

Journal of Scientific & Industrial Research



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✓ May 1966

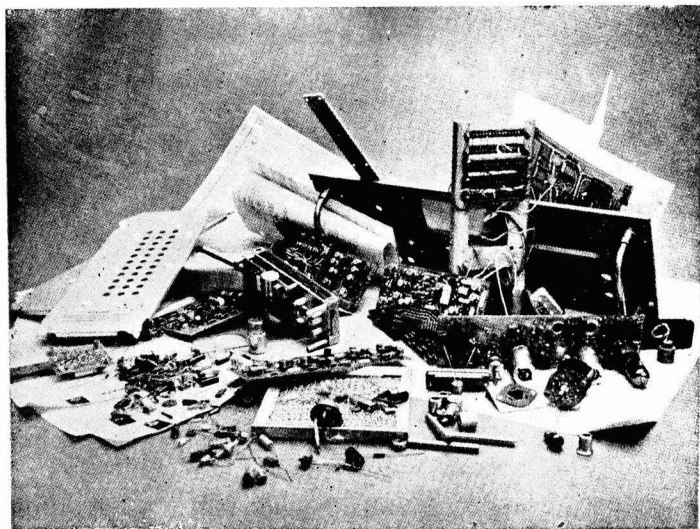
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ERRATUM

The bibliographic details of the book "Statistical Theories of Spectra: Fluctuations" reviewed in the January 1966 issue [25 (No. 1) (1966), page 32] are incorrect. The review, with correct bibliographic details, is published again on page 219 of this issue.

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Journal of Scientific & Industrial Research

VOLUME 25

NUMBER 5

MAY 1966

CONTENTS

CURRENT TOPICS

| | |
|--|-----|
| Production of Special Biochemicals | 191 |
| Information Centre on International Physics Activities ... | 192 |

| | |
|---------------------------------|-----|
| Laser & Its Applications | 193 |
| S. DEB | |

| | |
|--|-----|
| Cosmic Rays, Elementary Particle Physics & Astrophysics — A Symposium | 196 |
| K. A. NEELAKANTAN | |

| | |
|--|-----|
| Synthesis of Azasteroids Using Beckmann Rearrangement & Schmidt Reactions | 200 |
| HARKISHAN SINGH, VEERENDRA V. PARASHAR & S. PADMANABHAN | |

| | |
|----------------|-----|
| Reviews | 218 |
|----------------|-----|

Fundamentals of Semiconductor Devices; The Dynamics of Conduction Electrons; Optical Model of the Atomic Nucleus; Statistical Theories of Spectra: Fluctuations; Organic Chemistry; Organic Reactions: Vol. 14; Theoretical Basis of Inorganic Chemistry; Instrumental Methods of Analysis; Bridged Aromatic Compounds; Methods in Carbohydrate Chemistry: Vol. 5 — General Polysaccharides; Tenth Symposium (International) on Combustion; The Sorby Centennial Symposium on the History of Metallurgy; Improving the Effectiveness of Research and Development; Elements of Structural Geology; The Rational Use of Dyes in Biology; Metabolism of Steroid Hormones; Animal Gametes (Male); A Morphological and Cytochemical Account of Spermatogenesis

| | |
|---------------------|-----|
| Notes & News | 225 |
|---------------------|-----|

Pion with spin (spion); Plasma arc spraying; 1,4-Dihydropyridine; A novel acetylation; Memory fixation in goldfish; Active centre of phosphoglucomutase; Codons for termination and release of polypeptide chains from ribosomes; RNA-dependent RNA-polymerase; Linkages in glycoproteins; Binding of amino acids and peptides to gelatin; Preparation of specifically labelled milk proteins; Ahmedabad Textile Industry's Research Association; Dr S. R. Valluri; *NPL Technical Bulletin*

For Index to Advertisers, see page A21

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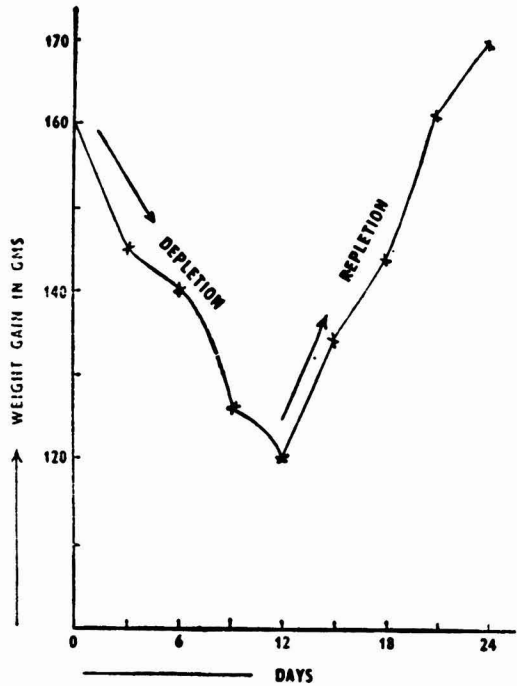
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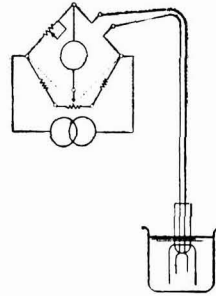


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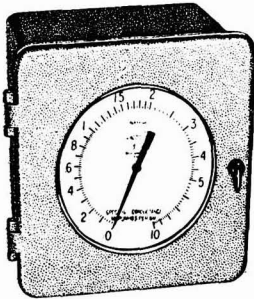
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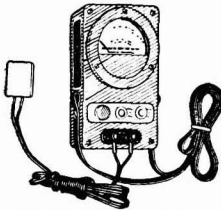
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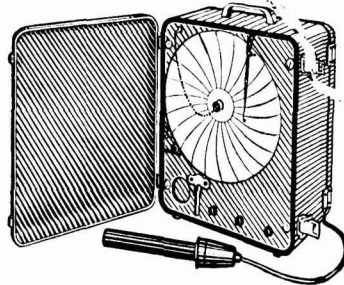
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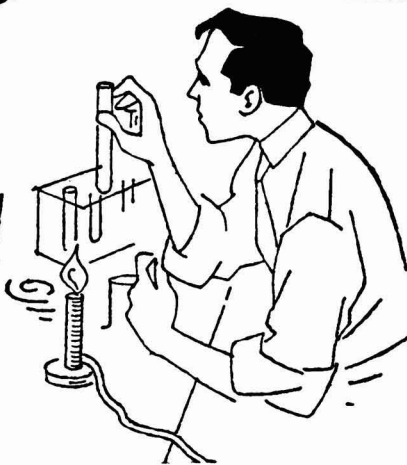
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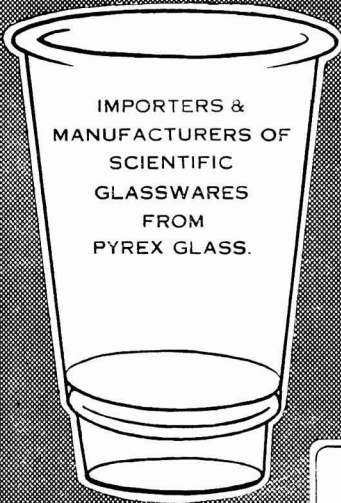
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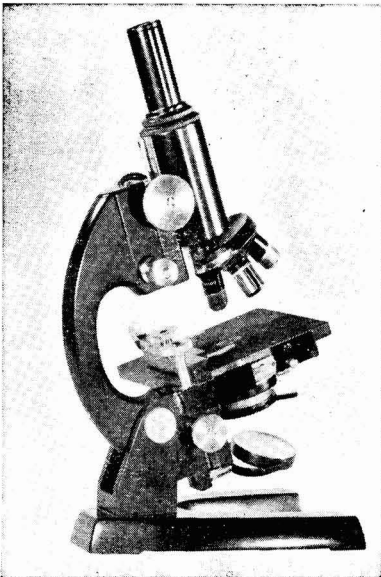
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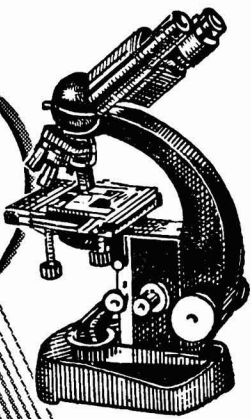
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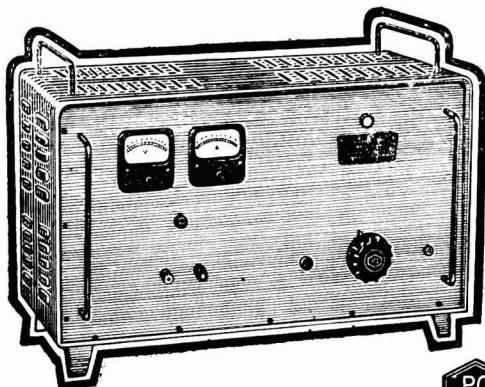
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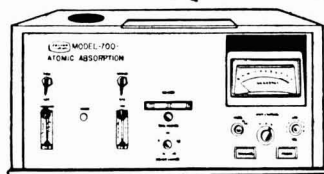


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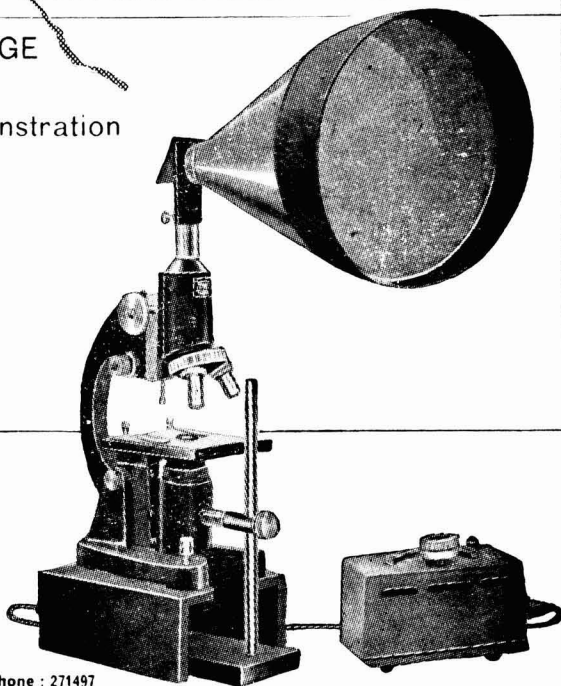
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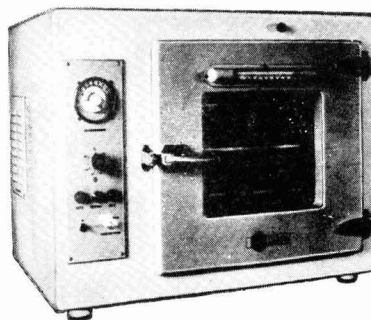
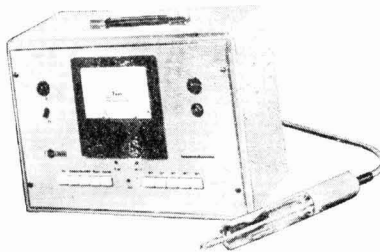
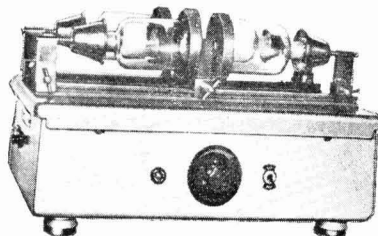
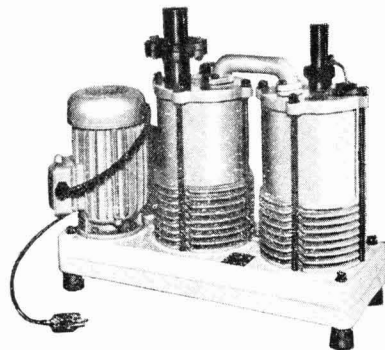


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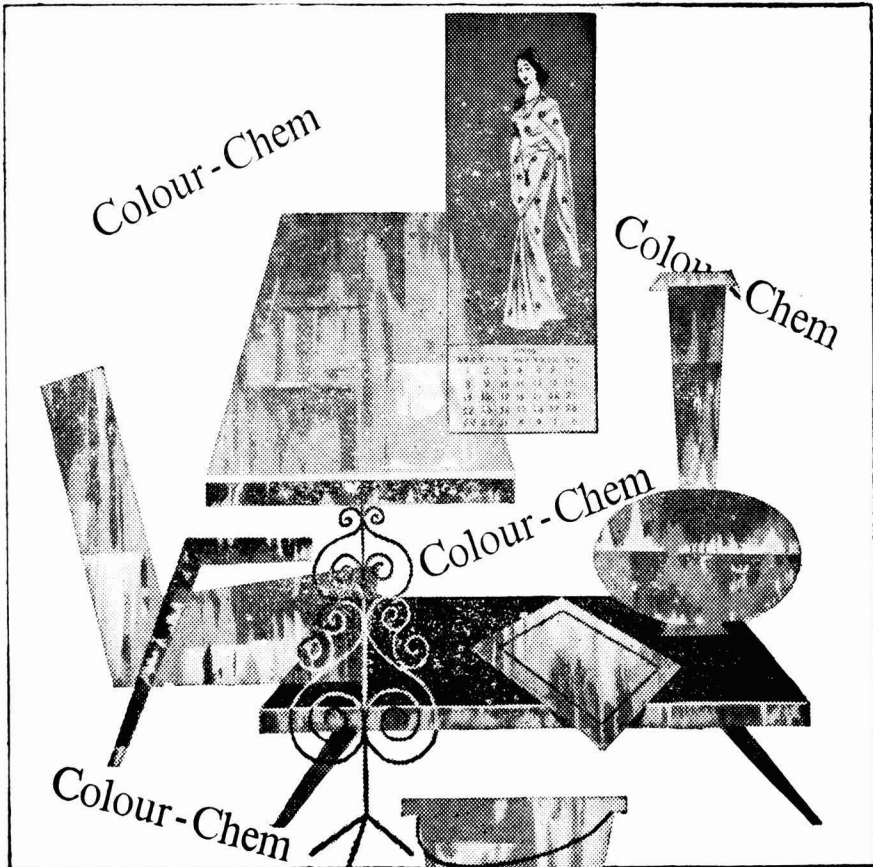
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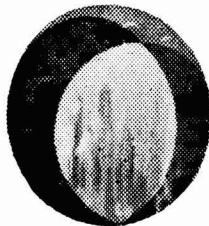
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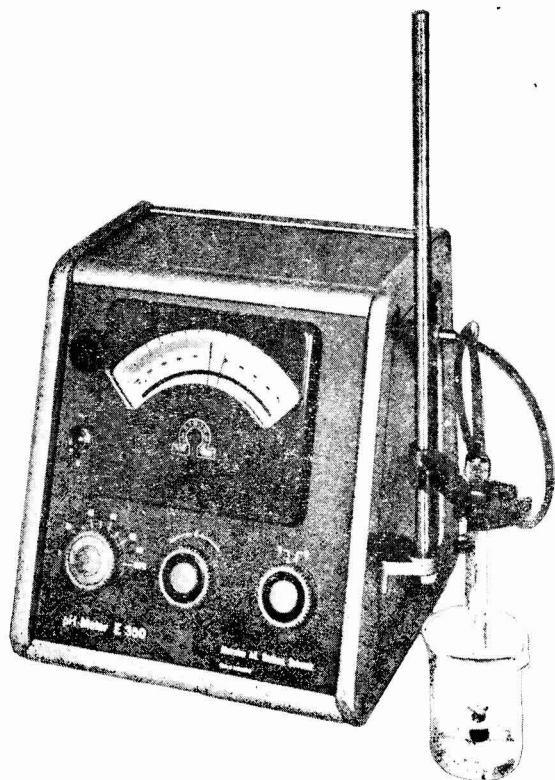
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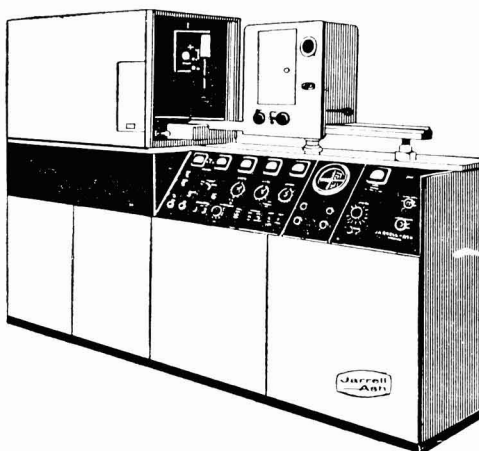
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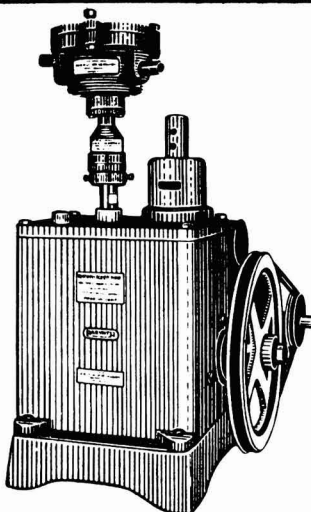
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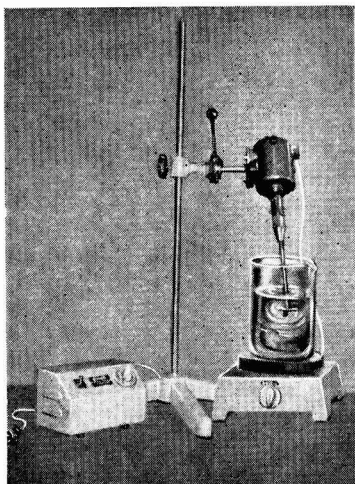
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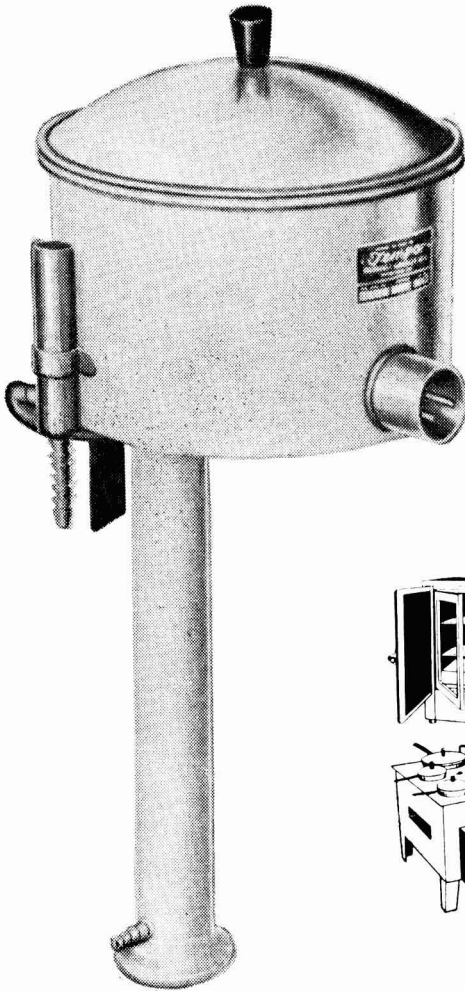
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INDEX TO ADVERTISERS

