for the superior student, problems essential to the development of the subject matter, supplementary mathematical notes and historical notes comprising pioneer original research papers. One of the vital parts of the volume is the drawings 'created' by E. D. Commins and F. Cooper. These novel features make the volume a unique feat in the art of writing undergraduate physics text-books.

L. RADHAKRISHNA

ELECTRICITY AND MAGNETISM: Berkeley Physics Course, Vol. 2, by Edward M. Purcell (McGraw-Hill Book Co. Inc., New York), 1965. Pp. xviii+459. Price $ 5.50

The book is in the form of a broad introduction to the subjects of electricity and magnetism. The treatment is highly instructive and the presentation maintains the desired level of clarity for students.

Equal emphasis has been given to the physical and mathematical aspects of the subject.

The treatment of the subject with the vectors will be found very useful for later studies. The usually confusing magnetic vector potential and its usefulness have been very clearly explained.

The book is highly useful for science and engineering courses.

D. SEETHAPATHIRAO

RELATIVITY AND THE NEW ENERGY MECHANICS by Jakob Mandelker (Philosophical Library, New York), 1966. Pp. xii+84. Price $ 4.00

This is a very well written book with a fine get-up and nice printing. Frequent use has been made of illustrative diagrams which contribute to clarity. The book provides a thorough analysis of the formalism of special relativity and a detailed critical examination of the physical meaning and significance of the various mathematical steps involved, such as the Lorentz transformed quantities, the law of addition of velocities, the variation of mass with velocity and the work-energy relationship. The world-metric of special relativity is regarded as a kinematical restriction on the motion of the particles rather than any geometrical structure associated with the world. The author attempts to search for the physical causes underlying this kinematical limitation imposed on motion. A new 'matter-energy mechanics' has been developed taking the inertia of any energy as the basic principle and the variation of mass with velocity as a logical necessity thereof. With the help of these two assumptions special relativity mechanics is reinterpreted in terms of the classical mechanics of a particle of varying mass. The limiting velocity of motion is regarded as due to a self-induced 'impedance' arising out of inertia of all energy including the kinetic energy. A new concept of contracted velocity is introduced and a new law of addition of velocities is derived which is in accordance with the result of Fizeau's experiment. An important modification has been introduced in the work-energy relationship.

The present treatment is a radical departure from the current relativistic viewpoint in the sense that the classical concepts of absolute space and time are retained in full as basic forms of cognition which have been rejected by special relativity at the expense of great psychological discomfort to many. The whole emphasis has been laid on discovering the underlying physics of the situation rather than on revising the fundamental concepts of space and time on looking for physical causes rather than on creating a new world-metric. But the concepts of absolute space and time are inextricably linked with the concept of simultaneity at distant places. If information from one place to another is propagated with finite speed, absolute simultaneity has no meaning from the observational point of view. The author does not discuss this problem which is the very genesis of the relativistic concept of space-time. He dismisses the question perhaps by saying that "because of its absolute, primordial-phenomenological status, space has basically to remain indeterminate and ungraspable by any physical means". How this concept of space can play an integral part in the formulation of physical laws is a debatable question. And, if it cannot, why should it be taken cognizance of in a physical theory at all?

If the author's viewpoint is accepted, it will necessitate a reinterpretation of the whole of the general theory of relativity. All the same, the book provides an interesting critical analysis of the special theory of relativity and this new approach along the old mechanistic lines of thought deserves close examination.

K. P. SINGH

CONSTRUCTION, REGLAGE ET ESSAIS DES INSTRUMENTS D'OPTIQUE by M. Lachenand (Dunod, Paris), 1966. Pp. xxvii+599. Price 98 fr.

The author of this book has very wide experience of designing and using various types of optical instruments in the Central Naval Artillery Laboratory, Paris. His experience includes design of several intricate optical instruments demanded by the French Navy during the world wars. He is also the author of several research papers published in *Revue d'Optique* and has carried out research at the French National Institute of Geography. He is also an adviser to several French industries engaged in manufacturing optical instruments.

The principal theme of this publication is a systematic study of the internal structure of different systems of optical images with emphasis on such aspects which are applicable in the case of visual type optical instruments. Different types of optical material like glasses, their characteristics for proper choice in the fabrication of instruments, other raw materials like plastics and artificial synthetic crystals useful in the infrared and ultraviolet regions are well described. Various types of optical instruments have been analysed, stress being on their practical applications. Details of several optical designs which cannot be easily calculated mathematically are presented and graphs depicting important relationships between the different parameters are given, making the book comprehensive.

The author has endeavoured to eliminate as far as possible purely academic data, which are of historical importance. A number of numerical solutions included enhance the value of the book greatly and many of these solutions use a purely mechanical method of calculation.

The first part of the book deals with optical materials used in the construction of various systems of images and their calculation, properties of plane mirrors, prisms, plane and parallel plates and physiological optics. In the second part, the author describes optical instruments used in astronomy, various types of optical microscopes, photographic objectives, optical instruments using reflection and semi-reflection, projectors and various other optical instruments. Calibration, standardization and testing of the optical instruments is discussed at length in the third and final part. Biographies of great discoverers in the field of applied optics are documented.

This book is thus a manual of 'optical gene' in which more emphasis is laid on geometrical optics than on physical optics and can be warmly recommended to all those engaged in the design and fabrication of optical instruments.

K. S. SARMA

THE ENCYCLOPEDIA OF PHYSICS edited by Robert M. Besançon (Reinhold Publishing Corp., New York, and Chapman & Hall Ltd, London), 1966. Pp. xii+832. Price $ 25.00

This encyclopedia has been compiled by the editor with the help of 320 leading authorities in various fields of physics. The volume will be primarily useful for physicists who wish to seek information regarding areas of physics outside their own. The encyclopedia will also be useful for workers in fields closely related to physics. Most of the articles have been written at a technical level, but each one carries enough descriptive material so as to be useful even to those who do not wish to follow the mathematics incorporated in some of the articles. The pains taken by the editor in compiling this volume will be greatly rewarded by its use all over the world. The references at the end of each article will help the readers in seeking more information than available in the articles, if they wish to do so.

R. P. SINGH

NEWER REDOX TITRANTS by A. Berka, J. Vulterin & J. Zýka; translated from the Czech by H. Weisz (Pergamon Press Ltd, Oxford), 1965. Pp. ix+246. Price 60s.

This is the English translation of a Czech monograph published in 1961. The German translation was published in 1964. The principal aim of the authors as set out in the preface is to survey the fresh ground covered in redox titrations since the publication of the two important books on the subject, viz. G. Jander's *Newre massanalytische methoden* (1956) and I. M. Kolthoff, R. Belcher, V. A. Stenger & G. Matsuyama's *Volumetric analysis: III — Titration methods; Oxidation-reduction reactions* (1957). Consequently, the conventional and well-established titrants, such as permanganate (in acid solution), dichromate and ceric salts, have been omitted from the treatment.