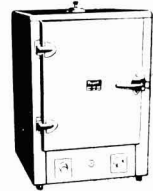
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| GANSONS PRIVATE LTD., BOMBAY | A7 | RATIONAL SALES ASSOCIATES, BOMBAY | A21 |
| GHARPURE & CO., CALCUTTA | A30 | SCIENTIFIC INSTRUMENT CO. LTD., ALLAHABAD | A2, 32 |
| GORDHANDAS DESAI PRIVATE LTD., BOMBAY | A33 | SCIENTIFIC SALES SYNDICATE, BOMBAY | A12 |
| INDIA SCIENTIFIC TRADERS, BOMBAY | A24 | SETT & DE, CALCUTTA | A24 |
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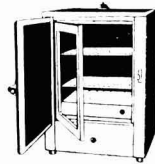
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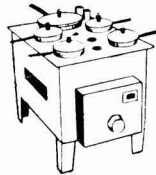
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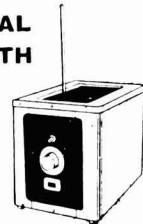
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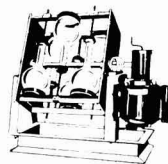
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Current Topics

Production of Special Biochemicals

IN recent years there has been considerable expansion in the teaching of and research in biochemistry in the country. Biochemical research is actively pursued in many research institutions, universities and in the laboratories attached to medical and agricultural establishments. Efforts are also being made to introduce biochemistry in all the universities. However, this expansion in biochemical teaching and research has to be matched by adequate supply and ready availability of specialized biochemicals and equipment required to maintain the standard of teaching and research. Also, the growing demand for biochemicals, both in quantity and variety, by clinical laboratories has to be met. It is estimated that more than 100 biochemicals, which are not produced in the country, are needed. It is in this context that the setting up of the Special Biochemicals Unit by the Council of Scientific & Industrial Research (CSIR) at the Vallabhbhai Patel Chest Institute, Delhi University, should be considered as an important development. The work of the biochemist in India is greatly hampered owing to the difficulty in importing many of the essential biochemicals because of serious shortage of foreign exchange. The inauguration of the unit on 6 April 1966 by Shri M. C. Chagla, Union Minister for Education and Scientific Research, should pave the way for overcoming this difficulty.

A Committee was appointed by the CSIR in 1963 to assess the needs of the country for various biochemicals. Based on an analysis of the replies received to a questionnaire sent to over 300 research establishments, the Committee found that more than 100 biochemicals are imported and the demand for these products far exceeds the supply. The Committee recommended that the production of biochemicals needed for teaching and research should be undertaken. The CSIR accepted the recommendation and decided to set up a unit for this purpose. The unit has been located at the Vallabhbhai Patel Chest Institute where an active division of biochemistry exists. Availability of raw materials (slaughter house products, yeast, etc.) and facilities for designing and fabricating the equipment needed have been the main considerations in locating the unit at Delhi. Above all, its location in Delhi University campus represents yet another example of the growing and fruitful collaboration between CSIR and the universities.

As set up at present, the unit represents only a modest beginning. A tentative budget of Rs 3 lakhs has been provided for 1966-67, of which Rs 2 lakhs are earmarked for equipment. The unit will be gradually expanded as it gains experience in production methods and acquires the necessary

complement of personnel and equipment. The unit started functioning from January this year and on the opening day, the Union Minister for Education and Scientific Research was presented with token samples of the first two products—adenosine triphosphate and cytochrome *c*—produced by the unit. It is proposed to produce gradually other products, such as nucleotides, phospholipids, phosphatides, enzymes and co-enzymes, sugar derivatives and amino acids. However, it is advisable that the unit works out a system of priorities, so that the preparation of products which are comparatively in greater and urgent demand is taken up.

Production of over 100 different biochemicals by a single unit is a formidable job and it cannot be tackled by merely expanding the size and facilities of the unit. Several specialized skills and training in different branches of chemistry and unit operations are required for the purpose. It is not easy to assemble all of them under one roof. A number of research establishments in the country have already perfected processes for the preparation of a variety of biochemicals and some of them have been produced on a semi-pilot plant scale. For example, the Central Drug Research Institute, Lucknow, has worked out processes for the preparation of over 30 biochemicals from indigenous raw materials and some of them on a large scale. It is desirable that such work is consolidated, and ways and means of active collaboration between the Special Biochemicals Unit and other laboratories engaged in or capable of preparing biochemicals are explored. This should be done not only to avoid duplication of effort but also to pool the available resources of trained personnel and specialized equipment to maximum benefit. The unit could perhaps serve as a centre for coordinating all activities concerning the production and supply of biochemicals in the country. It could also function as a nucleus for imparting training to workers in unit operations in preparative biochemistry as well as in the standardization of biochemicals.

The unit when fully expanded and equipped should be in a position to design and fabricate the equipment needed as well undertake the production of biochemicals on request. Also, it should then be able to undertake investigations on the shelf-life of biochemicals, and methods for their storage and preservation under tropical conditions. All these aspects may as well be discussed at a symposium of practising biochemists so that a more definite picture can be had of the problems involved in the preparation and supply of biochemicals in India.

The future growth of the unit and its successful working will largely depend on the active and willing cooperation of all the biochemists in the country.

The unit has been established primarily to serve the biochemists, and they have to be active partners in the work of the unit and ensure its success. The expenditure on the unit, even when it is fully expanded, will only be a small fraction of the large sums of money spent on research in life sciences, apart from the returns expected from the sale of the biochemicals. And, therefore, any investment on this project should be considered worth while and should receive full support as it may be the forerunner of a thriving fine biochemicals industry in the country.

Information Centre on International Physics Activities

TO meet the growing need for a central clearing-house of information on physicists with international interests, an Information Centre on International Physics Activities is now in operation at the American Institute of Physics under a grant from the National Science Foundation. The centre collects information on a worldwide basis about international commissions, conferences, and institutes, the location and principal officers of foreign academies of science, physics organizations, research and university centres, educational activities in physics, and the names and itineraries of foreign physicists coming to the US and of US physicists going abroad. The centre publishes an "Information Booklet on Physics Organizations Abroad", listing much of this material. The centre promptly answers individual questions, often by identifying other sources of information. Communicating across national boundaries, this new information centre serves the widening interests of physicists everywhere.

The observations of F. Behn Reggs, Director of the Centre [*Bull. Atom. Scient.*, (1965), 34-35],

on this centre, with particular reference to needs of the developing countries in respect of educational and technological assistance should receive due consideration by all those concerned with aid programmes and research planning. He observes: "For continued growth of an educational structure capable of supplying the men and women needed by the new technologies, the existing institutes and universities must strive towards a development free of external experts and consultants. Necessary though the experts may be during the early stages of a nation's development, the newly trained technical people cannot be proud of their responsibilities and achievements unless they are moving towards independence of foreign assistance. Their dignity, the vitality of their education, and the effectiveness of their research require it. It is equally valid that except for rare individuals, a foreign adviser cannot fully understand the real wishes of the people, their way of life, their politics, history, religion and culture.

"Physicists in both developing and technologically advanced nations recognize a clear need for a free international exchange of technological and educational information. This information should be available to all, but without advice or value judgments arising from special interests. In the long run, we must be satisfied that people from countries outside of our own know what is good for themselves and that they are the best judges of the information they receive. Unesco's example here has guided others well. Free access to information is an antidote to experts who may give advice with authority but with only slight responsibility for the relevance of the advice to the situation. Short-term aid programmes may give this impression. Physicists from abroad, particularly those from developing countries, should have access to many sources of information so they themselves can weigh conflicting opinions."

Laser & Its Applications*

S. DEB

Department of Telecommunication Engineering, Jadavpur University, Calcutta 32

IF the justification for a scientific conference lies in the success in focusing attention to a comparatively less frequently discussed aspect of a subject, the one on laser, held under the auspices of the New York Academy of Sciences on 4-5 May 1965, may be taken to be a well-justified one. The particular aspect of laser on which the conference had been able to lay emphasis was its technological and biomedical applications. Thus, out of the 29 papers presented at the conference no less than 20 are related to this topic either wholly or partly. Of these, again, as many as 15 deal with the biomedical aspect alone — many of these giving concrete results — which imparts a realistic touch to quite a few high hopes built up, in recent years, around this new electronic gadget. A brief survey of the proceedings of the conference should, therefore, be of interest.

Fundamental Science Aspects of Laser

The opening paper by J. Weber presents the basis of laser action. Of particular interest are some of the less well-known links in the chain of events in the earlier history of maser. Reference is made to the author's published work on the subject in 1953, i.e. a year before the first publication by C. H. Townes, and also to that of G. Gould in 1951. One gains the impression that these works complicate to some extent the issue of priority in the field. The next paper by W. R. Bennett deals with the advances in gas lasers since January 1963. The stimulated pumping type and double cascade lasers have been discussed. Experimental results of studies on radial profiles and saturation characteristics of He-Ne laser are given. Discussion on high power gas laser forms an interesting part of the paper. This laser requires an energy level configuration, such that excitation (pump) energy is comparable to $h\nu$ and upward transition between the two lowest states is possible through collisional encounters but not thermally. The population in the upper metastable state can thus be large. Sulphur is one such medium which satisfies these requirements (emission at $1\ \mu$ and a power of few watts/cm²). Another type of high power gaseous laser is that using ions of noble gases under which category Ar⁺ laser has already yielded quasi-continuous power of a few watts and pulsed power of hundreds of watts although at a very poor efficiency. A. Lempicki's paper on liquid laser describes, in a rather cryptic fashion, the pulsed europium benzoylacetonate laser giving emission at 6130 Å. at -100° to -150°C . The paper by B. Lax presents the basis and the operating characteristics of junction lasers, including the effect of an external magnetic field. An optical radar using such a laser is referred

to and the future possibilities of this and the cyclotron resonance laser are discussed. In the paper by A. Hardway Q-switching and the methods of generation of high pulsed power are discussed. The rotating mirror, the Kerr cell, the magnetic field control and the saturable filter type switching of a ruby laser are described. Only the abstract is available of P. A. Franken's paper on harmonic generation utilizing the quadratic term in the polarization-field relationship of certain media. Of particular interest is a rather exhaustive review by N. S. Kapany on fibre optics on which much of the possible flexibility of applied laser electronics depends. Transmission characteristics of glass-coated glass fibres and multiple fibres are quoted and waveguide character of these described. Quality of image formed by such fibres and the radiation damage of these fibres are also mentioned. Quartz is stated to be the best fibre material from the point of view of radiation damage. Purely fibre type lasers, recently developed, are also discussed with particular reference to their constructional features properties and optical switching. Fibre optics enables visual observation of remote parts of the body in biomedical applications and the paper also discusses photocoagulation of tissues by fibre optic probe and describes a laser fibre endoscope, hypodermic probe and a laser system for surface and retinal coagulation. Physics of thin films is discussed in the paper by O. S. Heavens. The author considers, among other things, broad band filters consisting of staggered layers and narrow band ones involving frustrated total reflection. S. P. S. Porto presents a survey of the work done in his laboratory on linear Raman effect experiments using laser as source. Six different types of sample holders are described and their light collection efficiencies are given. Details are given of several experimental arrangements along with results obtained from CCl₄ and C₆H₆ exposed to He-Ne laser radiation.

Technological Applications

R. A. Sherman, in his paper, describes the use of lasers for microdrilling and microwelding. It is pointed out that owing to the small cross-sectional area a laser beam cannot have high penetration in welding and massive metal removal. In microelectronics, however, its utility should be enormous. Absence of heat-affected zone also makes it the ideal choice for welding and drilling in semiconducting materials. A depth-to-width ratio 3.5:1 is obtained in joining light gauge metals and attachment of lead wires as fine as 1/2 mil becomes quite possible. Drilling of small holes in exotic and refractory metals is discussed. Illustrations are given, among other things, of formation of 1-3 mil slots in light gauge stainless steel, penetration of 1/8 in. Hastelloy C and hole drilling in nephrite stone. An experimental set-up for microdrilling

*Résumé of the proceedings of a conference on The Laser published in the *Annals of the New York Academy of Sciences*, 122 (Art³), 571-834.

is described and the procedure for obtaining good results is outlined. The paper by A. L. Bloom considers the application of laser in surveying, precision optical testing and alignment. The high degree of collimation, small effective source movement permitting a high degree of angular alignment and good discrimination against unwanted light are, it is pointed out, the factors which should make a laser extremely useful in precision survey work, while possibility of accurate alignment and exceptionally good time coherence are weighty considerations in its favour in interferometric applications. The point is illustrated by referring to the problems of alignment of the Twyman-Green interferometer, surface parallelism testing and optical polish testing of a surface. E. J. Woodbury discusses some aspects of a laser system design. He starts with a few basic relations in statistics and system probability and then enumerates the various sources of internal and external noise in a laser system. Effect of atmospheric turbulence is also taken into account. Range equations for single and double path transmission system, under various conditions of operation are given and these, in turn, give the output power required for a given range with a system. J. T. Verdeyen and J. B. Gerardo describe the use of laser for the determination of plasma refractive index. The theory of the interferometric technique developed by the authors is given. Briefly it consists of a gas laser whose output depends on the phase of a feedback signal which, in turn, is determined by the refractive index and hence the electron density of a plasma traversed by the same. Experimental results in support of the theory are also given. R. H. Wiley, in his short communication, examines the possibility of new photochemical effects in organic materials using laser light. Formation of diarylamine in methyl blue solution upon exposure to ruby laser pulses is reported.

Biomedical Applications

The subject of biomedical aspects of laser is introduced by N. S. Kapany who describes a number of instruments developed for investigation in this field. Other papers on the topic discuss broadly the effects of laser radiation on normal living cells and tissues and on enzymes, spectrochemical studies of tissues with laser beam, laser radiation therapy of malignant growths and treatment and damage of human retina by laser beams.

M. Bessis and M. M. Ter-Pogossian consider the general problem of micropuncture of cells by laser radiation. An apparatus utilizing a ruby laser is described. A schedule for preparation of the target is given and a convenient method of calculation of doses outlined. Possible approaches towards evaluation of results are discussed. In one case, the effect of death of an irradiated cell upon its environment was followed by time-lapse cinematography. The surrounding cells were found to travel towards it and phagocytize it in a few minutes. For a few individual cells, in the case of culture of *Euglena*, the neighbouring ones were, however, found to shy away for about 2 min. immediately following irradiation. N. M. Saks *et al.* describe a few experiments and the instrumentation involved

on microsurgery by ruby laser radiation on *Amoeba proteus* and *Nitella axillaris*. Presence of green pigments, e.g. chlorophyll or blue and green dyes, was found to enhance and localize the effect of irradiation. Cytoplasmic flow in amoeba often reversed direction upon exposure of a few 20 mJ. radiation. With sublethal doses the cell eventually recovered even after striking changes had been introduced. Repetitive irradiation led to cumulative effects resulting in a peculiar ballooning. With 100 mJ. pulses, the cytoplasm tended to pinch-off and in certain cases fragmentation of the amoeba followed. Nuclei irradiated with 20 mJ. pulses produced recoverable inhibition and spasmodic streaming of the cytoplasm. Irradiated amoeba showed significantly lower rates of reproduction than the non-irradiated ones. The growth rate of *Nitella* was found to be reduced by irradiation. D. E. Rounds *et al.* also found that exposure of pigmented cells to 1-2 J. ruby laser beam resulted in their death. The specimens used by Rounds *et al.* included those from human skin, human and animal retinal epithelium and also melanoma cells from mice and hamsters. Cardiac, skeletal and smooth muscle cells showed a decrease in rate of contractile activity following exposure, suggesting inactivation of certain enzymes. Rabbit endothelial cells showed a significant increase in giant cell formation and multinucleation from 3 to 5 days after exposure.

Laser-induced thrombosis in rats is discussed in a paper by J. A. Kochen and S. Baez. Blood cell adherence in the immediate vicinity of the exposed zone and thrombus formation were noticed within 1 min. of exposure to 15 mJ. radiation. Complete restoration of blood flow was observed 45 sec. after stoppage of lasing. Similar experiments on rat by these workers, described in a companion paper, showed that with small intensity only an enhanced vascular reactivity and adherence of colloidal carbon resulted in the region of lasing. Thrombus formation was found at higher intensities. Studies of flow of heparinized rat blood through glass capillary showed deposition of fused mass of red cells on the wall and evolution of gas bubbles. L. Goldman, in his paper, surveys the basis and experimental results on the biomedical effects of the exposure of human tissues to low and high energy laser beam. Black hair and skin were both found to be vulnerable to such exposures pointing to the necessity of adequate measures for protection of the skin while working with laser.

The observations of deeper probe into the mechanism of tissue damage are reported in the paper by M. Igelman *et al.* who describe experiments on the effect of ruby laser radiation on protein enzymes and enzymes containing porphyrin or DPN. The results are largely negative excepting for peroxidase which, however, is known to be inactivated by visible radiation as well.

Application of laser microprobe, capable of vaporizing microsites of biologic and pathologic substances, for spectrochemical analysis of calcified tissues is described in a paper by D. B. Sherman *et al.* With Q-switched ruby laser head giving 7-10 MW pulse a plume temperature of 10,000°K. could

be attained over a period of 1 min. and this was heated further to 80,000°K. by an auxiliary 2000 V. spark. Percentage compositions of inorganic materials of tooth, calculus and bones, based on spectral analysis carried out at such heated zones, are given.

Results of investigations on retinal damage due to laser radiation in rabbit eyes are reported by A. Kohtiao *et al.* Both pulsed ruby and CW gas lasers are used and it is stated that with shorter pulses a smaller total energy is needed to produce a given degree of retinal damage—the threshold for burn being 0.2 mJ. for pulsed and more than 2 mJ. for CW radiation. H. W. Straub, in his paper, outlines the genesis of a practical device for protection of human eye against laser beams. Employing an energy of 2×10^{-8} J. impacting on a 10 μ diam. area on the retina to be the safe limit, Straub recommends a filter composed of BG-18 Jena colour glass made by Schott and Genossen in Germany as a good protective device in battlefield where laser type fire control devices are likely to be extensively used in near future.

Retinal coagulation is considered in two papers. An equipment and the clinical procedure and technique are described in the paper by C. J. Campbell *et al.* Experimental results on 85 patients are given, there being a solitary case of failure for one having a large retinal tear with an operculum under traction. Another photocoagulating apparatus is described in the paper by H. M. Freeman *et al.* Results obtained with 9000 coagulations in pigmented rabbits and a number of human patients are described. The possibility of the use of laser for treatment of intraocular tumours—including malignant ones—is mentioned. It is pointed out that treatment with laser light involves very little risk of otherwise damaging the eye. Use of laser radiation in arresting malignant growth is discussed in two other papers. P. E. McGuff *et al.* describe results of treatment of autogenous hamster tumours and human patients with malignant melanoma. One 90 J. burst of radiation was found to be sufficient to cause dissolution of hamster tumour in 8 days after treatment on an average. In the case of the human patients two 180 J. bursts given 3 min. apart caused a dissolution in the third post-treatment week, even though subsequent microscopic examina-

tion of excised tissues revealed a slight trace of melanoma in one region. Some results suggest that in contrast to the case of X-rays, the entire tumour need not be exposed to radiation for dissolution. The skin which acts as a barrier between the radiation and affected region may have to be pigmented suitably for satisfactory results. The authors also describe fully the thermometric system used and outline briefly the possible mechanism of action of laser energy on the affected area. J. P. Minton *et al.* in their paper describe the results of treatment of Cloudman S-91 melanoma in certain mice with Nd and ruby laser radiation. These workers measured the temperature at various depths of the tumour after exposure. Deriving the relevant theoretical relation, the destruction of a given tumour is shown to be dependent upon the relationship between the diameter of the tumour and the amount of energy delivered and the absorption coefficient of the tumour for the radiation. The destructive effect is ascribed to thermal energy produced during impact of radiation, the tremendous pressure created beneath the skin being enough on occasions to cause bursting of the tumour.

Postscript by Weber

The special issue presenting the conference proceedings closes with a few stimulating comments from Weber. He emphasizes the papers on biomedical aspects and does not agree with those who make a radical distinction between maser type devices and vacuum tubes and are unable to see that in the latter case too energy is created out of stimulated emission from free electrons. Basing arguments on this fact, it is pointed out that both in respect of noise and power density the maser type devices still lag significantly behind the vacuum tube family. A comparison with the classical contribution by Young, Fresnel, Fraunhofer, Michelson, Fabry and Perot also leads him to conclude that relatively little has been achieved to date towards utilization of coherent light for investigation of fundamental phenomena in physics. In matters relating to the advancement of laser technique itself he holds a rather pessimistic view and concludes that greater advances are expected in application rather than in the excellence of lasers themselves.

Cosmic Rays, Elementary Particle Physics & Astrophysics— A Symposium

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THE Ninth All India Symposium on Cosmic Rays, Elementary Particle Physics and Astrophysics, organized by the Cosmic Ray Committee of the Department of Atomic Energy, Government of India, was held at the Tata Institute of Fundamental Research, Bombay, during 13-17 December 1965.

Cosmic rays, according to the present-day definition, consist of particle radiations, X-rays, γ -rays and neutrinos of extraterrestrial origin. There is definite evidence, from experiments carried out during the last IGY (International Geophysical Year), that the sun emits particles of high energies at times of intense solar activity. But the maximum energy of the particles emitted by the sun is approximately 10^{10} of the maximum energy of particles so far observed in the extrasolar or galactic cosmic radiation. The cosmic ray particles on entering the atmosphere interact with the nitrogen and oxygen atoms and produce secondary particles. In fact, many of the elementary particles like the positron, mu-meson, K-meson, lambda-hyperon, etc., were discovered from studies on the secondary cosmic radiation. Hence, research work on cosmic rays and elementary particle physics have progressed side by side for over a generation. Recently, the important suggestion that supernovae and quasi-stellar radio sources might be sources of cosmic rays has gained great importance from optical and radio astronomical studies. Further, the wide use of rockets and satellites in cosmic ray research including such fields as X-ray and γ -ray astronomy has brought to the forefront the cosmological and astrophysical aspects of cosmic radiation. In order to keep up with these latest trends in the development of the subject, astrophysics also has been brought under the scope of this year's symposium for the first time.

The delegates to the symposium were welcomed by Prof. R. R. Daniel, Chairman of the Cosmic Ray Committee. The late Dr H. J. Bhabha, Director, Tata Institute of Fundamental Research, and Chairman, Atomic Energy Commission, delivered the inaugural address. In his address, Dr Bhabha described how he started cosmic ray research in India with very modest means at the Indian Institute of Science, Bangalore, and how within about two decades large-scale facilities for cosmic ray research like the stratospheric balloons made by the Tata Institute of Fundamental Research and the equatorial rocket launching facility at Thumba have been developed in the country. He urged scientists in universities and research institutions to come forward with good ideas and make full use of these research facilities. The Tata Institute, he said, would be setting up within a couple of years, at Ootacamund, a large cylindrical radio telescope with a collecting area about five times that of the dish at Jodrell Bank in the United Kingdom. A number of optical

telescopes (40 and 48 in.) are also expected to come into operation at various parts in India. Dr Bhabha said that these, along with the radio telescope, would give considerable impetus to research in astronomy and astrophysics.

About 200 delegates attended the symposium and about 100 papers were read. A few foreign scientists who were in India at the time of this symposium also attended and made valuable contributions in the form of papers and discussions. The scientific programme was divided as follows: plenary sessions were held each morning when the present state of knowledge on the subjects covered was brought into focus by means of review talks. Contributed papers were classified under the following major sessions held in the afternoons: (i) modulation and solar terrestrial relations, (ii) primary cosmic radiation, (iii) astrophysics, (iv) extensive air showers, muons and neutrinos, (v) particle physics, (vi) high energy interactions, and (vii) cosmic ray history. Because of the large number of original contributions received, parallel sessions had to be held every afternoon.

Plenary Sessions

The first review talk, in this session, was given by Prof. B. M. Udgankar of the Tata Institute of Fundamental Research (TIFR), Bombay, immediately after the inauguration. His talk 'Particle physics — A survey' gave a bird's-eye view of some of the interesting areas in particle physics which have been the scene of intense activity and interesting developments during the last couple of years. He dealt with the theoretical aspects of the crucial experiment suggesting CP (charge, parity) violation carried out by Cristenson, Cronin, Fitch and Turlay of the Princeton University, USA, in the middle of 1964. He briefly surveyed resonances, symmetry and Regge-pole phenomenology.

Prof. M. R. Kundu (TIFR) gave a review talk on 'Solar activity at radio wavelengths'. He pointed out that radio observations of the sun at centimetre, metre and decimetre wavelengths, made during the IGY, have greatly enhanced our knowledge about processes taking place in the sun, when it is active. His talk was illustrated by a very interesting film brought from the United States which clearly showed phenomena like loops, prominences, flares, etc., originating on the sun.

Prof. K. R. Ramanathan (Physical Research Laboratory, Ahmedabad) gave a status report on the IQSY (International Quiet Sun Year) programme with particular reference to data collected in India. The active observation period of the IQSY was from 1 January 1964 to 31 December 1965. The Indian National Committee for the IQSY will continue its work of collecting and consolidating the data from India and arrange for their publication before the

end of July 1967, when a final international assembly and symposium will be held in London. Prof. Ramanathan remarked that India has a reasonably good network of observatories for meteorology, geomagnetism and ionospheric studies.

Dr P. V. Ramanamurthy (TIFR) gave an excellent review on the subject 'Neutrino physics and neutrino astrophysics'. He dealt with high energy neutrinos ($E_\nu > 10$ GeV.) and low energy neutrinos ($E_\nu \approx 1-10$ MeV.). The high energy neutrinos are produced somewhat indirectly in collisions of cosmic rays with the nuclei of oxygen and nitrogen in the earth's atmosphere. He described various experiments under operation and preparation in India and elsewhere in the world, to study high energy atmospheric neutrinos as well as the low energy ones of solar and extrasolar origin. He also summarized the astrophysical information, particularly on the interior of celestial bodies, that can be inferred from such studies.

'Symmetries and bootstraps' was the subject of a review talk by Dr Virendra Singh (TIFR). Investigations carried out in the bootstrap framework promise to provide answers to the dynamics behind symmetries. The concept of symmetries has helped in the classification of the known elementary particles. In the bootstrap philosophy one regards all strongly interacting elementary particles (hadrons) as composites made out of each other. The forces between hadrons are provided by exchanges of the hadronic systems themselves and involve as inputs the masses, coupling constants, etc. Imposing the self-consistency requirement, that the output and input values of all these parameters be equal, one could obtain the mass ratios and coupling constants of all the hadrons.

Dr Vainu Bappu, Director, Kodaikanal Observatory, gave a very interesting talk on 'Quasi-stellar sources'. He summarized the evidence which has led astronomers to believe that quasi-stellar radio sources are exceedingly bright and luminous galaxies located at cosmic distances from us. He also described quasi-stellar galaxies which have all the other properties of the quasi-stellar radio sources except that they do not emit radio waves. The Kodaikanal Observatory is shortly to embark on a photometric and spectroscopic programme on these objects with a view to getting information on the light variation and possible spectrum variations of the quasi-stellar sources and galaxies.

Prof. S. Biswas (TIFR) reviewed the recent discoveries made in the very low energy nucleonic component of the primary cosmic radiation, while Prof. R. R. Daniel focused attention on the newly discovered weak components of cosmic origin, namely X-rays, gamma rays and electrons.

Dr U. R. Rao [Physical Research Laboratory (PRL), Ahmedabad] in his review talk entitled 'Cosmic ray modulation and intensity variations' discussed the anisotropic variations like the solar diurnal variation and semi-diurnal variation. He also discussed a few space probe and satellite experiments that are currently under way to study the anisotropic changes of cosmic ray intensity.

Dr S. Naranan (TIFR) reviewed the present state of knowledge on 'Extensive air showers', while

Prof. Yash Pal, also of the same institute, reviewed our knowledge on the nature of high energy interactions. He discussed various theoretical models in vogue at present.

Dr P. B. Price, Visiting Professor at TIFR, from General Electric Research Laboratory in Schenectady, New York, USA, discussed how fossil tracks of very heavy cosmic ray nuclei left behind in meteorites can be made visible by chemical etching methods and how such tracks can provide information about the early histories of the solar system and the cosmic radiation. He presented very interesting results obtained by him on the Patwar meteorite which fell in East Bengal in 1935. Various applications of dielectric track detectors were also reviewed by him.

Dr Rama (TIFR) discussed the application of cosmic ray produced radio isotopes in fields like meteorology, geophysics, oceanography, etc., in a review talk entitled 'Isotopic changes induced by cosmic rays in terrestrial materials'.

Modulation and Solar Terrestrial Relations

A large number of papers in this session were presented by workers from the Physical Research Laboratory (PRL), Ahmedabad.

G. Subramanian (PRL) reviewed briefly the various models of interplanetary modulation proposed recently to explain the anisotropy in the intensity of cosmic rays which cause the solar diurnal variation.

In a couple of papers comparative studies were made of the characteristics of the daily variation of the nucleonic component of cosmic ray intensity for the year 1964, with similar analysis of IGY data. It was shown that, while various theories are in agreement with the average pattern of experimentally observed daily variation, they are not able to explain the large day to day fluctuations. Studies regarding the latitude dependence of the anisotropy of galactic cosmic ray intensity was the subject of another paper from PRL. The changes in the direction of maximum, the direction of minimum and the strengths of the anisotropy during the years 1958-63 and their significance in obtaining information about interplanetary space were brought out.

G. Subramanian and V. A. Sarabhai (PRL) proposed a model for the density distribution of cosmic ray particles in interplanetary space near the orbit of the earth. They showed how the model explains the observed direction of maximum of the semi-diurnal anisotropy, its energy spectrum and latitude dependence.

S. P. Tarafdar (TIFR) presented his model of the 'Interplanetary medium'. Starting with the assumption that the magnetic pressure in the interplanetary medium is much higher than the gas kinetic pressure, he worked out the magnetic field strength, the gas velocity, the particle density, etc. He claimed that the results obtained from his model are in agreement with the recent satellite observations of these quantities.

In two other papers from PRL, the authors discussed how the 27-day patterns of the coronal index of the spectral line 5303 Å. emitted by the sun, when studied in relation to the K_p index, the cosmic ray mean intensity and the daily variation, help us in understanding the electromagnetic

condition of interplanetary space. They find that the zonal character of solar activity is exhibited by coronal emission and, that for the period 1959-63, there is considerable difference in the level of activity in northern and southern hemispheres of the sun.

Primary Cosmic Radiation

The electron component of the primary radiation was discussed in two papers presented by the TIFR group. While the first paper dealt with some calculations made on the production of electrons in interstellar space, the second paper described an experiment from which the flux and energy spectrum of primary electrons of energy > 16 GeV. have been measured for the first time. The latter work was carried out by R. R. Daniel and S. A. Stephens.

Helium nuclei in the primary cosmic radiation were discussed in the next few papers presented. A collaboration experiment between the TIFR, the Delhi University and IIT, Delhi, is in progress to determine the energy spectrum of these nuclei in the energy region 6-20 GeV. nucleon. The experiment makes use of the variation of the geomagnetic cut-off energy for different arrival directions of the particles in the east-west plane. A determination of the energy spectrum of alpha particles below 600 MeV./nucleon at a time very near the solar minimum was the subject of another paper. The isotopic composition of the helium nuclei was discussed in a few papers.

A joint work of TIFR and the Panjab University on the composition and energy spectrum of heavy nuclei of the primary cosmic radiation was presented by N. Sreenivasan (TIFR) and V. S. Bhatia (Panjab University). This experiment was performed in a stack of emulsions flown over Fort Churchill, Manitoba, Canada. Results on very low energy cosmic ray heavy nuclei, detected by sending nuclear emulsions in an Aerobee rocket, were discussed by K. A. Neelakantan (TIFR). This work was done at NASA, Goddard Space Flight Centre, USA.

M. C. Durgapal (Jodhpur University) made an interesting suggestion in the form of a paper entitled 'Effect of gravitation on the origin of cosmic particles'. According to him, at low gravitational potential, when a star collapses, the whole star becomes unstable and gives out spontaneous emission of alpha particles and other nuclear particles. This work was done in collaboration with G. L. Gehlot.