In all, there are thirty chapters, each chapter describing the use of one oxidant or reductant. The first fourteen chapters are devoted to oxidants, chapter fifteen deals with hydrogen peroxide which can function both as oxidant and reductant, while all the remaining chapters but one relate to reductants. Some of the less common redox reagents, such as potassium manganate, N-chlorobenzamide, 2,6-dichlorophenol indophenol, phenyl arsine oxide (which have only been investigated more recently) have been referred to in the last chapter. Fairly exhaustive references to the original papers are given at the end of each chapter. While trivalent cobalt in periodate and tellurate as well as tripositive cobalt in carbonate are among the most powerful oxidizing agents, their practical applications except in very special situations appear to be limited. Likewise, pentavalent and trivalent tungsten as reductants are of doubtful practical utility. Lead tetra-acetate holds promise because it is reported to differentiate between the *cis* and *trans* isomers of α-glycols, but the kinetics of the reaction may require further investigation.

Based on the authors' own experience, a critical evaluation of the reagents has been presented wherever possible. There are very few printing errors,

but one appears in page 121, second line under 'iron'. Iron (II) ions should read iron (III) ions. The general get-up is also attractive, consistent with the Pergamon tradition. The inquiring student as well as the professional analytical chemist will find much in this volume that will hold his interest.

P. R. SUBBARAMAN

ENGINEERING RADIATION HEAT TRANSFER by J. A. Wiebelt (Holt, Rinehart & Winston Inc., New York; *Distributors in India*: India Book House, Bombay), 1966. Pp. ix+278. Price $ 9.50

This book, consisting of eight chapters and six appendixes, will be welcomed by engineers and physicists who are working on the problem of radiative energy transfer at infrared wavelengths, a problem that is of considerable interest in space research. After introducing the necessary basic concepts of electromagnetic theory and thermodynamics of thermal radiation in the first two chapters, the author discusses the Planck's law of radiation, and the characteristics of 'black-body' systems and 'real systems' in the third chapter. The fourth chapter is devoted to the study of energy exchange between different bodies through radiation, and the basic concepts of 'flux algebra' are well presented. The integral equation for radiative heat transfer is deduced in the fifth chapter, and several methods of solving it are also given, stressing the relative merits of various methods. A numerical example, given to illustrate the solution of the integral equation, is quite useful to those who had no previous knowledge of numerical methods. In this chapter, the finite difference approximation of integral equation and the computational techniques for radiant systems are briefly and clearly presented. In the sixth chapter the author introduces non-gray, semi-gray systems, and discusses specular-reflectance and diffuse emittance of bodies. Here, the relation between the radiative, the conductive and the convective modes of heat transfer is briefly discussed. In the seventh chapter energy transfer in absorbing the emitting systems (particularly the gaseous media) is discussed. The experimental aspects of radiation detectors, radiation sources and pyrometers are given in the eighth chapter. The first three appendixes give data for total emittance, spectral emittance, and spectral reflectance of some of the engineering materials. The fourth appendix contains the mathematical formulae for radiant intercharge configuration factors, together with the numerical values represented graphically. The fifth appendix gives a programme written in Fortan language for IBM 1620 to solve radiant exchange problem for a non-gray diffuse surface enclosure. The sixth appendix gives tables of geometric mean beam lengths for isothermal gas radiation.

The presentation of the material is clear and the book should be equally useful to teachers of graduate and undergraduate students and to the students who wish to study by themselves. A number of problems and references at the end of each chapter enhance the value of the book. There is a good index at the end of the book.

G. S. MURTHY

EXPERIMENTAL METHODS FOR ENGINEERS by J. P. Holman (McGraw-Hill Book Co. Inc., New York), 1966. Pp. xii+412. Price $ 9.95

Experimentation is the cornerstone of scientific and engineering advances. Undergraduate laboratory training in engineering has not materially changed during the last few decades. The meagre resources for laboratories, lack of interest and foresight in planning new experiments, and the obsolescence of large-scale expensive equipment have focused national and international attention on how to remedy this serious lacuna. It is accepted by one and all that the problem of bringing the future engineer into contact with the real physical world by means of laboratory experience is of supreme importance.

Measurement is the backbone of experimentation. The basic engineering measurements of displacement, velocity, acceleration, force, temperature, flow and power have been dealt with in a lucid manner. Thermal conductivity, viscosity, diffusion coefficients, thermal and nuclear radiation measurements have also been included in the book.

The chapter on analysis of experimental data as also estimating accuracies of transducers and measurement devices is very well done. A brief treatment of control devices, and data processing is given in the last two chapters. In the appendix are included physical properties of fluids and metals commonly encountered in engineering.

This is an exceedingly good book on experimental methods for mechanical engineers and is recommended without reservation for use in the engineering colleges for undergraduate students.

A. RAMACHANDRAN

GRAPHIC HANDBOOK OF CHEMISTRY AND METALLURGY by Pai Yen Loung (Chemical Publishing Co. Inc., New York), 1965. Pp. 144. Price $ 10.00

The book is a unique collection of graphs relating to important chemical, physical, mechanical and metallurgical properties of elements. In all a hundred graphs are presented from which one can learn at a glance the relationships that exist among elements in respect of several of their properties. An additional feature of the compilation is that the pages are split with identical graphs in the top and bottom half. This enables an easy comparison of one graph with another. For example, an alloying curve can be readily compared with the periodic curves of physical and chemical properties.

The book contains (1) a combined periodic table in which the Mendeleev and the Bohr tables have been printed together to establish a close relationship between the two; (2) a set of comparative periodic curves of chemical and physical properties of elements covering over forty different properties; and (3) comparative periodic binary alloying curves of elements.

Of considerable interest to the metallurgist will be the binary alloying curves which show that the alloying properties of metallic elements are also periodic in nature and related to other periodic properties, such as atomic radius, electronic structure and crystal structure. In these graphs the

alloying elements have been grouped into five classes depending upon their mutual solubility or formation of chemical and intermetallic compounds. Thus at a glance one can obtain a fund of information about the alloying properties of elements in relation to their other properties.

This elegant presentation of well-organized graphs of periodic properties of elements will be valuable to scientists and engineers alike.

J. BALACHANDRA

A BIBLIOGRAPHY OF INDOLOGY (ENUMERATING BASIC PUBLICATIONS ON ALL ASPECTS OF INDIAN CULTURE): Vol. 2 — INDIAN BOTANY, Part II (K-Z): Compiled by V. Narayanaswami (National Library, Calcutta), 1965. Pp. xxx+412. Price Rs 8.25

Towards the end of 1961, the National Library, Calcutta, brought out the first part of this valuable bibliography comprising a list of all botanical works published under authors' names beginning with A to J; the second part covers the remaining alphabets, K to Z. The literature covered in these two volumes comprises work done in India or on Indian botany from 420 B.C. to the end of 1958-59 and includes mainly floristic work pertaining to India and some of the neighbouring areas. This comprehensive compilation has been possible by the devoted work of the author, while he was in the Botanical Survey of India, where as Systematic Botanist, he had accumulated a list of all the literature published on various aspects of floristic botany and which he supplemented by a visit to some of the botanical institutions of India after retirement. Thus, the bibliography includes literature on morphology, anatomy, embryology, cytology, cytogenetics and economic botany.

There are very few source books of this nature furnishing information on the work done in India in the various branches of botany. Rev. Blatter published the first such bibliography on taxonomic botany in 1911, and this has been recently brought up to date by Rev. Fr Santapau, who listed all publications up to 1952-53. These two bibliographies are, however, restricted only to systematic or taxonomic botany, while the present work is more broad-based. Besides giving the names of the authors and titles of their works, it contains also brief notes regarding their contents. More than 11,000 entries have been reported, part one listing 5374 entries and part two containing 5764 entries. A special and valuable feature of this bibliography is the inclusion of a large number of works in Indian languages, ancient and modern, the existence of which is not known to many botanists.