A paper entitled 'Composition of the source of cosmic rays' was presented by S. Ramadurai and M. V. K. Appa Rao (TIFR). The last interesting paper in this session was the paper by N. W. Nerurkar (PRL) on the rigidity spectra of primary cosmic rays.

Astrophysics

This session was devoted mainly to radio and optical astronomy. Under radio astronomy, the technical features of a cylindrical telescope, to be set up at Ootacamund by TIFR, was described by G. Swarup. He mentioned that observations using the method of lunar occultation are going to be carried out when the telescope is ready. A multi-element grating interferometer radio telescope has been constructed at Kalyan, near Bombay, to study the radio emission of the sun. Preliminary results

from observations on the sun at 612 Mc/s. using this equipment were presented by the TIFR group.

K. D. Abhyankar (Osmania University, Hyderabad) described the programme of the 48 in. optical telescope to be set up at the Nizamiah Observatory. He said that, when the telescope is in operation, a part of its time will be open for use by other astronomical groups in this country. He also presented papers describing the research work on stars like ζ CrBA and ν -Eridani carried out by himself and his colleagues in the astronomy department of the Osmania University at Hyderabad. S. M. Alladin (Nizamiah Observatory) presented a theoretical paper titled 'Dynamics of colliding galaxies'.

A detector using electro-optic devices was described by J. C. Bhattacharya of Kodaikanal Observatory to measure weak magnetic and velocity fields on the solar surface.

P. J. Lavakare (TIFR) discussed the existing experimental evidence for the emission of solar neutrons and described an experiment undertaken by TIFR for detecting them.

Extensive Cosmic Ray Induced Air Showers, Muons and Neutrinos

An air shower array has been set up by the TIFR group, at Ootacamund, 2.2 km. above sea level. This array consists of: (i) 20 scintillation counters spread over an area of a circle of radius 40 m. to measure electron densities, (ii) 5 fast scintillators coupled to a 60-channel nanosecond chronotron timing system for determining arrival directions of the showers, (iii) an energy flow detector of area 4 sq. m. comprising liquid scintillators, (iv) N-(nuclear active) particle detectors at the centre of the array, and (v) a total absorption spectrometer of area approximately 1.5 sq. m. Results were presented on: (a) the energy spectrum of high energy N-particles > 50 GeV. in showers of size between 10^4 and 10^7 particles, (b) the properties of extensive air showers rich in mu-mesons, and (c) the time lags with respect to the shower front of nuclear interacting particles of various energies. From results obtained in (c) it is possible to conclude that the major part of nuclear active particles of energy > 5 GeV. cannot be pions; the results also strongly suggest the production of a large number of nucleon-antinucleon pairs in air showers. Recently, there has been some theoretical speculations on the production, in high energy collisions, of particles much heavier than nucleons. The TIFR group, from their experiments conducted with the Ootacamund air shower array, presented negative evidence for such particles, but with some reservation.

Another paper was presented by the TIFR group giving some preliminary experimental results on the absolute number of muons of energy > 220 GeV. and > 640 GeV. in showers of primary energy 10^{15} - 10^{16} eV. For this purpose, they used a large air shower array on the surface and muon detectors deep underground in the Kolar Gold Mines in Southern India. Some theoretical calculations pertaining to the above results were presented by Yash Pal and his colleagues. The experimental results on extensive air showers were obtained by B. V. Sreekantan, S. Naranan and their colleagues.

V. S. Narasimhan (TIFR) presented a paper describing a collaboration experiment between TIFR, Osaka University (Japan) and University of Durham (UK) to detect high energy neutrinos. This experiment is being conducted in the Kolar Gold Fields at a depth equivalent to 7500 m. of water. He presented definite evidence for interactions produced by such neutrinos in their detector system. The results obtained in this experiment are of great importance, since neutrinos being elusive particles with no mass, no charge and almost no interaction with matter are extremely difficult to detect experimentally.

Particle Physics

S. N. Biswas (Delhi University) gave a review talk on 'Symmetries'. Ideas regarding symmetries have been empirically quite successful in helping us to classify a large number of observed particles into a relatively small number of compact multiplets with interesting relations between their masses, coupling constants, etc. Isospin symmetry or SU_2 grouped particles of the same spin, parity, strangeness, and baryon number, but of different charge, into one multiplet. In SU_3 , the multiplets were enlarged; the members were allowed to have different strangeness and isospin too (subject to certain restrictions), but they had to have the same spin, parity and the same baryon number. The next step was SU_6 in which members of a multiplet could now have different spins too (again subject to certain regulations), but they must still have the same parity and the same baryon number. In SU_9 symmetry, members of a multiplet are allowed to have not only different charge, isospin, strangeness and spin (as in SU_6), but also different parity and baryon number.

A. N. Mitra (Delhi University) presented a paper entitled 'A non-relativistic quark model for baryons', according to which any two quarks — particles much heavier than nucleons — are assumed to interact in P-waves. Arguments favouring P-wave to S-wave forces were presented.

B. Dutta Roy [Saha Institute of Nuclear Physics (SINP), Calcutta] presented a paper on 'Positive parity mesonic states and unitary symmetry'. Evidence was presented for an octet of 0^+ (scalar) and a nonet of 1^+ (axial-vector) mesons, which together form a 35-plet $SU(6)$ supermultiplet. Pure $SU(6)$ was applied to the decays and satisfactory agreement with experiment was obtained.

P. Babu (TIFR) gave a talk on 'Current algebra'. Gell-Mann suggested that symmetries be considered in terms of the algebras generated by the weak and electromagnetic currents of strongly interacting particles. In dealing with approximate symmetries, current algebras provide a more powerful tool than description in terms of groups.

The newly discovered Π , K and hyperon resonances and Regge-pole mechanism were the subject of a few papers presented by the groups from Delhi University, SINP and TIFR. H. S. Mani [National Physical Laboratory (NPL), Delhi] presented a comprehensive and lucid review on the 'Recent theoretical speculations on T-violations'.

Hyperfragments or hyper-nuclei are those in which a lambda-hyperon is bound to a nuclear fragment.

Papers on hyperfragments were presented by nuclear emulsion groups from Delhi, Kurukshetra, Gauhati and TIFR. B. Bhowmik (Delhi University) reviewed the present status on the hyperfragment work. Most of the papers dealt with hyperfragments produced in interactions of high energy beam particles with emulsion nuclei. 'The mass and lambda binding energies of short-range hyperfragments decaying mesonically and non-mesonically' was the main subject of discussion.

High Energy Interactions

About six papers presented by delegates from Panjab University and one from the Osmania University dealt with interactions caused by 17 GeV pions with emulsion nuclei. Characteristics of these interactions such as transverse momentum distribution for secondary pions and protons, coherent production of pions due to Coulomb and diffraction dissociations and detailed analyses of these interactions on the basis of isobar models, were discussed. Three papers, one from the Osmania University, one from Delhi University and one from IIT, Delhi, dealt with the characteristics of interactions produced by 24 GeV. protons with emulsion nuclei. The analyses presented were similar to those on 17 GeV. pions.

J. Moyer (IIT, Kanpur) presented a paper on 'Pion-proton elastic and charge exchange scattering at 500-1000 MeV.', while T. O. Binford (visiting scientist at TIFR from the University of Wisconsin, USA) presented the cross-sections and the angular distributions for the reactions $\pi^- P \rightarrow \Sigma^0 K^0$ and $\pi^- P \rightarrow \Lambda^0 K^0$ at incident pion momenta of 1128, 1235, 1277 and 1326 MeV./c. The latter work was carried out at the University of Wisconsin.

A few papers on techniques were included in this section of which mention may be made of the paper by M. S. Sinha (Regional Engineering College, Durgapur) on the characteristics of a double magnet cosmic ray spectrograph to be built at Durgapur. He mentioned that investigations on a model magnet show that steel produced at Rourkela is more suitable than imported steel for construction of the magnet.

Cosmic Ray History

P. S. Goel (IIT, Kanpur) gave a review talk on 'Cosmic ray records in meteorites'. Since meteorites are subject to cosmic ray bombardment while in outer space, they are excellent space probes for studying cosmic ray intensity variations both in space and in time. Radioactive dating methods show that meteorites were formed some 4.5×10^9 years ago. Studies of radio nucleides in meteorites have led to the conclusion that the cosmic ray intensity averaged over the last 50,000 years is the same, within better than 10 per cent, of the averaged intensity over the last 500 years.

A paper entitled 'Solar proton interactions in the interplanetary dust' was presented by D. Lal and V. S. Venkatavaran (TIFR). Radioactivity of ^{26}Al measured by them in sediments raised from the Pacific Ocean is larger by a factor of 10 than that expected due to production by cosmic ray interactions in the atmosphere. Assuming that this increase is due to low energy solar protons interacting in interplanetary dust, the solar proton intensity in the

past 10^6 years has been deduced to be of the order of $100/\text{cm}^2$ sec. for particles of energy > 10 MeV.

Concluding Remarks

Some concluding remarks were made by Prof. M. G. K. Menon, now Director, Tata Institute of Fundamental Research. He mentioned that many of the papers read, at this symposium, were as good as any that are read in international conferences he has attended; this was a good feature of this symposium. On the other hand, there were some

papers which were not in keeping with the advances made in the field. This, he said, is sometimes due to the fact that in research one often tends to become technique-oriented or problem-oriented to such an extent that stagnation sets in. He felt that it is as important to know when to stop a research programme as to know when to start it. He mentioned problems like X-ray astronomy, infrared spectroscopy of the sun and experiments using rockets in which groups from universities can take part with modest equipment.

Synthesis of Azasteroids Using Beckmann Rearrangement & Schmidt Reactions

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THE field of steroids has provided a host of interesting problems of isolation of the steroid entities from natural sources, their structure elucidation, biochemical studies, preparation of many analogues of potential medicinal interest, and search of suitable methods for industrial production of steroid drugs. During this unending process many problems of fundamental organic chemistry involving reaction mechanistic and stereochemical aspects have been successfully solved and many new compounds of diverse pharmacological activity and therapeutic interest have been discovered.

Azasteroids, broadly speaking nitrogen containing steroids, are receiving concerted attention of chemists and pharmacologists alike. Alauddin and Martin-Smith^{1,2} and Martin-Smith and Sugrue³ have reviewed biological activity in steroids possessing nitrogen atoms, both of natural and synthetic origins. Out of the numerous synthetic azasteroids prepared many have shown one kind or the other of biological activity, and some are of clinical significance. In many cases synthesis of azasteroids may have been conducted on empirical grounds, but in certain instances research in the preparation of new azasteroids has been motivated by sound reasoning based on theoretical concepts of drug action. There are known azasteroids which have anabolic, antihormonal, antihypercholesterolaemic, vasodilatory, anticancer, neuromuscular-blocking, CNS depressant, or antimicrobial activity.

Insertion of nitrogen atom into steroid nucleus has been effected mostly by reactions with steroid ketones; like the Beckmann rearrangement of oximes and Schmidt reaction, and by suitable

reactions with the respective *seco*-keto acids and oxasteroids. The other methods of preparation include imide synthesis, Curtius and Hoffmann rearrangements, total synthesis, etc. The lactams and imides can be reduced to the respective basic azasteroids which are used to prepare various related analogues. The literature on insertion of hetero atoms into the steroid nucleus was surveyed by Tökés⁴, and his compilation is a useful compendium of the representative references on the topic.

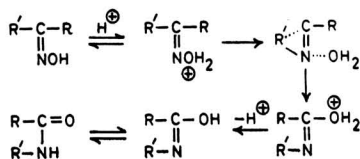
Recently, there has been reported the development of photochemical reactions applicable to nitrogenous steroids⁵⁻¹⁰. The microbiological transformation of azasteroids¹¹⁻¹⁵ and discovery of microbiological amidation of steroids¹⁶ are routes of interest for obtaining various azasteroid analogues.

There is a good amount of literature on synthetic nitrogenous steroids, and out of the preparative methods we shall discuss here in detail only the use of Beckmann rearrangement and Schmidt reactions in the synthesis of azasteroids; the application of these reactions to steroids where the resulting products are not necessarily nitrogenous are also discussed.

Beckmann Rearrangement and Schmidt Reactions

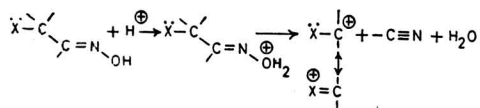
The Beckmann rearrangement¹⁷ involves the transformation of ketoximes to amides, or say of cyclic ketones to lactams. The reaction is catalysed by acids including Lewis acids. In the reaction, there is involved partial ionization of the oxygen-nitrogen bond of the oxime giving an electron deficient nitrogen atom and that results in the

simultaneous intramolecular migration of the group *anti* to the departing hydroxyl group:

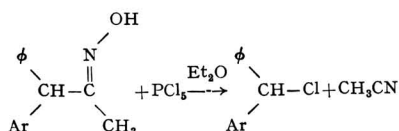


When the migrating carbon atom is asymmetric there is retention of configuration about the migrating carbon, possibly on account of the breakage of the C-C bond and the formation of the new C-N bond taking place on the same side of the asymmetric carbon.

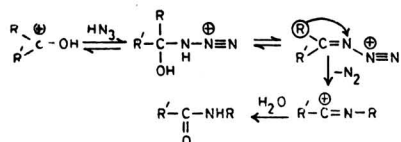
Apart from normal Beckmann rearrangement to amides, there are many cases of oximes undergoing cleavage to nitriles and some other fragment, and this type of reaction is known as 'second-order' Beckmann rearrangement. It has been observed for α -hydroxy oximes, α -keto oximes, α -oximino acid, α -imino oximes, α -amino oximes, α,α -diaryl oximes, β -thioether oximes, bridged α,α -dialkyl oximes, α -trisubstituted oximes and α -ether oximes¹⁸. It is stated that in all these cases the α -substituents can stabilize the carbonium ion or the incipient positive charge formed during the cleavage, which could be represented as shown below:



A direct displacement path has, however, been suggested for the cleavage of α -keto oximes. In an extremely facile cleavage of 1,1-diaryl-2-propanone oxime under Beckmann rearrangement conditions (PCl_5 -ether) to alkyl chloride and methyl cyanide, Hassner and Nash¹⁹ report a case involving exclusively cleavage with displacement.

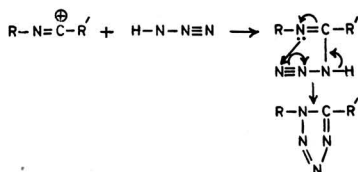


The Schmidt rearrangement^{20,21} is in fact a group of reactions involving the use of hydrazoic acid (HN_3) in sulphuric acid—polyphosphoric acid has also been used. Carboxylic acids are converted into amines (having one carbon atom less), the reaction going through the formation of an insoluble isocyanate. The ketones change to amides, under the reaction conditions, possibly through the following mechanism:



In Schmidt reaction also as in the case with Beckmann rearrangement, the migrating group shifts with retention of configuration. It is generally observed that the bulkier of the two groups in the parent ketone migrates preferentially.

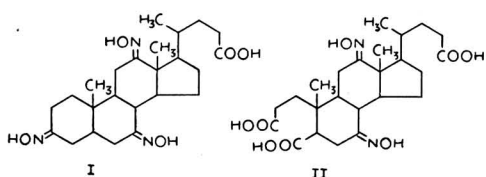
When an excess of HN_3 is used a substituted tetrazole may be formed, possibly by the action of a second molecule of hydrazoic acid on the carbonium ion:



After this brief introduction to Beckmann rearrangement and Schmidt reaction we consider application of the reactions to the synthesis of azasteroids and in so doing we shall also deal with certain theoretical aspects regarding the reactions as observed during the work on steroids. For convenience sake we discuss the relevant literature under the heads: Reactions with simple ketones, Reactions with α,β -unsaturated ketones, Rearrangement of α -oximino ketones, Rearrangement of α -hydroxy ketones, and Other rearrangements.

Reactions with Simple Ketones

The earliest reports of Beckmann rearrangement of the steroid oximes have been by Schenck and coworkers²². He carried out the rearrangement under classical rearrangement conditions (15-20 min. heating at 95° with 90 per cent sulphuric acid) to prepare lactams from the ring ketoximes derived from bile acids, viz. compounds (I) and (II); the structures of the products were, however, not rigidly established.

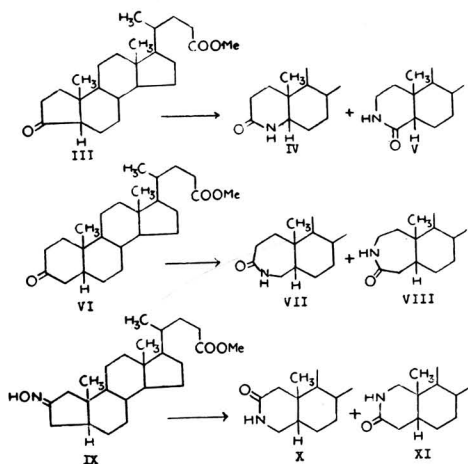


Introduction of N in Ring A

Hara²³ has carried out synthesis of azasteroids in cholane series through Beckmann rearrangement of the respective oximes. Thus, the oxime of methyl 3-oxo-A-norcholanate (III) on heating with tosyl chloride (TsCl) for 1 hr at 50°, followed by chromatographic resolution, yielded mostly the lactam (IV) and a minor quantity of the lactam (V). When the same reaction was done with PCl_5 as catalyst in ether-benzene solution at 0° (letting stand for 2 hr), only the lactam (IV) was isolated. Similar type of reactions of the oxime of methyl 3-oxocholanate (VI) using TsCl and PCl_5 as catalysts yielded the lactams (VII) and (VIII), with the latter in traces only. On doing the Schmidt reaction in

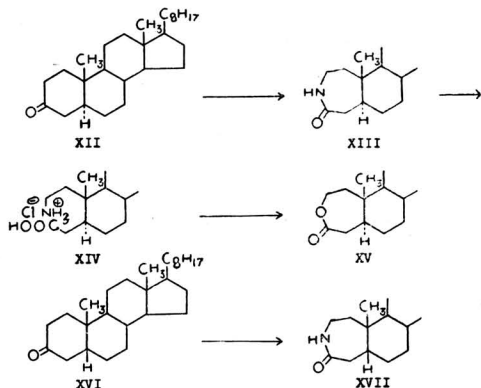
chloroform with sulphuric acid, from the keto ester (III) only (IV) was obtained in 15 per cent yield, whereas (VI) gave (VII) and (VIII) respectively in 40 and 15 per cent yields. The structure of 4-aza lactam (IV) was established by Hara²⁴ by first reducing the lactam with LiAlH_4 , and dehydrogenation of the product by heating with powdered Se, which resulted in isolation of 1-azachrysenes as one of the products. The structure of 4-aza-A-homo lactam (VII) was proved by acid hydrolysis, Hofmann degradation, and ozonolysis to the known 5-oxo-3,4-*seco*-A-norcholanic acid. The structures of 3-azasteroids (V) and (VIII) followed by exclusion.

Hara²⁵ also conducted the rearrangement of methyl 2-oxo-A-norcholanoate oxime (IX), using TsCl and PCl_5 . Two lactams, (X) and (XI), the former in comparatively larger quantity, were isolated.



In cholestane series, aza-A-homo steroids were first reported from Shoppee school at the University of Sydney. Shoppee and Sly²⁶ prepared lactams (XIII) and (XVII) by rearrangement in dioxane with SOCl_2 at 40° for 15 min. of the oximes of 5α -cholestan-3-one (XII) and 5β -cholestan-3-one (XVI) in 63 and 36 per cent yields respectively. The structures (XIII) and (XVII) were determined by a similar sequence of reactions. Thus, the lactam (XIII) on hydrolysis with a mixture of hydrochloric and acetic acids furnished the amino acid hydrochloride (XIV), deaminated by nitrous acid to the analogous hydroxy acid, which immediately formed the known lactone (XV).

Following the report of Conley²⁷ that polyphosphoric acid is superior to other catalysts in the Beckmann and Schmidt rearrangements, Doorenbos and Wu²⁸ conducted the reactions with the oximes of the cholestanones. The Beckmann rearrangement of the oximes of (XII) and (XVI) in polyphosphoric acid at $120-30^\circ$ for 30 min., or Schmidt reaction of the ketones with sodium azide in polyphosphoric acid at $50-60^\circ$ for 10 hr, yielded

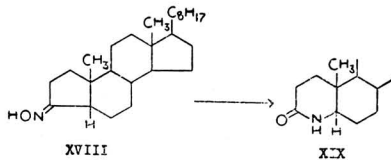


the respective lactams (XIII) and (XVII), the yield in each case being at least 86 per cent.

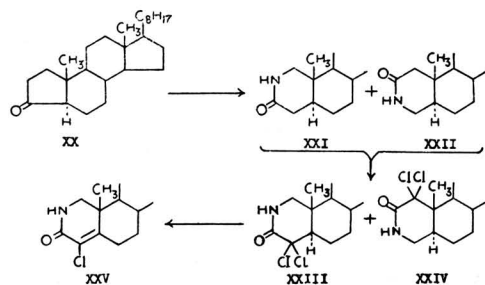
A mention may be made here of the method of Craig and Naik²⁹ for Beckmann rearrangement which proceeds by the preparation of the benzene (or *p*-toluene)-sulphonate ester of the respective ketoxime followed by rearrangement on a column of alumina. Thus, 5α -cholestan-3-one oxime gave, via the *p*-toluene-sulphonate ester, 85 per cent of 3-aza-A-homo- 5α -cholestan-4-one (XIII); m.p. $271-3^\circ$.

The homogeneity of the lactams (XIII) and (XVII), the structures of which were rigorously established earlier²⁶, is now doubted by Shoppee as discussed later in this article.

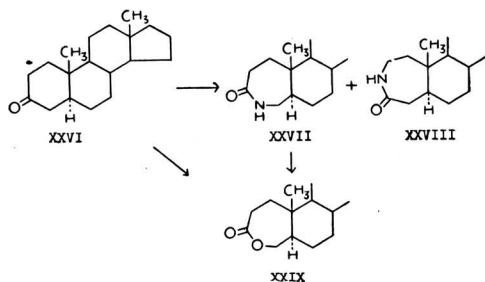
In continuation with the discussion on the reactions with cholestanones it may be mentioned that Shoppee *et al.*³⁰ and also Edward and Morand³¹ submitted the oxime of A-nor- 5β -cholestan-3-one (XVIII) and obtained a single lactam 4-aza- 5β -cholestan-3-one (XIX), the 5β -configuration of which has been ascertained.



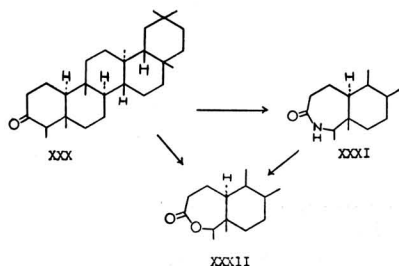
Further, Doorenbos and Havranek³² on conducting the reaction of A-nor- 5α -cholestan-2-one (XX) with hydrazoic acid in polyphosphoric acid obtained an inseparable mixture of the lactams (XXI) and (XXII), in 77 per cent yield. By doing the Beckmann rearrangement of oxime of (XX) in polyphosphoric acid the same mixture resulted, but in lower yield (30 per cent). The mixture of lactams thus formed when treated with phosphorus pentachloride³³ yielded a mixture of dichloro lactams, in 1:1 ratio, which could be separated into (XXIII) and (XXIV). The formation of (XXV) by treating (XXIII) with collidine, and inability of HCl elimination from (XXIV) under the same conditions, indicated the structures of the lactams (XXI) and (XXII) respectively.



Shoppee extended his work on azasteroids to the preparation of 3-aza and 4-aza-A-homoandrostanes³⁴. The oxime of 5 α -androstan-3-one (**XXVI**) underwent Beckmann rearrangement (SOCl_2 , -20° , 3 min.; KOH , 20°) nearly quantitatively; careful chromatography followed by extensive fractional crystallization of the product gave 4-aza-A-homo-5 α -androstan-3-one (**XXVII**, m.p. 303° , and the molecular compound (**XXVII** and **XXVIII**); m.p. $262-4^\circ$. Similar reactions were carried out in 5 β -androstanone series. Compounds (**XXVI**) and (**XXVII**) were converted to lactone (**XXIX**) by standard procedures.

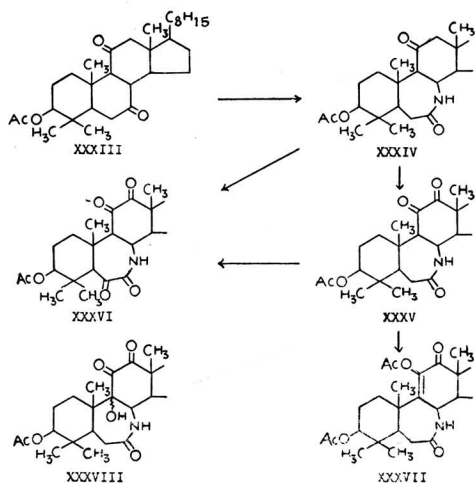


It may not be out of place to mention that attention has also been given to Beckmann rearrangement of 3-ketoximes^{35,36} obtained from triterpenoids (β -amyrin, allobetulin) of the familiar dimethylcyclohexanol ring A structure. Stevenson³⁷ has prepared 4-aza-A-homofriedelan-3-one (**XXXI**) by Schmidt reaction of friedelin (**XXX**) and Beckmann rearrangement of friedelin oxime. The lactam (**XXXI**) has been converted to known 4-oxa-A-homofriedelan-3-one (friedelolactone) (**XXXII**) by treatment with dinitrogen tetroxide in carbon tetrachloride at low temperature.

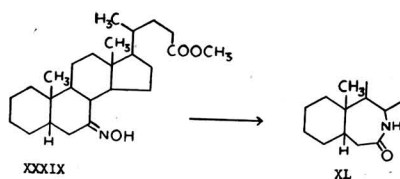


Introduction of N in Ring B

Coming to the discussion on B-aza steroids first could be mentioned the work of Barton and co-workers³⁸ who during their research on the constitution of lanostadienol obtained (**XXXIV**) by Schmidt reaction with (**XXXIII**) or Beckmann rearrangement of its monoxime. The structure of (**XXXIV**) was formulated on the basis of theoretical considerations, selenium dioxide conversions (**XXXIV** \rightarrow **XXXV**, **XXXV** \rightarrow **XXXVI**, and **XXXIV** \rightarrow **XXXVI**), and characterization of the diosphenol acetate (**XXXVII**) (λ_{max} . 242 $\text{m}\mu$; ϵ 9000). Ruzicka and co-workers³⁹ prepared the aza analogue (**XXXIV**), but in contrast to the report of Barton and co-workers³⁸ they report the conversion of (**XXXIV**) to (**XXXVIII**) with selenium dioxide and not the lactam (**XXXVI**), and found that on further oxidation ring C is ruptured.

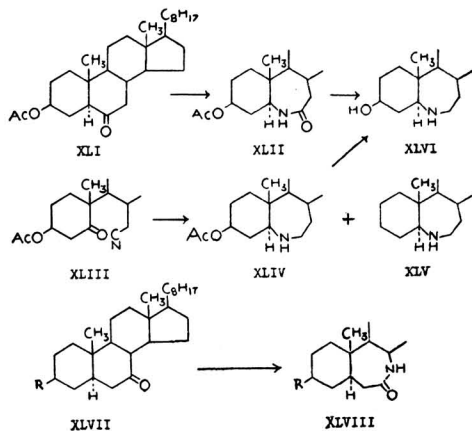


Hara⁴⁰ conducted the synthesis of methyl 7-oxo-7a-aza-B-homocholatanate (**XL**) and methyl 6,7a-dioxo-7-aza-B-homocholatanate, the former being prepared by rearrangement of methyl 7-oximinocholatanate (**XXXIX**) by treatment with TsCl in pyridine at 100° for 1 hr.



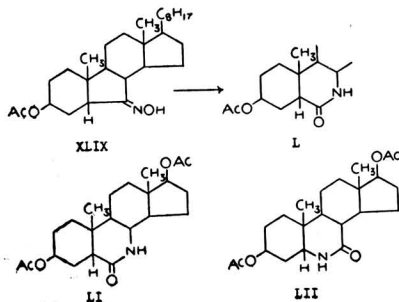
Doorenbos and Singh⁴¹ prepared 3 β -acetoxy-6-aza-B-homo-5 α -cholestan-7-one (**XLII**) and 3 β -acetoxy-7a-aza-B-homo-5 α -cholestan-7-one (**XLVIII**; $\text{R}=\text{AcO}$) by Schmidt reaction in polyphosphoric acid of the ketones (**XLI**) and (**XLVII**; $\text{R}=\text{AcO}$) respectively. Knof⁴² conducted the Beckmann rearrangement of the respective ketoxime in benzene

solution with SOCl_2 , and obtained the azasteroids (XLII) and (XLVIII). Knof reports the preparation of 3 β -acetoxy-5,6-*seco*-cholestan-5-on-6-oic acid nitrile (XLIII) which on hydrogenation was found to give a mixture of 3 β -acetoxy-6-aza-B-homocholestan-3 β -ol (XLIV) and 6-aza-B-homocholestan-3 β -ol (XLV). On saponification (XLV) gave 3 β -hydroxy-6-aza-B-homocholestan-3 β -ol (XLVI), which could also be obtained by lithium aluminium hydride reduction of (XLII).



Shopee *et al.*⁴³ have recently prepared 7a-aza-B-homo-5 α -cholestan-7-one (XLVIII; R=H) from the oxime of 5 α -cholestan-7-one (XLVII; R=H) by treatment with SOCl_2 at -10° (added to 4*N* KOH at 90°). The structure 7-oxo-7a-aza is based on the interpretation of the NMR spectrum, which shows (i) a complex signal at τ 4.5 for the proton in the NH-group, readily exchangeable for deuterium, and (ii) a multiplet at τ 6.76 of area corresponding to the single 8 β -proton adjacent to the nitrogen atom. It is argued that 7a-oxo-7-aza structure would require the presence of a multiplet of area corresponding to the two 6 α - and 6 β -protons.

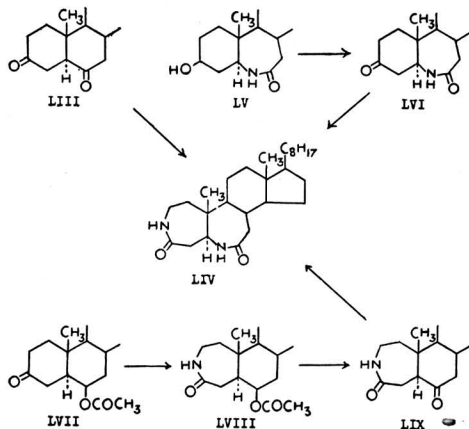
Another case of the introduction of N in ring B was reported by Morisawa *et al.*⁴⁴ of the Sankyo Co., Tokyo, who prepared 3 β -acetoxy-7-aza-5 β -cholestan-6-one (L) by Beckmann rearrangement of 3 β -acetoxy-B-nor-5 β -cholestan-6-one oxime (XLIX). The same workers⁴⁵ prepared 3 β ,17 β -diacetoxy-7-aza-5 β -androstan-6-one (LI) and 3 β ,17 β -diacetoxy-6-aza-5 β -androstan-7-one (LII) by Beckmann rearrangement of 3 β ,17 β -diacetoxy-B-nor-5 β -androstan-6-one oxime (XLIX).



toxy-6-aza-5 β -androstan-7-one (LII) by Beckmann rearrangement of 3 β ,17 β -diacetoxy-B-nor-5 β -androstan-6-one oxime. The structures of (L) and (LI) were confirmed by converting them into 3 β -acetoxy-7-oxa-5 β -cholestan-6-one and 3 β ,17 β -diacetoxy-7-oxa-5 β -androstan-6-one respectively by treating with N_2O_4 in CCl_4 followed by thermal decomposition of the N-nitroso derivative in boiling heptane. Morisawa⁴⁶ reports that oximation of 3 β -acetoxy-B-nor-5 α -cholestan-6-one gives *anti*- and *syn*-oximes, which can be separated by chromatography on alumina, and these on Beckmann rearrangement followed by LiAlH_4 reduction give 6-aza-5 α -cholestan-3 β -ol and 7-aza-5 α -cholestan-3 β -ol. Likewise, he prepared *anti*- and *syn*-oximes of 3 β ,17 β -diacetoxy-B-nor-5 α -androstan-6-one which were converted into 6-aza-5 α -androstan-3,17-diol and 7-aza-5 α -androstan-3,17-diol.

Introduction of N in Rings A and B

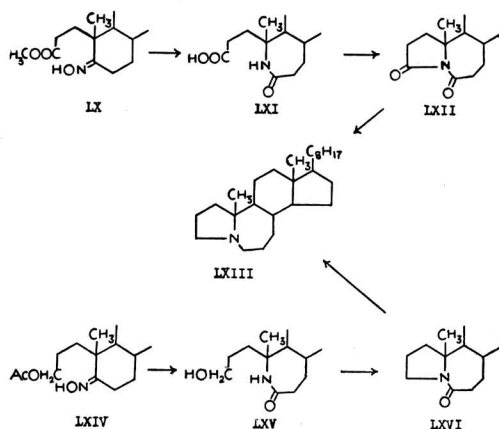
In each of the cases cited above nitrogen has been introduced in either of the rings A or B. Doorenbos and Singh^{47,48} prepared a steroid involving introduction of nitrogen in both rings A and B. 5 α -Cholestan-3,6-dione (LIII) yielded on Schmidt reaction in polyphosphoric acid a diazasteroid which was tentatively assigned the structure of 3,6-diaza-A,B-bishomo-5 α -cholestan-4,7-dione (LIV). The latter was also obtained from 6-aza-B-homo-5 α -cholestan-3 β -ol-7-one (LV)⁴¹, which was oxidized to the ketone, 6-aza-B-homo-5 α -cholestan-3,7-dione (LVI) by chromic acid and then converted to the diazasteroid (LIV) by means of Schmidt reaction. The third route was from 6 β -acetoxy-5 α -cholestan-3-one (LVII), which was converted to (LVIII), hydrolysed, and oxidized to 3-aza-A-homo-5 α -cholestan-4,6-dione (LIX) and the latter was subjected to Schmidt reaction to yield the diazasteroid (LIV). The assignment of structures in this work has been on provisional basis, and no rigorous proof of structure is available as yet.