Difficulties are encountered in such a compilation, and minor discrepancies, omissions and errors are inevitable. On the bibliographic side, a serious discrepancy that attracts attention is the way the names of Indian authors are indexed. For example, Prof. M. O. P. Iyengar is listed under his personal name, Parthasarathi Iyengar, although he is well known to botanists all over the world as Iyengar and majority of his publications are under this surname. In citing authors' names, the criterion should be the name as used in the publications.

Regarding the coverage of botanical works, the inclusion or exclusion of titles appears to have been based on the author's interest. For a systematic botanist, the dates of publication of various parts, particularly of old taxonomic works, like those of Roxburgh, Wight, Royle, etc., are necessary and no information is available on these aspects. For example, the various volumes of Wight's Icones were published on different dates and a note on them appears in Wight's Icones, Volume VI, page 7. Secondly, in indexing the various families of Flora of British India, some of the parts are not covered. For example, under T. Anderson, the family Guttiferae is missing, and similarly the family Leguminosae by J. G. Baker, the families Simarubaceae, Ochnaceae and Burseraceae by A. W. Bennet. There is no reference to *Wealth of India — Raw material series*, in Part I or Part II, though in the introduction this work has been cited as a work of reference on economic botany.

These omissions and discrepancies apart, the publication should be welcomed by all research workers in Indian botany. The author as well as the National Library, Calcutta, deserve the gratitude of all botanists.

K. R. RAMANATHAN

DOCUMENTATION RESEARCH AND TRAINING CENTRE ANNUAL SEMINAR 3: DEPTH CLASSIFICATION, SUBJECT HEADING (Documentation Research & Training Centre, Bangalore), 1965. Pp. 569. Price Rs 20.00

Ever since the inception of the Documentation Research & Training Centre (DRTC) at Bangalore, the rate of progress in classification research has been pretty fast. Several new ideas have sprung up during this period. The papers contributed to the third seminar organized during December 1965 by the DRTC on depth classification and subject heading represent, in fact, a measure of the progress made so far. The methodology for designing depth schedules based on the postulates and principles evolved by Dr S. R. Ranganathan has resulted in the construction of a number of schedules for many basic classes. As many as ten papers contributed to this seminar amply demonstrate the methodology propounded by Ranganathan. The mixed notation used for Colon classification has received the greatest attention for further improvement and refinement, and the paper "Zero increases hospitality" discusses the addition of another zone for array isolates, making zero as an empty digit. This is bound to prove very helpful. The difficulties that arise in the choice of a helpful sequence of quasi-characteristics have been overcome by taking quasi-characteristics in groups and deciding their sequence. This idea has been discussed in a paper by Neelameghan and Gopinath. Dr Ranganathan gives a lead in the compilation of classified personal bibliography. There is an interesting paper on classification systems by Wahlin.

The two papers on subject heading are very important. In the first, Dr Ranganathan traces the development of 'chain procedure' as a method of deriving subject headings. In discussing the potentialities of this method, he comes out with

a definition for a basic class. In the second paper, Neelameghan and Gopinath demonstrate the use of auxiliary words and individualizing elements for resolving homonyms in subject headings. This massive volume includes the papers as well as the proceedings of the seminar.

T. N. RAJAN

BOOK NOTE

BASIC CONCEPTS IN QUANTUM MECHANICS by Alexander Kompaneyets (Reinhold Publishing Corp., New York), 1966. Pp. vi+152. Price $ 3.95

This book is an attempt to present the intricate concepts of quantum mechanics without sophisticated mathematics — a difficult but desirable one — demanding only a good knowledge of geometrical optics from the reader. Thus it serves as a valuable introductory book on the subject. Throughout these pages, the author has endeavoured his best to enable the reader who is familiar with classical mechanics, to envision the entirely new ideas like uncertainty principle, quantization of energy and angular momentum, electron spin, etc., in a simple manner with a judicious use of optical and acoustical analogies. However, the task of explaining more advanced topics inherently intertwined with mathematical formulas, e.g. Dirac theory, becomes formidable as the author himself could not but confess that "the beauty of the Dirac theory cannot be conveyed in words without formulas, any more than a musical composition can without notes or sounds".

PUBLICATIONS RECEIVED

MODULATION, RESOLUTION AND SIGNAL PROCESSING IN RADAR, SONAR AND RELATED SYSTEMS by R. Benjamin (Pergamon Press Ltd, Oxford), 1966. Pp. xii+184. Price 55*s*.

APPLICATION OF FRACTURE TOUGHNESS PARAMETERS TO STRUCTURAL METALS: Vol. 31, edited by H. D. Greenberg (Gordon & Breach Science Publishers, New York), 1966. Pp. x+406. Price $ 10.50 (paper); $ 24.95 (cloth)

FUNDAMENTALS OF RADIOBIOLOGY: Vol. 5, by Z. M. Bacq & P. Alexander (Pergamon Press Ltd, Oxford), 1966. Pp. xii+562. Price 24*s*.

GENERAL AND INORGANIC CHEMISTRY FOR UNIVERSITY STUDENTS by J. R. Partington (Macmillan & Co. Ltd, London), 1966. Pp. xxiii+926. Price 72*s*.

LABORATORY HANDBOOK OF TOXIC AGENTS edited by C. H. Gray (The Royal Institute of Chemistry, London), Second edition, 1966. Pp. ix+190. Price 24*s*.

COMPILATION OF MASS SPECTRAL DATA by A. Cornu & R. Massot (Heyden & Son Ltd, London), 1966. Pp. 617. Price £ 14.10; $ 42.00

PHOTOELECTRETS AND THE ELECTROPHOTOGRAPHIC PROCESS by V. M. Fridkin & I. S. Zheludev (D. Van Nostrand Co. Inc., New York), 1966. Pp. xii+195. Price $ 4.95

TECHNIQUES IN FLAME PHOTOMETRIC ANALYSIS by N. S. Polue'ktov (D. Van Nostrand Co. Inc., New York), 1966. Pp. xvi+219. Price $ 4.95

INSTABILITY CONSTANTS OF COMPLEX COMPOUNDS by K. B. Yatsimirskii & V. P. Vasilév (D. Van Nostrand Co. Inc., New York), 1966. Pp. ix+206. Price $ 4.95

THE MÖSSBAUER EFFECT AND ITS APPLICATION TO CHEMISTRY by V. I. Gol'danskii (D. Van Nostrand Co. Inc., New York), 1966. Pp. 119. Price $ 4.95

PORTLAND CEMENT TECHNOLOGY by J. C. Witt (Chemical Publishing Co. Inc., New York), 1966. Pp. xiii+346. Price $ 16.50

HANDBOOK OF DIFFERENTIAL THERMAL ANALYSIS by W. J. Smothers & Yao Chiang (Chemical Publishing Co. Inc., New York), 1966. Pp. ix+633. Price $ 17.50

SECOND SYMPOSIUM ON CATECHOLAMINES, Pharmacological Reviews, Vol. 18, No. 1, March 1966, edited by George H. Acheson (Williams & Wilkins Co., Baltimore), 1966. Pp. 803. Price $ 15.00

REFRACTOMETRY AND CHEMICAL STRUCTURE by S. S. Batsanov (D. Van Nostrand Co. Inc., New York), 1966. Pp. 250. Price $ 4.95

INDUSTRIAL CHEMISTRY — INORGANIC: ROYAL INSTITUTE OF CHEMISTRY MONOGRAPHS FOR TEACHERS X, by D. M. Samuel (The Royal Institute of Chemistry, London), 1966. Pp. 46. Price 7*s*. 6*d*.