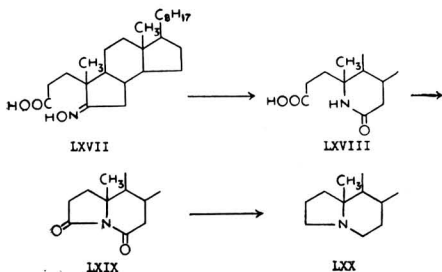
Introduction of N at Position 5

An interesting example of the use of Beckmann rearrangement for the preparation of

different analogues of the steroid skeleton is provided by Rodewald and Wicha⁴⁹ who prepared A-nor-B-homo-5-azacholestane (LXIII), in which N is at the junction of rings A and B. The oxime (LX) on Beckmann rearrangement with SOCl_2 at -20° and hydrolysis of the mixture with 4*N* KOH gave 3,5-*seco*-4-nor-B-homo-5-azacholestan-5a-on-3-oic acid (LXI), which on ring closure with BF_3 in ether or P_2O_5 in toluene gave A-nor-B-homo-5-azacholestan-3,5a-dione (LXII) in 20 and 70 per cent yields respectively. In an alternative route the oxime (LXIV) on Beckmann rearrangement and hydrolysis (to remove Ac) gave 3,5-*seco*-4-nor-B-homo-5-azacholestan-3-ol-5a-one (LXV) which could be cyclized with POCl_3 to yield A-nor-B-homo-5-azacholestan-5a-one (LXVI). Compounds (LXII) and (LXVI) on reduction with LiAlH_4 in dioxane gave the base (LXIII).

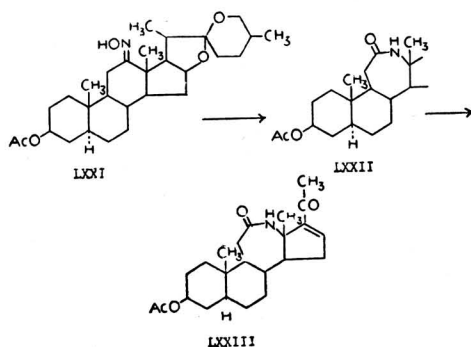


Rodewald and Wicha⁵⁰ now report the synthesis of A-nor-5-azacholestane (LXX). They prepared 3,5-*seco*-4-nor-B-nor-5-oximinocholestan-3-oic acid (LXVII) which on Beckmann rearrangement afforded 3,5-*seco*-4-nor-5-azacholestan-6-on-3-oic acid (LXVIII). The acid (LXVIII) on treatment with P_2O_5 gave 90 per cent of A-nor-5-azacholestan-3,6-dione (LXIX) which was reduced with LiAlH_4 in dioxane to (LXX).

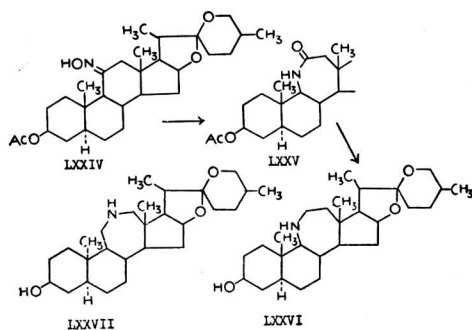


Introduction of N in Ring C

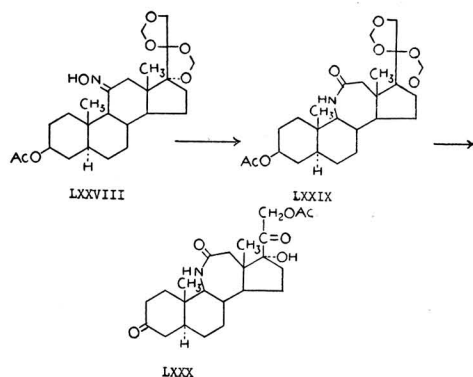
Mazur⁵¹ of the G. D. Searle & Co. carried out the Beckmann rearrangement of hecogenin acetate oxime (LXXI) using TsCl in pyridine at $95-105^\circ$ for 3 hr, and obtained the azasteroid (LXXII). An evidence in favour of structure (LXXII) will be discussed later. Mazur⁵² has degraded (LXXII) to 3 β -acetoxy-12a-aza-C-homo-5 α -pregn-16-ene-12,20-dione (LXXIII) and the latter has been utilized to prepare various analogues.



Zderic and Iriarte⁵³ prepared 11-ketotigogenin acetate oxime (LXXIV), which on treatment with phosphorus oxychloride in pyridine rearranged to yield the lactam 9a-aza-C-homotigogenin-11-one 3-acetate (LXXV). The location of the nitrogen was evident from the observation that (LXXVI), LiAlH_4 reduction product of (LXXV) and authentic 11a-aza-C-homotigogenin⁵⁴ (LXXVII) were dissimilar.



Zderic and Iriarte⁵³ also prepared 9a-aza-C-homo-5 α -pregnane-3 β ,17 α ,21-triol-11,20-dione-BMD 3-acetate (LXXIX) by Beckmann rearrangement of 5 α -pregnane-3 β ,17 α ,21-triol-11,20-dione-BMD 3-acetate 11-oxime (LXXVIII) in phosphorus oxychloride-pyridine system. The acetate (LXXIX) was converted to 9a-aza-C-homo-5 α -pregnane-17 α ,21-diol-3,11,20-trione 21-acetate (LXXX) by standard methods. Attempts made to introduce a 1,4-diene system in (LXXX) were unsuccessful.



Introduction of N in Ring D

Taking a clue from interesting physiological properties of some D-ring lactones, Kaufmann⁵⁵ prepared some six-membered D-ring lactams. He carried out Beckmann rearrangement of dehydroisoandrosterone acetate oxime, 4-androstene-3,17-dione 17-oxime, and estrone benzoate oxime, with *p*-acetamidobenzenesulphonyl chloride in pyridine solution, getting dehydroisoandrololactam acetate (LXXXI; X=AcO), testolactam (LXXXII), and estrolactam benzoate (LXXXIII; R=C₆H₅CO) respectively in 50 per cent yields. The 17-aza-D-homosteroid structures for these compounds were excluded, as dehydroisoandrololactam (LXXXI; X=OH) on selenium dehydrogenation at 350° gave 1-azachrysen (LXXXIV).

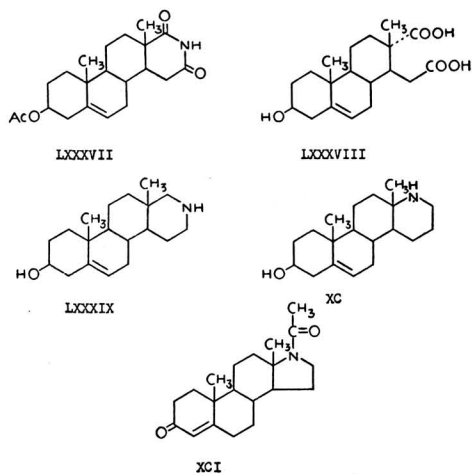
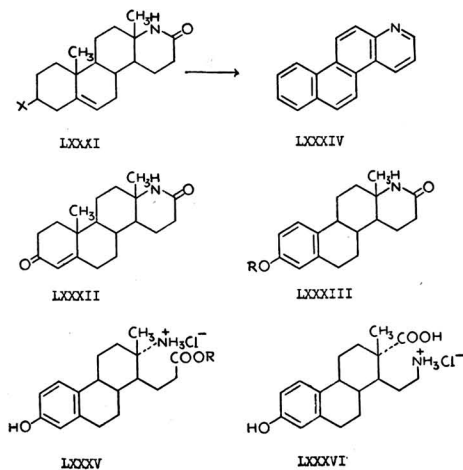
Later, Regan and Hayes⁵⁶ found that rearrangements of 17-ketosteroid oximes with thionyl chloride in dioxane gave in general slightly higher yields of the desired lactams than Kaufmann's *p*-acetamidobenzenesulphonyl chloride-pyridine method. In the case of estrone oxime, in which the latter method was reported to yield only intractable tars, Regan's procedure gave the lactam, 17a-aza-D-homoestrone (LXXXIII; R=H), in 90 per cent yield. Further, Regan and Hayes corroborated

the structure proofs in favour of 17a-aza-D-homosteroids being products of 17-one oximes rather than 17-aza-D-homosteroids, by hydrolysing (LXXXIII; R=H) with hydrochloric acid in boiling acetic acid to 3-hydroxy-13 α -amino-13,17-*seco*-1,3,5(10)-estratrien-17-oic acid hydrochloride (LXXXV; R=H), which being the primary carboxylic acid could be readily esterified with 0.9M HCl in methanol within 3 hr at 65°. Whereas, if 17-azalactam were the precursor the acid (LXXXVI) formed being tertiary carboxylic acid would not readily esterify with methanol and an acid catalyst. Regan and Hayes also rearranged 3 β -acetoxy-5-androstene-16,17-dione 16-oxime with excess of SOCl₂ to obtain 3 β -acetoxy-16,17-*seco*-5-androstene-16,17-imide (LXXXVII), in 65 per cent yield, the structure of which was established by its alkaline hydrolysis to known dicarboxylic acid (LXXXVIII). The LiAlH₄ reduction of the imide (LXXXVII) gave 17-aza-D-homo-5-androsten-3 β -ol (LXXXIX). This amine was found to be different when compared with 17a-aza-D-homo-5-androsten-3 β -ol (XC) obtained by similar reduction of (LXXXI; X=AcO). This provided a further proof for 17a-aza-D-homosteroids being the products of the rearrangement of 17-ketosteroid oximes.

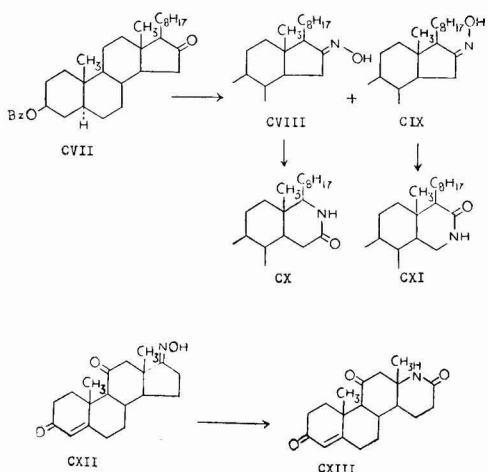
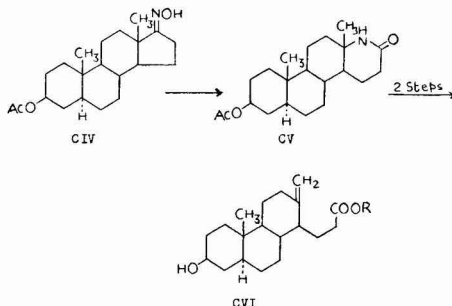
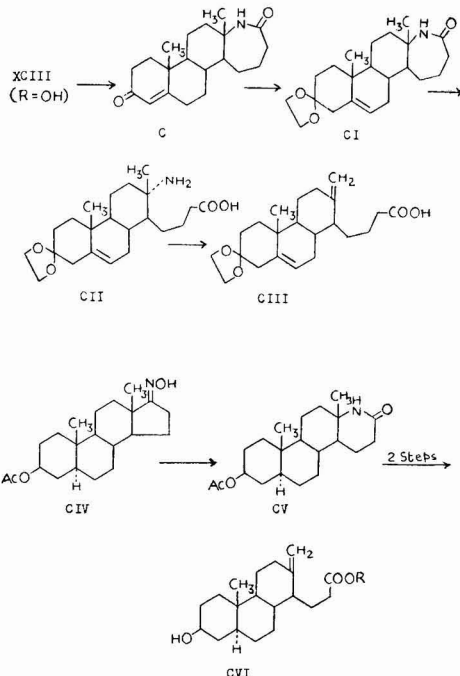
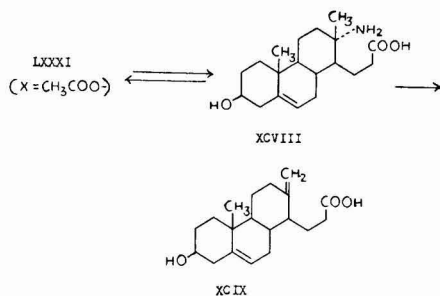
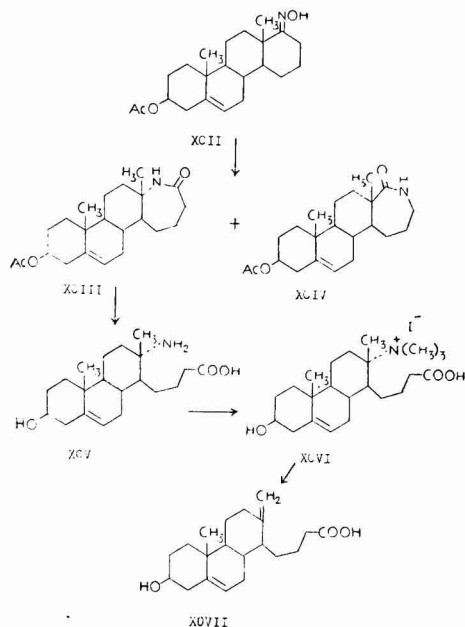
In search for serofluculants in androstane series, Blickenstaff and Foster⁵⁷ prepared 3 β -chloro-17a-aza-D-homo-5-androsten-17-one (LXXXI; X=Cl) by Beckmann rearrangement of 3 β -chloro-5-androsten-17-one oxime. The alternative method^{58,59} preparing the same was chlorination of (LXXXI; X=OH) obtained from the acetate (LXXXI; X=AcO)⁵⁶. All the three aza-D-homo compounds, however, were found to be too insoluble to be tested for serofluculation activity.

Recently, Rakhit and Gütt^{58,59} have made use of the D-ring lactam (LXXXI; R=Ac) to synthesize an interesting progesterone analogue 17-azaprogesterone (XCI).

In continuation of our discussion on D-aza steroids, we may now mention that in connection with the elucidation of the structure of aldosterone, Heusser *et al.*⁶⁰ while attempting to introduce O function



at the angular Me group at C-18, prepared a new group of degradation products involving in the first stage D-ring lactam through Beckmann rearrangement of the respective oximes using *p*-acetamidobenzenesulphonyl chloride-pyridine system. Thus, the rearrangement of 3 β -acetoxy-17 α -oxo-D-homo-5-androstene oxime (XCII) yielded a mixture of the lactams (XCIII) and (XCIV) out of which the former could be saponified to the acid (XCV) with KOH in butanol, while compound (XCIV) was resistant. The quaternary salt (XCVI) of the acid (XCV) yielded on Hofmann degradation 3 β -hydroxy-13,17-*seco*-5,13(18)-etiadienic acid (XCVII).

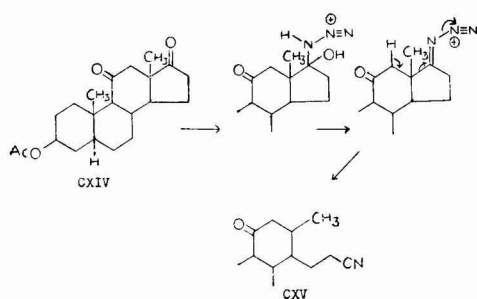


Heusser and coworkers⁶¹ also carried out the lactam preparations and degradations as exhibited by formulae (XCVIII) to (CVI). Again, while conducting the synthesis of 18-norprogesterone and 13 α ,17 α -norprogesterone, Heusser and coworkers⁶² prepared compound (LXXXI) and converted it into 3,3-ethylenedioxy-13,17-*seco*-5,13(18)-androstadien-17-oic acid of the type (CIII).

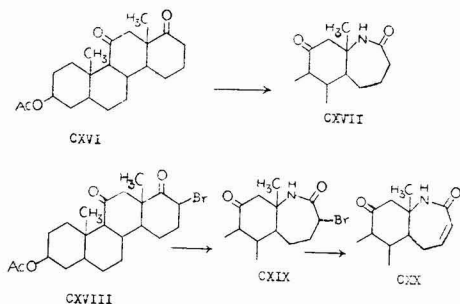
Tsuda and Hayatsu⁶³ obtained from 16-keto-cholestan-3 β -ol benzoate (CVII) the α -oxime (CVIII) and β -oxime (CIX) in 7:1 ratio, the Beckmann rearrangement of which with TsCl-pyridine yielded the lactams (CX) and (CXI) respectively, the latter being in poor yield.

Nagata⁶⁴, using rearrangement system similar to that of Craig and Naik²⁹, treated 17-hydroximino-4-androstene-3,11-dione (CXII) in pyridine with *p*-acetamidobenzenesulphonyl chloride and chromatographed the product on alumina and thus obtained 17 α -aza-D-homo-4-androstene-3,11,17-trione (CXIII) and 3,11-dioxo-13,17-*seco*-5,12-androstadiene-17-nitrile.

While exploring an approach to the partial synthesis of aldosterone at one of the steps, Barton



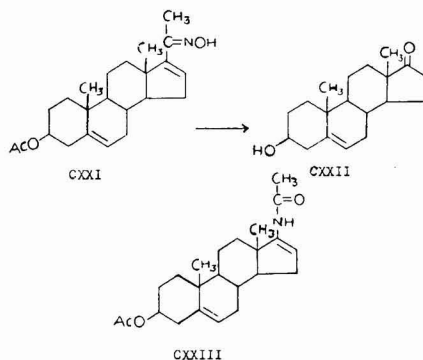
*et al.*⁶⁵ tried Schmidt reaction with 3β-acetoxyandrostane-11,17-dione (CXIV), but this afforded mainly the unsaturated nitrile (CXV), presumably by the mechanism indicated. However, in contrast to it, 3β-acetoxy-D-homoandrostane-11,17a-dione (CXVI) gave on Schmidt reaction (CHCl₃, H₂SO₄, NaN₃, 0-5°, 30 min.) the desired lactam 3β-acetoxy-17b-aza-D-bishomoandrostane-11,17a-dione (CXVII). They also reported the formation of 3β-acetoxy-17α-bromo-17b-aza-D-bishomoandrostane-11,17a-dione (CXIX) from 3β-acetoxy-17α-bromo-D-homoandrostane-11,17a-dione (CXVIII). The bromo lactam (CXIX) on refluxing with collidine and working up gave 3β-acetoxy-17b-aza-D-bishomo-16-androstene-11,17a-dione (CXX).



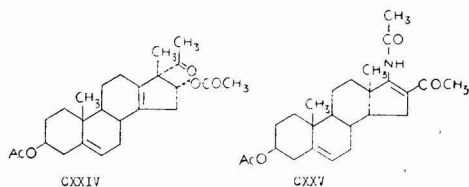
Suvorov *et al.*⁶⁶ obtained 16β-(δ-acetamido-γ-methylvaleroxy)-17β-acetamido-5-androsten-3β-ol acetate in 75 per cent yield by Beckmann rearrangement of the 16β-(δ-acetamido-γ-methylvaleroxy)-5-pregnen-3β-ol-20-one acetate oxime by treatment with excess POCl₃ in dry pyridine for 3 hr at 0°. Ruggieri *et al.*^{67,68} have also carried out Beckmann rearrangement and Schmidt reactions with some 20-oxosteroids.

Reactions with α,β-Unsaturated Ketones

We may begin this discussion by reference to the work of Rosenkranz *et al.*⁶⁹ which has been of great practical significance. These workers carried Beckmann rearrangement of 5,16-pregnadien-3β-ol-20-one 3-acetate 20-oxime (CXXI) using *p*-acetamidobenzenesulphonyl chloride as catalyst and obtained dehydroepiandrosterone (CXXII). Working under appropriate conditions the intermediate amide (CXXIII; λ_{max}^{EtOH} 240 mμ, log ε 3.82) could also be isolated. Taking a hint from Hauser and

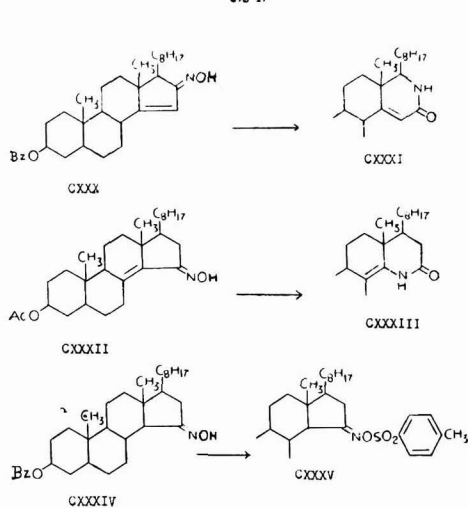
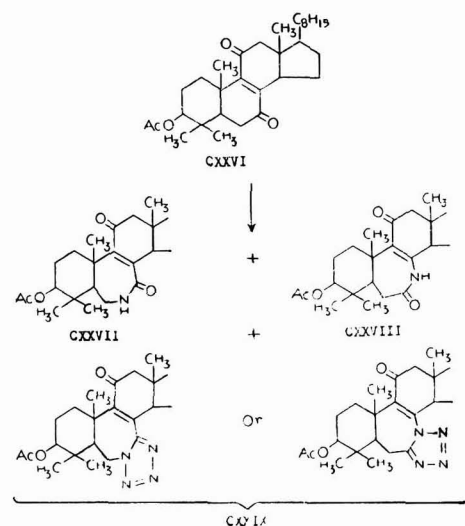


Hoffenberg⁷⁰ who used boron trifluoride in the Beckmann rearrangement of some acetoximes and obtained in fairly good yield the corresponding amides, Romo and Vivar⁷¹ considered the use of boron trifluoride as catalyst in the rearrangement of Δ¹⁶-20-ketoximes. They found that the oxime (CXXI) when subjected to the action of boron trifluoride etherate in benzene solution gave dehydroepiandrosterone acetate (CXXII, acetate), but when the rearrangement of the oxime (CXXI) was carried out in acetic anhydride it undertook a different course and two products were obtained, the structures of which were established to be 17β-methyl-18-nor-5,13(14)-isopregnadiene-3β,16z-diol-20-one diacetate (CXXIV) and 16-acetyl-17-acetyl-5,16-androstadien-3β-ol acetate (CXXV) respectively.



The first example of the introduction of nitrogen atom into a ring of steroid nucleus having α,β-unsaturated ketone function through the use of reactions under discussion was reported by Barton and coworkers⁵⁸, who during their study on the constitution of lanostadienol conducted the following reaction. Treatment of compound (CXXVI) in chloroform solution with conc. sulphuric acid and powdered sodium azide at 0° for 45 min. gave a mixture which was resolved into compound (CXXVII; λ_{max}^{EtOH} 249 mμ, ε 7500), (CXXVIII; λ_{max}^{EtOH} 293 mμ, ε 13000), and (CXXIX; λ_{max}^{EtOH} 265 mμ, ε 6500). The mixture of (CXXVII) and (CXXVIII) in approximately the same proportions was obtained by Beckmann rearrangement of (CXXVI).

Tsuda and Hayatsu⁶³ reported the Beckmann rearrangement of 16-keto-14-cholestenyl benzoate oxime (CXXX) with *p*-toluenesulphonyl chloride-pyridine to 17-aza-16-keto-14-D-homocholesten-3β-ol benzoate (CXXXI). Similarly, 15-keto-8(14)-cholesten-3β-ol acetate oxime (CXXXII) yielded 15-aza-16-keto-8(14)-D-homocholesten-3β-ol acetate (CXXXIII), but interestingly the analogue



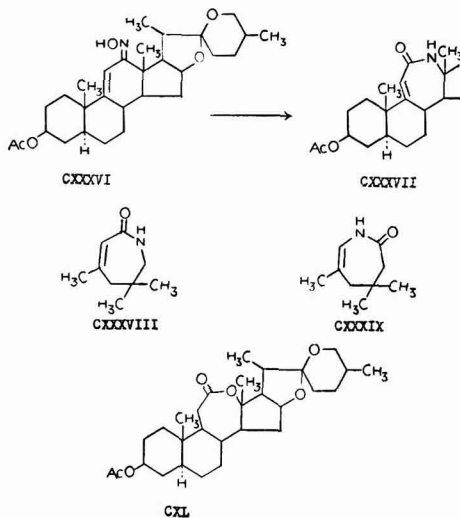
(CXXXIV) which has no double bond in ring C did not rearrange under the conditions employed and only the oxime tosylate (CXXXV) was the product.

The more difficult task in work on azasteroids is the determination of the structures which may be the result of Beckmann rearrangement and Schmidt reactions. The point is further illustrated by the discussions which follow.

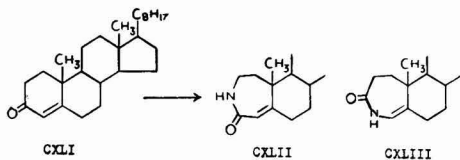
Mazur⁵¹ prepared 3 β -acetoxy-12 α -aza-C-homo-5 α ,22 α -spirost-9(11)-en-12-one (CXXXVII) by rearrangement of 3 β -acetoxy-5 α ,22 α -spirost-9(11)-en-12-one oxime (CXXXVI), which was obtained from hecogenin acetate by oxidation with selenium dioxide in pyridine at room temperature and oximation. The rearrangement of compound (CXXXVI) to (CXXXVII) was done with TsCl in pyridine at room temperature. The structure (CXXXVII) was tentatively assigned by comparing the ultraviolet absorption with the then known

α,β -unsaturated amides of α,β -unsaturated amines. The compound (CXXXVII) has $\lambda_{\text{max}}^{\text{MeOH}}$ 220 μ (ϵ 15800). The point was further considered by Mazur⁷² who, on the basis of studies on isomeric isophorone oximes, conclusively showed that α,β -unsaturated lactams of the type (CXXXVIII) showed maximum around 220 μ while those of type (CXXXIX) absorb around 240 μ .

The compound (CXXXVII) on catalytic hydrogenation yielded 3 β -acetoxy-12 α -aza-C-homo-5 α ,22 α -spirostan-12-one (LXXII) which was also obtained directly by rearrangement of hecogenin acetate oxime. The ultraviolet absorption data of compound (CXXXVII) was taken to be an indirect proof for structure (LXXII). An unequivocal proof of the correctness of this structure was provided by Bladon and McMeekin^{73,74} who treated the lactam acetate (LXXII) with sodium nitrite in acetic acid and acetic anhydride at 0° and isolated hecololactone acetate (CXL), identical with authentic specimen⁷⁵.

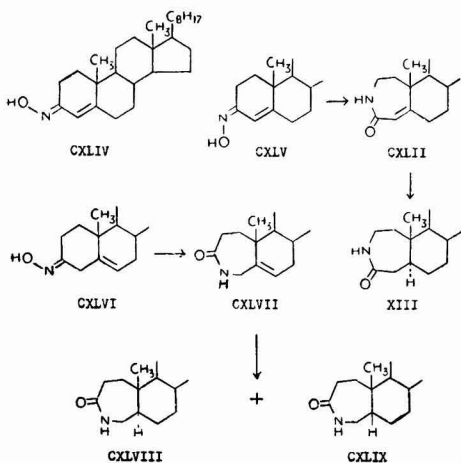


As the androgenic, progestational and cortical hormones contain α,β -unsaturated ketone system in ring A, Doorenbos and Singh⁷⁶ sought to prepare by Schmidt reaction A-homosteroid analogues from steroids having α,β -unsaturated ketone systems in ring A. The initial studies were carried out with 4-cholesten-3-one (CXL I). Nothing crystalline was obtained when the Schmidt reaction was carried out by the usual procedures with sulphuric acid as the catalyst. However, when polyphosphoric acid was used as the solvent and catalyst, there was obtained 3-aza-A-homo-4 α -cholesten-4-one (CXLII; $\lambda_{\text{max}}^{\text{EtOH}}$ 220 μ , $\log \epsilon$ 4.05). The enamine lactam structure (CXLIII) was excluded and structure (CXLII) assigned to the product, (a) because of its resistance to hydrolysis, (b) since its spectrum resembles that of known steroidal α,β -unsaturated lactams, and differs from known steroidal enamine lactams, and (c) because it is hydrogenated to a



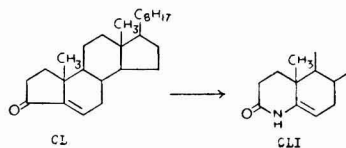
mixture of 3-aza-A-homo-5 α -cholestan-4-one (XIII) and 3-aza-A-homo-5 β -cholestan-4-one (XVII).

Shoppee *et al.*⁷⁷ examined the Beckmann rearrangement of 4-cholesten-3-one oxime as a model for the preparation of azasteroids in the androstane series. The oxime of cholestenone had been reported by Ralls⁷⁸ to exist in two forms regarded as geometrical isomerides, 'A'-form as *syn*-isomer (OH *syn* to the 4,5-double bond), and 'B'-form as *anti*-isomer. On the basis of the accepted *trans* interchange in Beckmann rearrangement, Shoppee *et al.*⁷⁷ conclude as a result of the reactions detailed that the 'A'-form is the *anti*-oxime (CXLIV) and the 'B'-form, contrary to Ralls' conclusion, is the *syn*-oxime (CXLV). The oximes of α,β -unsaturated steroid ketones, according to Shoppee *et al.*⁷⁷, undergo the Beckmann rearrangement less readily than those of saturated ketones. Rapid addition of the *syn*-oxime (CXLV) of 4-cholesten-3-one to an excess of SOCl_2 , cooled to -20° , gave an almost colourless solution becoming pale yellow, when this solution was at once poured into cold water, only traces of the lactam (CXLII) could be isolated, but by immediate addition to 4*N* sodium hydroxide at 20° , or better at 80° , about 20 per cent yield of the lactam was obtained, with some unchanged oxime. The *anti*-oxime (CXLIV) failed to rearrange even under more vigorous conditions; in benzene, where the *anti*- is converted into *syn*-form, the product was the same lactam (CXLII) which show absorption at 222μ ($\log \epsilon$ 4.1). The structure of the lactam as (CXLII), 3-aza-A-homo-4a-cholesten-4-one, rather than isomeric 3-oxo-4-aza structure (CXLIII) is supported by the UV and IR spectral characteristics^{51,72,76}, and looks identical with the same compound obtained by Doorenbos and Singh⁷⁶

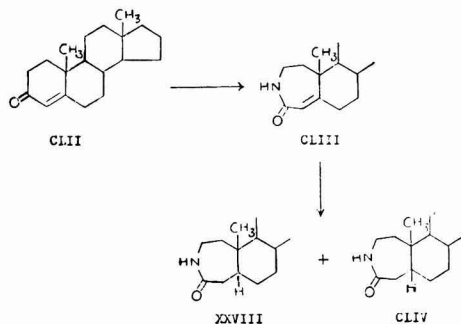


by Schmidt reaction with 4-cholesten-3-one. The lactam (CXLII) was hydrogenated⁷⁷ with palladium-calcium carbonate or palladium-charcoal in ethanol, giving 3-aza-A-homo-5 α -cholestan-4-one (XIII), m.p. 294.6° , $[\alpha]_D + 41^\circ$. This compound, but with m.p. 268.71° , $[\alpha]_D + 16^\circ$, was described by Shoppee and Sly²⁶ as the sole product of the Beckmann rearrangement of 5 α -cholestan-3-one, but it now appears that this reputed compound is an inseparable mixture or a molecular compound of the 3-aza-A-homo-5 α -cholestan-4-one (XIII) and the isomeric Beckmann rearrangement product 4-aza-A-homo-5 α -cholestan-3-one (CXLVIII). In analogy with this finding it is suggested that the preparation, m.p. 166.74° (clear 195°), $[\alpha]_D + 42^\circ$, described by Shoppee and Sly²⁶ as 3-aza-A-homo-5 β -cholestan-4-one (XVII) may have been an inseparable mixture or a molecular compound of (XVII) and 4-aza-A-homo-5 β -cholestan-3-one (CXLIX). The lactams (CXLVIII) and (CXLIX) were resolved from hydrogenation product of 4-aza-A-homo-5-cholesten-3-one (CXLVII), the latter being obtained by Beckmann rearrangement of *anti*-oxime (CXLVI) of 5-cholesten-3-one.

Shoppee *et al.*³⁰ report that the oxime of Δ -norcholest-5-en-3-one (CL) on Beckmann rearrangement with SOCl_2 at -20° gives single lactam 4-azacholest-5-en-3-one (CLII), m.p. 253° , $[\alpha]_D - 92^\circ$, λ_{max} 235μ ($\log \epsilon$ 4.1), which is identical with the same compound prepared⁷⁹⁻⁸¹ from Windaus keto acid.

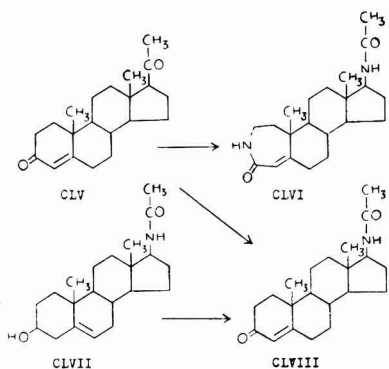


Shoppee and Krueger³⁴ have prepared 3-aza-A-homoandrost-4a-en-4-one (CLIII) by Beckmann rearrangement of non-crystalline oxime of androst-4-en-3-one (CLII). The lactam (CLIII) exists in two polymorphic forms with the same UV absorption spectrum ($\lambda_{\text{max}}^{\text{EtOH}}$ 222μ , $\log \epsilon$ 4.15) and showing IR absorption spectra different in the solid state but identical in the solution. Hydrogenation of either polymorph over palladium-calcium carbonate in ethanol gave 3-aza-A-homo-5 α -androst-4-one (XXVIII); a small quantity of the epimeric 5 β compound (CLIV) was also isolated and the



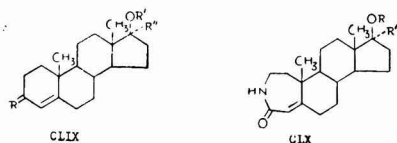
proportion of it was greater on hydrogenation with platinum in acetic acid.