LABORATORY HANDBOOK FOR OIL AND FAT ANALYSTS by L. V. Cocks & C. Van Rede (Academic Press Inc., New York), 1966. Pp. xxiv+419. Price 90*s*.

THE PRINCIPLES OF ELECTROMAGNETIC THEORY AND OF RELATIVITY by Marie-Antoinette Tonnelat; translated from the French by Arthur J. Knodel (D. Reidel Publishing Co., Dordrecht), 1966. Pp. xii+475. Price 70 fr.

THE PEPTIDES — Vol. 2: SYNTHESIS, OCCURRENCE AND ACTION OF BIOLOGICALLY ACTIVE POLYPEPTIDES by Eberhard Schröder & Klaus Lübke (Academic Press Inc., New York), 1966. Pp. xxvii+632. Price $ 30.00

NOTES & NEWS

Electronic halftone image recording technique for antenna radiation patterns

A new technique of using an electronic storage tube to generate a mosaic of diameter-modulated dots to record the intensity of two-dimensional antenna patterns has been developed at the Advanced Technology Corporation, Timonium, Md, USA. In this technique, a Tektronix 564 recording oscilloscope is used with auxiliary circuits in which the video signal from the pattern scanner is passed through a halftone dot generator before being displayed on the oscilloscope, to provide a uniform, medium resolution reliable display. Such images can be stored for about 1 hr with no degradation in either image quality or intensity. The video signal is sampled at regular time intervals to provide about 60 samples per fast scan line. Each sample pulse initiates a set of two waveforms which are decaying sinusoids in phase quadrature, which are added to the sweep signals at the 'X' and 'Y' inputs to the oscilloscope. They cause the beam to trace out a small logarithmic spiral pattern at a point on the screen determined by the values of the sweep voltages at the sampling instant. The frequency and the decay rate of the spiral are chosen so that the turns of the spiral run together and it appears as a round spot, its diameter being proportional to the voltage of the corresponding video sample. As the probe scans at a uniform rate, a sequence of dots is generated and distributed uniformly on the viewing surface. The sampling interval is made adjustable to maintain about 60 dots per horizontal line in spite of changes in the rate of scanning. Although halftone registration of the radiation pattern is obtained at the cost of resolution, the technique will be useful where it is necessary to examine the gross characteristics of a large number of patterns quickly. Also, it will be possible to recover, to an accuracy determined by the selected dot spacing, quantitative image intensity information by measuring the size of the stored dot with a magnifying optical comparator [*Proc. Inst. elect. electron. Engrs, N.Y.*, **54** (1966), 319].

Laser-induced thermal etching of metal and semiconductor surfaces

Observations made at the National Physical Research Laboratory, Council for Scientific & Industrial Research, Pretoria, on thermal etching using a laser beam for the first time, on metal and semiconductor surfaces, have revealed a new type of etch pattern, viz. formation of etch hillocks, in thermal etching. Earlier studies on thermal etching on metals were made by evaporation and are characterized by two mechanisms, viz. (a) the formation of grooves at the grain boundaries of polycrystalline metals and (b) terrace or striation formation on the surface of single crystals. The formation of etch hillocks in the process of thermal etching might be the result of the unusual method used to produce thermal etching. The method of laser-induced thermal etching may find applications in the study of thermal etching of surfaces under high vacuum or at a low ambient temperature, since laser radiations could be focused through a window.

In the experiment conducted, the output from a ruby laser was focused using a 1·95 cm. focal length lens on the polished surface and subjected to only one pulse. This process usually produced a superficial crater with a diameter of about 1·2 mm. The etched area was replicated using the selected area technique. The replica is a shadow-cast primary replica suitable for investigation by transmission electron microscopy. In order to determine whether the laser produced surface structures were pits or hillocks, a second replica of the same area was made and this area was then sprayed with a dilute solution of polystyrene latex particles in water. The dried replica was then shadowed and coated with carbon as before. A comparison of the directions of the shadows of the latex then confirmed the type of feature to be hillocks. The presence of hillocks could also be shown by making a 'thorough focus' observation on the original metal surface with a high-powered optical microscope [*Nature, Lond.*, **210** (1966), 191].

ClCO, a new free radical

A new free radical, ClCO, has been generated, identified and chemically characterized. An intermediate in the reaction of chlorine to phosgene, ClCO, which has so far eluded direct observation, has been generated using the matrix isolation technique wherein the free radical is produced from a mixture of the source of the free radical (HCl) and an inert gas diluent by irradiation at liquid hydrogen temperatures. The IR spectrum of the free radical thus produced is taken with an IR double beam prism-grating spectrophotometer attached to the cryostat.

HCl, frozen at 77°K. to remove non-condensable gases, was mixed with argon and chemically pure carbon monoxide and the mixture was allowed to strike the cesium iodide window of a cryostat from a jet directed at an angle of 45° with respect to the window. It was possible to conduct simultaneous deposition and photolysis either with an external radiation source directed through the observation window of the cryostat or with the vacuum ultraviolet photolysis lamp. All observations were made at 14°K. The three vibrational fundamentals were located at 281, 570 and 1880 $cm.^{-1}$. The reaction was found to take place with virtually zero activation energy [*NBS tech. News Bull.*, **49** (1965), 204].

A simple synthesis of cyclopropene

The existing methods for the synthesis of cyclopropene are extremely laborious. A simple cyclopropene synthesis, based on Fischer and Applequists' preparation of 1-methylcyclopropene from

methallyl chloride and sodium amide, has been achieved.

The reaction involves dropwise addition of allyl chloride to a stirred suspension of sodium amide and, after the completion of the addition, sweeping the product into a trap by passing nitrogen at a slow rate through the system. The cyclopropene thus formed can be freed of the contaminants by vapour phase chromatography. Cyclopropene is identified by its NMR spectrum and its Diels-Alder reaction with cyclopentadiene.

The reaction mechanism involves the formation of vinylcarbenes and subsequent cyclization to cyclopropene derivatives. In spite of the low yields (10 per cent) under various conditions tried, the simplicity of the method and the ready availability of the starting materials make the method commercially superior to the older ones [*J. org. Chem.*, **31** (1966), 638].

A new aldehyde synthesis

Aliphatic and alicyclic carboxylic acids, with a few exceptions, can now be readily converted to the corresponding aldehydes by selective reduction of the acid chloride or the phenyl ester with lithium tri-*tert*-butoxyaluminohydride (LTBH) in tetrahydrofuran [*J. org. Chem.*, **31** (1966), 283].

Equal volumes of 1*M* solution of tri-*tert*-butoxyaluminohydride and the phenyl ester in tetrahydrofuran are allowed to react at about 0°C. for 4 hr. The aldehyde is formed in 70 per cent yield. The uniqueness of the new reagent is illustrated by the fact that neither lithium aluminium hydride nor its tri-*tert*-methoxy derivative could produce even a trace of the aldehyde under the experimental conditions, even after 24 hr.

Notable exceptions to this otherwise general reaction of preparing aldehydes are phenyl cyclopropylcarboxylate and phenyl benzoate.

Synthesis of 9-phenanthrol and anthrone

A method applicable on a large scale has been devised for the synthesis of 9-phenanthrol and anthrone starting from phenanthrene and anthracene respectively, giving overall yields of 75 per cent. In a three-stage synthesis, copper (I) catalysed nucleophilic conversion of the aryl halides into the alcohols via their alkyl ethers is utilized. The bromo derivatives, viz. 9-bromophenanthrene and 9-bromoanthracene, are refluxed under nitrogen for 16 hr with a solution containing 2,4,6-collidine, copper (I) iodide and an excess of sodium methoxide. Hydrolysis of the resulting ethers, with 1*N* hydrogen bromide in acetic acid in the case of 9-methoxyphenanthrene, and with hydrogen chloride in aqueous acetic acid in the case of 9-methoxyanthracene, yields 9-phenanthrol and anthrone. The novel feature in these syntheses is the use of a particular homogeneous system to effect copper (I) catalysed nucleophilic replacements, $ArHal \rightarrow ArOAc$, accompanied by little or none of the reductive dehalogenation, $ArHal \rightarrow ArH$, which was the predominant reaction previously observed between aryl halides and alkoxides in heterogeneous system containing copper (I) oxide as catalyst [*Chem. & Ind.*, (1966), 812].

Structural assignment of hydroxy analogues of coenzyme Q

Several compounds in the coenzyme Q group have been described in which one of the two methoxy groups has been replaced by another substituent, but no technique is available so far for distinguishing which of the methoxy groups has been replaced or modified. A new procedure utilizing NMR spectroscopy has now been developed which allows unambiguous structural assignments of such compounds. The procedure involves the conversion of a substituent in the quinone nucleus into a deuterated methoxy group followed by ring closure to the chromenol or chromanol derivative. The separate absorptions of the two methoxy groups in the NMR spectra of the chromenol and chromanol have been structurally assigned, and the comparison of the structure of the deuterated derivative with that of its undeuterated analogue allows unequivocal structural assignments.

Certain hydroxyquinones related to both coenzyme Q_7 and Q_{10} have been assigned structures in the past on the basis of the understanding that such hydroxyquinones were single compounds. The new method has revealed that these compounds are in reality approximately 50 : 50 mixtures of the two possible hydroxy derivatives. It has also been possible to apply this method for solving the orientation of the amino and methoxy groups in rhodoquinone [*J. Amer. chem. Soc.*, **88** (1966), 564].

Determination of water in nonpolar liquids

A new method based on isotope dilution and involving the exchange of the water present in the sample with D_2O has been developed for the analysis of water in nonpolar organic liquids. The method avoids the difficulty of calibrating with organic-water standards of conventional methods and eliminates the correction for isotope effects if water is determined directly by tracer techniques. For the determination, a known amount of the sample is equilibrated with a known amount of heavy water at constant temperature and the resulting change in the heavy water concentration is determined by the infrared difference spectroscopic method. After making the necessary blank correction for the traces of light water and the nonpolar liquid contamination in heavy water, the solubility is calculated from the difference in concentration of heavy water and the amounts of heavy water and the sample taken.

A series of solubility measurements were carried out for water in benzene at various temperatures and from the data, thermodynamic functions of solution, ΔG, ΔH, ΔC_p and ΔF for the transfer of 1 mole of water from the benzene phase to the vapour phase were calculated. The method is accurate up to $\pm 0{\cdot}8$ per cent. However, it is restricted to liquids having a low solubility in water and without any exchangeable hydrogen [*Can. J. Chem.*, **44** (1966), 1365].

Spot test for iron

A novel and simple procedure for the detection of iron, free from interference from foreign

ions has been reported. 1-Phenethyl-4-carbethoxy-2-oxo-3-hydroxypyrroline, known to occur exclusively in enolic form and highly soluble in chloroform, is found to be an effective reagent for the detection of iron (up to 12 p.p.m. as ferric chloride), present in common organic liquids and inorganic salts. Dilute solution of the reagent in chloroform (10 per cent) gives a deep red-coloured iron chelate in the organic layer, when shaken with aqueous solutions of ferrous and ferric salts. The colour of the chelate is not masked by the presence of common cations and anions except carbonate, cyanide, citrate and oxalate. The interfering anions can be precipitated by adding excess of silver nitrate solution before testing. The chelate is stable between *p*H 1·9 and 4·0. Though quantitative estimation of iron had been achieved by comparison of the colours produced in control experiments using known concentrations of Fe^{3+}, the solutions show considerable deviations from Beer's law [*Chem. & Ind.*, (1966), 1341].

Mechanical strength of alkali-aluminosilicate glasses

The methods used to strengthen glass and its limitations have been surveyed in a recent issue of the *Philips research reports* [1966 (*Suppl.*) (No. 3), pp. 106]. The survey has shown that the mechanical strength of glass is very largely determined by the tensile stresses produced in the glass by loading (round surface damage, such as cracks, which act as stress concentrators). The production of compressive stresses in the surface layer of the glass is found to give a decided increase in mechanical strength. The effect of exchanging a small ion against a bigger one in alkali-aluminosilicate glasses at a temperature below the strain point of the glass has been studied and it has been found that appreciable stresses are built up in this way, in directions parallel to the surface. The magnitude of these stresses is mainly determined by the composition of the glass and the total number of ions exchanged. Maximum stresses are built up in glasses where the molar ratio Υ between the alkali oxide (Me_2O) and Al_2O_3 is equal to 1 (compressive stresses up to 100 kg./mm.²) for the exchange of K^+ for Na^+ ions. If the difference ΔTb between the treatment temperature and the strain point of the glass becomes smaller than about 100°C., viscous relaxation of stresses begins to play a role and, depending on the treatment time, maxima can occur in the stress as a function of the concentration (and as a function of the distance to the surface; the maximum stress does not occur at the surface in this case). The variation of the stress (and the stress-relaxation process) can be described mathematically with the aid of an iteration process. A structural change occurs during the ion exchange. The ion-exchanged glass has a higher density than the glass of the same composition obtained by melting (even in the absence of stresses). This 'densified' structure can relax to a normal structure in the same temperature range as that in which relaxation of the stresses occurs. If some of the sodium ions in a sodium-aluminosilicate glass are replaced by lithium ions, the stress built up by ion exchange with K^+ ion is less than for the lithium-free glass, although the difference in ionic radius between Li^+ and K^+ ions is greater than that between Na^+ and K^+ ions. The mechanical strength of the glass after ion exchange decreases little (by 5-25 kg./mm.²) when the glass is given a standard damage, as long as the thickness of the ion-exchanged layer does not fall below a certain (fairly critical) value. For sodium-aluminosilicate glasses after ion exchange with K^+ ions, this layer thickness is about 70 μ. If half of the sodium in these glasses is replaced by lithium, the layer thickness required is strangely enough smaller (about 30 μ). The measured strengths of the glasses (up to 100 kg./mm.²) agree more or less with the maximum compressive stress in the glasses (as long as the layer is thick enough). The interdiffusion coefficient (rate of ion exchange) and the activation energy of the diffusion process in the glasses investigated depend strongly on the concentrations of the alkali ions taking part in the ion exchange and on the composition of the original glass. The interdiffusion coefficient as a function of $1/\Upsilon$ (at a constant alkali-oxide content) has a maximum at $1/\Upsilon = 1$ and a minimum at $1/\Upsilon = 0{\cdot}3$-$0{\cdot}5$. Introduction of lithium in the original glass lowers the interdiffusion coefficient (of K^+ or Ag^+ ions for Na^+ and Li^+ ions). The chemical resistance to HCl and HF shows a sharp change at the composition $Me_2O.Al_2O_3.4SiO_2$ (more general in a certain composition range with an SiO_2 content of 66·7 mole per cent). In the series of glasses investigated, the chemical resistance as a function of $1/\Upsilon$ (at a constant Me_2O content) is relatively high at $1/\Upsilon \approx 0{\cdot}3$. It is assumed that the aluminium ion in aluminosilicate glass occurs in three different configurations. At each temperature and each composition an equilibrium exists between these structural units. At $1/\Upsilon = 1$, practically only $Al(-)O_4$ groups are present, and there are no non-bridging oxygen ions. In the range $0{\cdot}3 < 1/\Upsilon < 1$, non-bridging oxygen ions are formed (in SiO_4^- groups) and AlO_6^- groups are formed from $Al(-)O_4$ groups. At low values of $1/\Upsilon$ (less than 0·2-0·3), aluminium is mainly in the form of AlO_6^- groups. At $1/\Upsilon > 1$, aluminium is mainly in the form of 'triclusters' (AlO_{4Cl}).

Physics Education

This new journal devoted to the cause of raising the standard of the teaching of physics is being published from May 1966 by the Institute of Physics and the Physical Society, London. With an editorial board consisting of eminent physicists and highly experienced teachers, the journal aims to bring to its readers information on new methods, techniques and apparatus for the teaching of physics. The first issue contains articles on various aspects of physics which will fill in the background for readers in different specialized fields as well as articles on fields where physics overlaps on other disciplines. The journal will be a valuable addition to the libraries of all educational institutions. Further information regarding subscription, etc., may be had from the Institute of Physics

and the Physical Society, 47 Belgrave Square, London SW1.

Physics and Chemistry of Liquids

Commencing from February 1967, Gordon & Breach Science Publishers will publish this new interdisciplinary periodical devoted to experimental and theoretical studies on liquids — metallic and nonmetallic. The emphasis will be on fundamentals rather than on applied fields. The subscription rate will be $ 25.00 per volume consisting of four issues for institutions and $ 9.50 for individuals.

Progress Reports

International Tin Research Council

The annual report of the International Tin Research Council for the year 1965 presents briefly the progress made during the year in various research projects relating to the chemistry and metallurgy of tin and indicates the trends for future work.

Research in the tinplate field was concerned mainly with the study and development of testing procedures, investigation of coating structure and the study of passivation films. The guiding principle has been to develop the maximum degree of protection from the tin coating, without a significant increase in processing costs. Nucleation and growth processes of the compound layer at the tin-steel interface have been investigated. Both nucleation characteristics and crystal form of the intermetallic layer have been found to be influenced by preplating with thin coatings of another metal, especially nickel or tin-nickel alloy. Methods of isolating the metallic layers in the tinplate coatings have been much improved. A method developed uses selective anodic dissolution at controlled potentials, thus enabling the isolation of either the entire coating or the compound together with any undercoating metal. A simple rapid technique for nondestructive determination of $FeSn_2$ in the coating using X-ray diffraction has been developed and this may form the basis of a procedure for continuous monitoring of the thickness of the intermetallic layer on the production line. A quick method of estimating the trivalent chromium in the passivation films of tinplates has been developed based on the potential variation of tinplate during anodic polarization. It has been found that some mild steels with normal chromium content when held in critical near passive conditions, such as chemical polishing, develop chromium-rich surfaces — a finding which may have a bearing on the preparation of steel for electroplating.

From studies on the role of tin additions in cast iron, it has been found that both tin and chromium promote a fully pearlitic matrix in flake iron. But chromium in excess would cause 'chilling' at corners and hard carbide spots, whereas tin besides being free of such disadvantages can stabilize the pearlite against breakdown at elevated temperatures. An addition of 0·3 per cent tin, instead of the usual 0·1 per cent, has been advised. Owing to its action as a solid solution hardener and certain other undesirable effects on the properties of steel, tin is not being used as an addition in steel making. Studies are in progress to overcome such difficulties and steel to the specification EN 32B with 2 per cent tin has been fabricated, without undue difficulty. Aluminium and nickel did not give encouraging results as additions to tin-steel, but cerium showed some beneficial effects. It has been found that tin when added to low carbon steels has no effects on the rate of spheroidization or the rate of diffusion of carbon.

Dispersion-hardened tin prepared by extruding compacted tin powder has been found to have higher strength than cast tin and much improved creep properties at high temperatures. The room temperature strength and hardness of the extruded material increased with decreasing particle size; but this increase in hardness is more than compensated by the accompanying decrease in ductility. Dispersion-hardened tin, individually and in combination with a tin-steel backing, is being tried as a bearing material.

Other metallurgical studies carried out during the year include preparation and investigation of bronzes containing Group IVA transition metals, structural studies of bronzes manufactured by the continuous cast method with intermittent withdrawals, and soldering using tin and tin alloys.

Synthetic and mechanistic studies of polytin compounds through the hydrogenolytic fission of tin-nitrogen bonds by organotin hydrides have been carried out. A polar mechanism involving an electrophilic attack of the organotin hydride hydrogen on nitrogen has been proposed. A number of polymetal compounds with germanium and tin in the side chains and several polymers with alternating tin and germanium atoms in the main chains have been prepared using hydrogenolysis. A study of tin-tin bonds has been initiated.

Announcements

▪ *An International Symposium on the Physics of Selenium and Tellurium*, sponsored by the Selenium-Tellurium Development Association Inc., New York, will be held in Montreal, Canada, during 12-13 October 1967. Papers will be presented covering all aspects of the physics of selenium and tellurium, like crystal growth, optical properties and electrical properties by participants from USA, UK, USSR, France, Germany, Czechoslovakia and other countries. Enquiries regarding the symposium should be addressed to Selenium-Tellurium Development Association Inc., 11 Broadway, New York.

▪ *The Canadian Congress of Applied Mechanics* will be held at Laval University, Quebec City, during 22-26 May 1967, as part of Canada's Centennial Year activities. Engineers and scientists may submit papers in solid mechanics, fluid mechanics and thermodynamics, mathematics as applied to mechanics and on experimental methods. Abstracts in not more than 3 typed pages in French or English should reach Prof. W. D. Baines, Department of Mechanical Engineering, University of Toronto, Ontario, by 31 January 1967.

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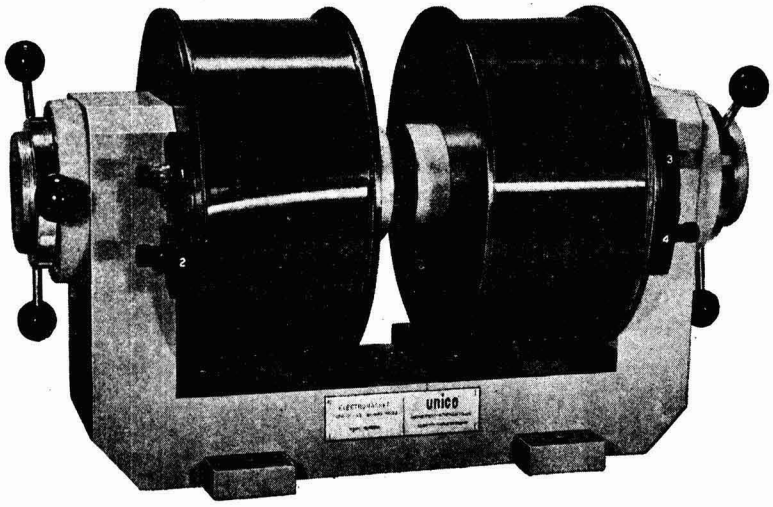

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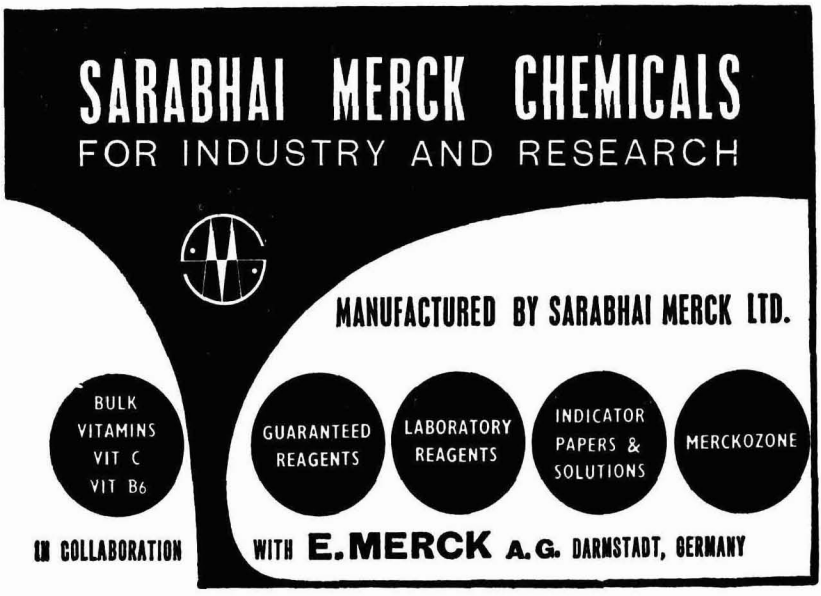

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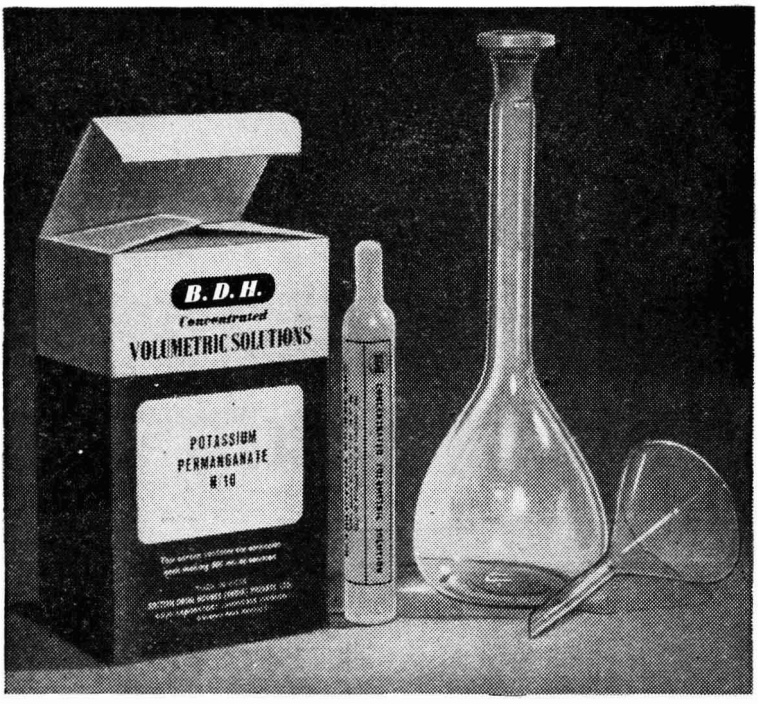

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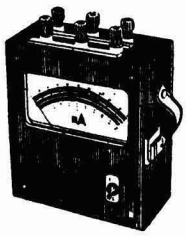

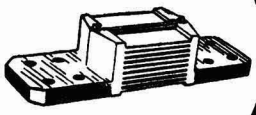

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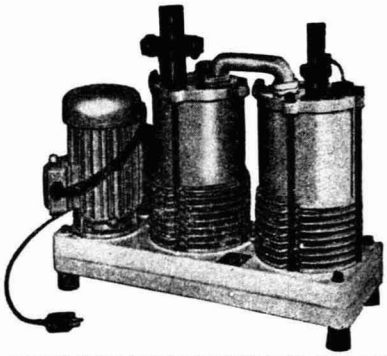
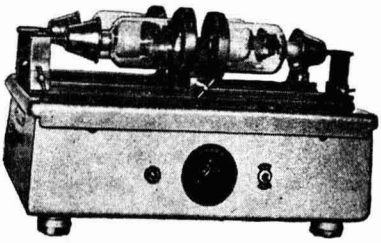


DOUBLE STAGE ROTARY VACUUM PUMP, TYPE Z-30



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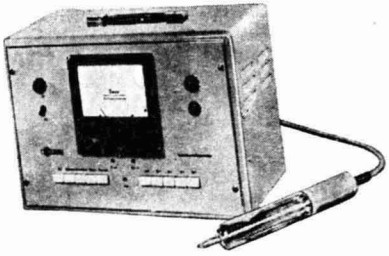

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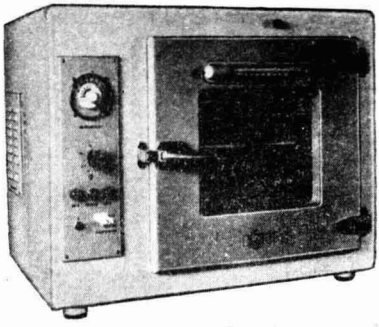

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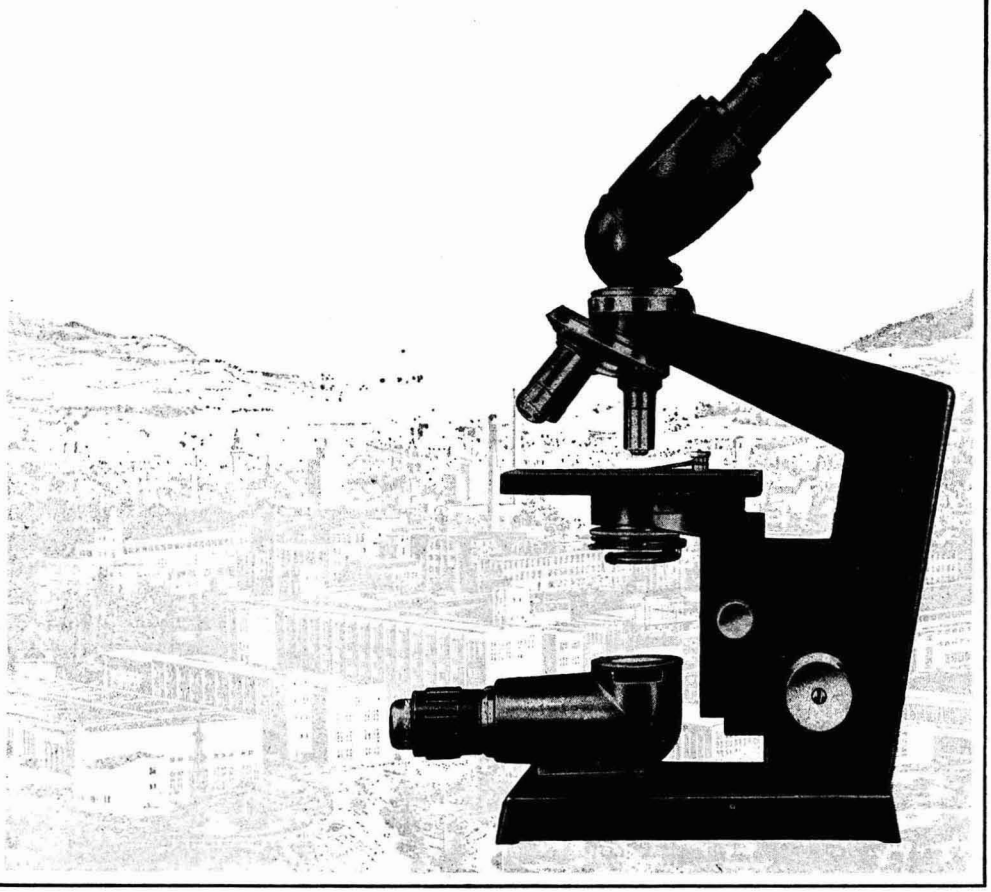

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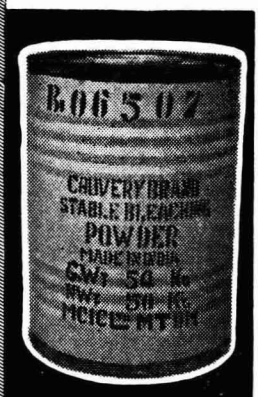

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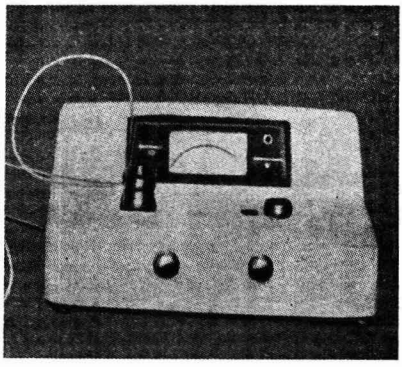

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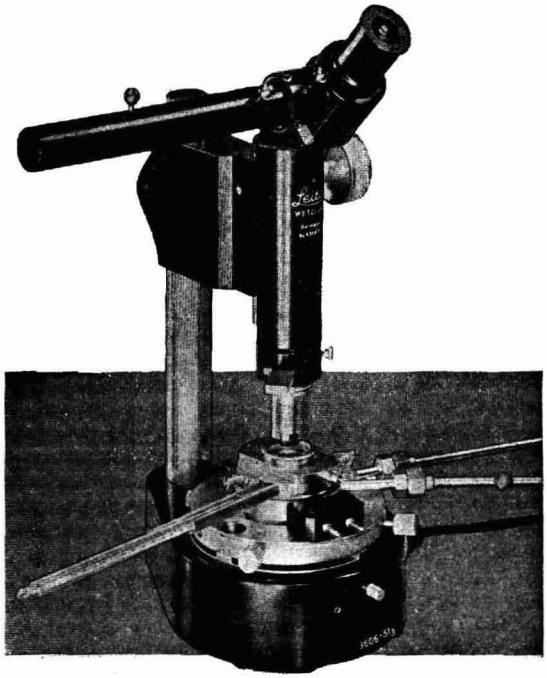

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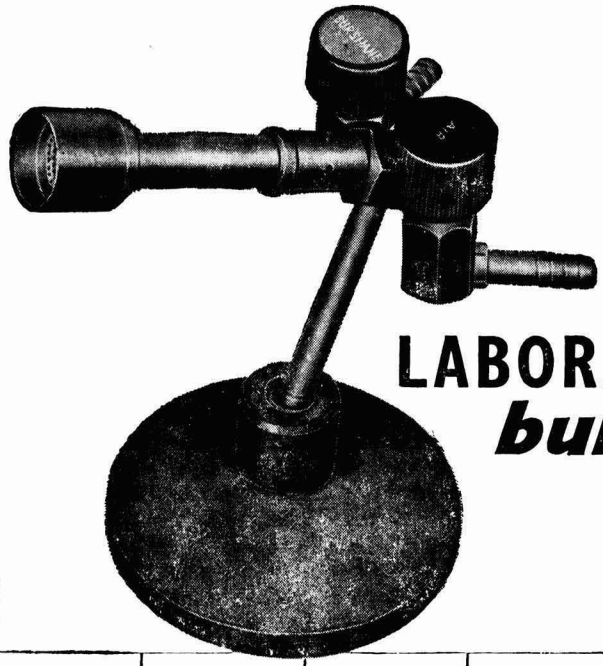

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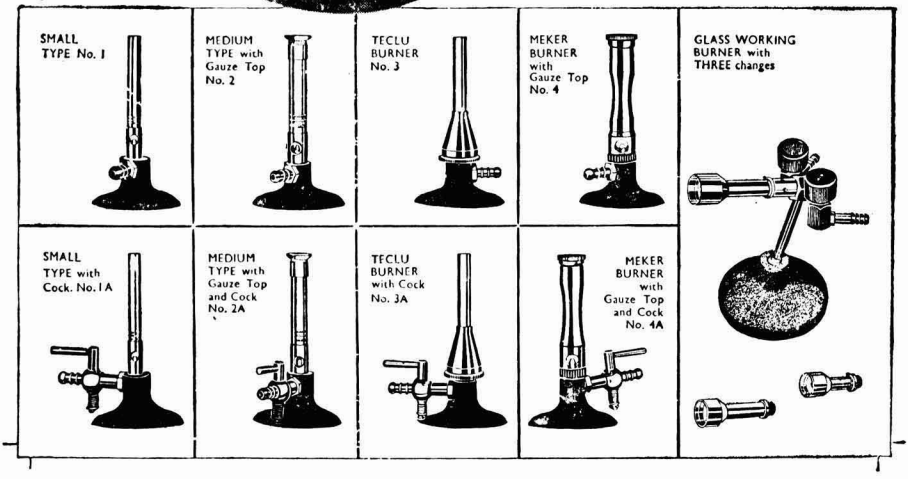

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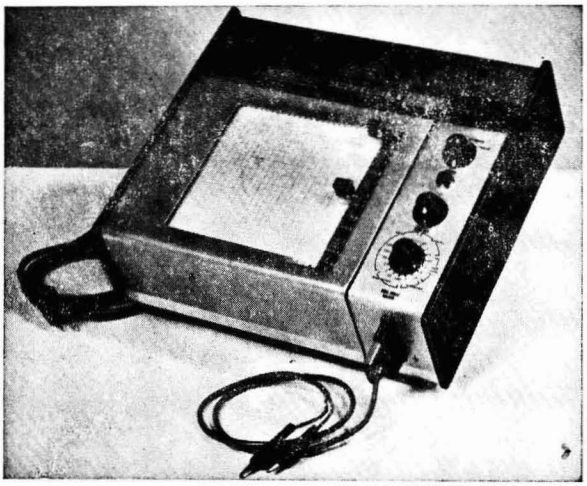

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