Doorenbos and Singh⁷⁶ also obtained 3-aza-A-homoandrosterone derivatives by carrying out the Schmidt reaction with progesterone (CLV) under the conditions developed for cholesterolone. 17 β -Acetylamino-4-androsten-3-one (CLVIII) was obtained with 1 mole of sodium azide and 17 β -acetylamino-3-aza-A-homo-4a-androsten-4-one (CLVI) with an excess of sodium azide. The structural assignments were made on the basis of analysis and spectral studies. Compound (CLVIII) was prepared by Oppenauer oxidation of 17 β -acetylamino-5-androsten-3 β -ol (CLVII) and shown by means of mixed melting point and spectra being identical with the sample prepared by the Schmidt reaction of progesterone.



The other examples of the use of 4-en-3-ones for the synthesis of aza analogues are the works of Mazur⁸² and Mazur and Pappo⁸³. The general synthetic scheme of Mazur⁸² involved the protection of the 17-hydroxyl group as an ester, formation of the oxime, Beckmann rearrangement (SOCl_2 in dioxane) to a seven-membered lactam and saponification of the 17-ester. Thus, testosterone propionate gave the oxime (CLIX; $\text{R}=\text{NOH}$, $\text{R}'=\text{C}_2\text{H}_5\text{CO}$, $\text{R}''=\text{H}$) which was rearranged to the lactam (CLX; $\text{R}=\text{C}_2\text{H}_5\text{CO}$, $\text{R}'=\text{H}$) and hydrolysed to 3-aza-17 β -hydroxy-A-homo-4a-androsten-4-one (CLX; $\text{R}=\text{R}'=\text{H}$). 17 α -Methyltestosterone acetate via oxime (CLIX; $\text{R}=\text{NOH}$, $\text{R}'=\text{CH}_3\text{CO}$, $\text{R}''=\text{CH}_3$) yielded lactam (CLX; $\text{R}=\text{CH}_3\text{CO}$, $\text{R}'=\text{CH}_3$), which after saponification formed 3-aza-17 β -hydroxy-17 α -methyl-A-homo-4a-androsten-4-one (CLX; $\text{R}=\text{H}$, $\text{R}'=\text{CH}_3$).

Mazur felt that NMR spectroscopy might distinguish between *syn*- and *anti*- α,β -unsaturated oximes due to the expected shift in the vinyl proton position caused by the proximity of the hydroxyl group in the *syn*-oxime. Considering the observation of Phillips⁸⁴ that *syn*- and *anti*-aldoximes showed



different chemical shifts of the aldehyde proton, the isomers of isophorone oxime of known configuration were studied. Thus, the NMR spectrum of *syn*-isophorone oxime showed a downfield shift of the vinyl proton of 42 cycles/sec. downfield relative to the *anti*-oxime. The same effect was observed with 4-en-3-oximes — 43 cycles/sec. downfield shift for *syn*-(CLIX; $\text{R}=\text{NOH}$, $\text{R}'=\text{C}_2\text{H}_5\text{CO}$, $\text{R}''=\text{H}$), 42 cycles/sec. for *syn*-(CLIX; $\text{R}=\text{NOH}$, $\text{R}'=\text{H}$, $\text{R}''=\text{CH}_3$) — and in addition, the 19-methyl peak was shifted downfield 2-3 cycles/sec. in the *syn*-isomer relative to the *anti*-isomer. The same results are reported by Slomp⁸⁵. Thus, combination of NMR and UV spectra permit assignment of configuration to unsaturated ketoximes and their Beckmann rearrangement products and, within limits, allow percentage composition of mixtures to be determined.

Another indication drawn by Mazur⁸² from his research on 3-aza-A-homoandrogens is that the Beckmann product is not necessarily related configurationally to the starting oxime according to the accepted mechanism of the rearrangement. He observed that under the conditions employed (SOCl_2 in dioxane) apparently led to a thermodynamically controlled product for the α,β -unsaturated oximes. In both cases, only one lactam (the 3-aza-4a-en-4-one) could be isolated which would be the isomer expected from the *syn*-oxime. However, NMR showed testosterone propionate oxime (CLIX; $\text{R}=\text{NOH}$, $\text{R}'=\text{C}_2\text{H}_5\text{CO}$, $\text{R}''=\text{H}$) to be a mixture of *syn*- and *anti*-isomers containing only about 10 per cent *syn*-isomer. 17 α -Methyltestosterone acetate gave the pure *anti*-oxime with no detectable amount of *syn*-oxime present. Unexpectedly, 17 α -methyltestosterone oxime (CLIX; $\text{R}=\text{NOH}$, $\text{R}'=\text{H}$, $\text{R}''=\text{CH}_3$) was a 1:1 mixture of *syn*- and *anti*-forms, presumably a molecular complex. One cannot say with certainty whether the conditions of the rearrangement caused isomerization of the starting oximes or whether the reaction proceeded through an intermediate having little or no configurational stability. In any case, Mazur⁸² suggests the need for caution in relating stereochemistry of oximes with the structure of derived lactams.

Shroff^{85a} has recently studied the C(2)-methylene resonance signals of the enamine and α,β -unsaturated lactams. For example, 4-methyl-4-aza-5-pregnene-3,20-dione and 17 β -acetylamino-3-aza-A-homo-4a-androsten-4-one (CLVI) methylene proton resonances at 2.54 and 3.22 respectively. The chemical shift (0.7 p.p.m.) can be useful in distinguishing between the two types of lactams.

Shoppee *et al.*⁸⁶ observed recently that the 3-oxime of pregn-4-ene-3,20-dione (CLXI; $\text{R}=\text{O}$) which showed peaks at τ 3.52 (*syn*-) and 4.23 (*anti*-) in a ratio corresponding to 86 per cent *anti*-, on Beckmann rearrangement with SOCl_2 at 10° gave a 15 per cent yield of 3-aza-A-homopregn-4a-ene-4,20-dione (CLXII; $\text{R}=\text{O}$) (λ_{max} . 224 μ , $\log \epsilon$ 4.2) as the only product, the yield of the same lactam (CLXII; $\text{R}=\text{O}$) being raised to 58 per cent by conducting the reaction in dioxane. This finding supports the observation by Mazur⁸² that the product from the Beckmann rearrangement, with

SOCl_2 in dioxane, of the oximes of unsaturated ketones is not necessarily related configurationally to the starting oxime. Evaluating in retrospect, on the basis of NMR studies, the work reported earlier by Shoppee *et al.*⁷⁷, it is found that the oxime of 4-cholesten-3-one (m.p. 152°) previously reported as the *syn*-isomer (CLXV) is a mixture of *syn*- (τ 3.52) and *anti*- (τ 4.23) in the ratio 2:3. It is mentioned that the ratio of *anti*- to *syn*-isomers appears to be due to fractional crystallization rather than the interconversion of the isomers, since the pure *anti*-oxime of 4-cholesten-3-one has been found to be unchanged when recrystallized from solvents of different polarity.

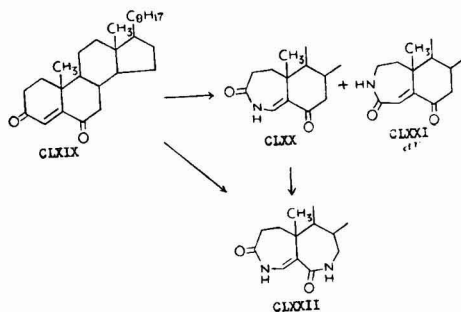
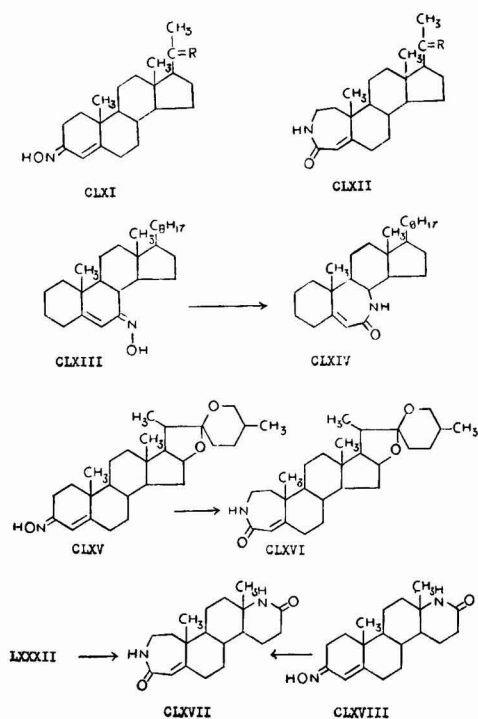
Recently, Shoppee and coworkers^{86a} have reported that *anti*-isomer of 5 α -cholest-1-en-3-one oxime does not rearrange with SOCl_2 at 20°. However, the *syn*- component yields 4-aza-3-oxo-A-homo-5 α -cholest-1-ene in good yield under the same conditions. The NMR data are discussed.

Shoppee *et al.*⁴³ have extended work on the preparation of azasteroids from α,β -unsaturated ketones to the B-ring of steroids. Cholest-5-en-7-one oxime (CLXIII), which proved to be *syn*-isomer by its NMR spectrum, has been found to rearrange with SOCl_2 in dioxane or with SOCl_2 alone to give 78 and 88 per cent yields respectively of the expected lactam (CLXIV; $\lambda_{\text{max}}^{\text{EtOH}}$ 221 m μ , log ϵ 4.2), which by hydrogenation with palladium-charcoal in ethanol affords the saturated lactam (XLVIII; R=H).

The discussion on the reactions with α,β -unsaturated ketones may be concluded by referring to some results from our own laboratory. Singh and Kaw⁸⁷ prepared the oxime (CLXV) from 22 β -spirost-4-

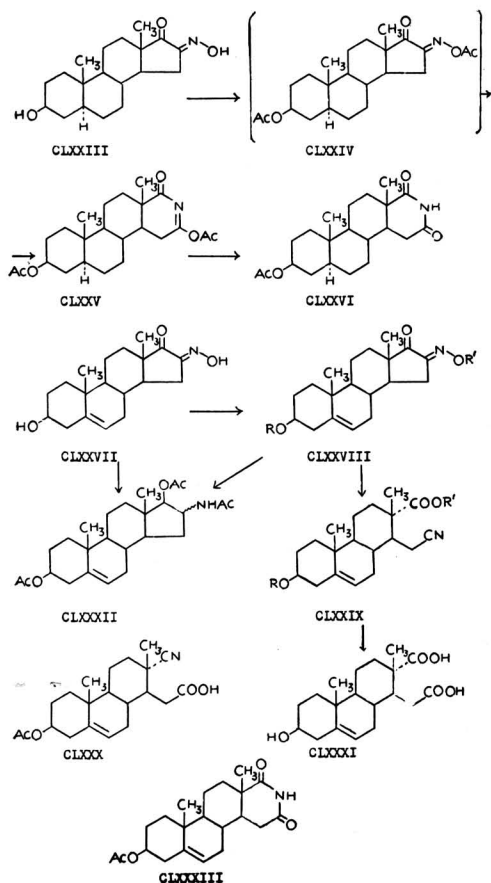
en-3-one and rearranged it to 3-aza-A-homo-22 β -spirost-4a-en-4-one (CLXVI; $\lambda_{\text{max}}^{\text{EtOH}}$ 217 m μ , log ϵ 4.40) by SOCl_2 in dioxane system. Singh and Parashar⁸⁸ have prepared 3,17a-diaza-A,D-bishomo-androst-4a-ene-4,17-dione (CLXVII) ($\lambda_{\text{max}}^{\text{MeOH}}$ 219.5 m μ , log ϵ 4.31) from (LXXXII) by Schmidt reaction in polyphosphoric acid. The lactam (CLXVIII) has also been obtained by rearrangement of the oxime (CLXVIII).

Another interesting problem which has engaged the attention of Singh and Padmanabhan⁸⁹ pertains to synthesis of aza analogues from cholest-4-ene-3,6-dione (CLXIX; $\lambda_{\text{max}}^{\text{EtOH}}$ 251.5 m μ). Schmidt reaction with compound (CLXIX) using equimolar quantity of sodium azide in polyphosphoric acid gave products 'A' ($\lambda_{\text{max}}^{\text{EtOH}}$ 279 m μ , log ϵ 4.19) and 'B' ($\lambda_{\text{max}}^{\text{EtOH}}$ 228 m μ , log ϵ 3.95; 278 m μ , log ϵ 3.73). Assuming the greater reactivity of 3-keto- to 6-keto group and drawing analogy with the work of Barton and coworkers⁸⁸ on same kind of system, the structure (CLXX) is assigned to 'A', and 'B' is considered to be a mixture of (CLXX) and (CLXXI). The dione (CLXIX) has been converted by using bimolar quantity of sodium azide to the diaza analogue, 4,7-diaza-A,B-bishomocholest-4-ene-3,6-dione (CLXXII; $\lambda_{\text{max}}^{\text{EtOH}}$ 250 m μ , log ϵ 4.1). The same compound has been obtained by Schmidt reaction with (CLXX). The structure (CLXXII) is based on the observation that there is hypsochromic shift in going from (CLXX) to (CLXXII), an indication for the formation of an α,β -unsaturated lactam in contrast to enamine lactam.



Rearrangement of α -Oximino Ketones

Heard *et al.*⁹⁰, while attempting to prepare 3 β -acetoxy-16-acetoximinoandrost-17-one (CLXXIV) from 3 β -hydroxyandrostane-16,17-dione 16-oxime (CLXXIII) by allowing the latter to stand overnight in a mixture of acetic anhydride and pyridine at room temperature, got a diacetate to which they assigned the structure (CLXXV) which is in agreement with the hypsochromic shift in the UV maximum. The diacetate (considered to be CLXXV) has absorption peak at 223 m μ (ϵ 9431) as compared with the peak at 240 m μ shown by (CLXXIII), and the structure (CLXXIV) was excluded considering that if this were the structure then there should have been bathochromic shift as had been observed in the case of testosterone

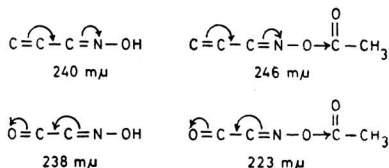


oxime (λ_{\max} , 246 μ , ϵ 23976) which causes a bathochromic displacement of the absorption maximum of 5 μ as compared with testosterone (λ_{\max} , 241 μ , ϵ 19520). Further proof in favour of structure (CLXXV), considered to be Beckmann rearrangement intermediate, given was the reaction of (CLXXV) by alcohol followed by base to form a compound, soluble in sodium carbonate, and believed to be the imide (CLXXXVI).

The earlier work of Kendall and coworkers⁹¹ and Regan and Hayes⁹⁰ (which possibly had not been critically evaluated by Heard⁹⁰) had shown that the acylation of 16-oximino-5-androsten-3 β -ol-17-one (CLXXVII) with acetic anhydride gives a product which was assigned the structure 3 β -acetoxy-16-oximino-5-androsten-17-one (CLXXVIII; R=Ac, R'=H), m.p. 183-4⁹¹, on the basis of elemental analysis and conversion with SOCl₂, conditions for a Beckmann transformation, to a ring-D imide (CLXXXIII).

In the course of related studies, Hassner and Pomerantz⁹² on doing acylation of (CLXXVII) obtained a monoacetate and a diacetate, which in contrast to the assignments by Kendall and coworkers⁹¹, Regan and Hayes⁹⁰ and Heard⁹⁰, are taken to be

3 β -acetoxy-16,17-*seco*-5-androstene-16-nitrile-17-oic acid (CLXXIX; R=Ac, R'=H), m.p. 184-5°, and 3 β -acetoxy-16-acetoximino-5-androsten-17-one (CLXXVIII; R=R'=Ac). The diacetate (CLXXVIII; R=R'=Ac) was very unstable and was converted readily into compound (CLXXIX) (R=Ac, R'=H) by aqueous acid or base, on recrystallization from aqueous solvents, or even on standing in air. The incorrectness of the assignment of structure (CLXXVIII; R=Ac, R'=H) to the monoacetate was obvious from the IR spectrum which showed absorption bands at 2250 cm^{-1} , characteristic of a nitrile group, and 2600-2700 and 1700 cm^{-1} indicative of a carboxy group. These results and the chemical properties of the material, namely formation of methyl ester and solubility in aqueous sodium carbonate, suggested the structure (CLXXIX; R=Ac, R'=H) or (CLXXX) for the monoacetate, but the latter structure was excluded as (CLXXIX) on hydrolysis by heating with potassium hydroxide in glycerol gave the known dicarboxylic acid (CLXXXI). That the structure of the diacetate was (CLXXVIII; R=R'=Ac) rather than analogous to Beckmann rearrangement intermediate (CLXXV) proposed by Heard⁹⁰ was proved by showing that the steroid nucleus was intact in (CLXXVIII; R=R'=Ac), for example, (CLXXVII) and (CLXXVIII; R=R'=Ac), both on reduction with lithium aluminium hydride followed by acetylation gave the product 3 β ,17 β -diacetoxy-16-acetamino-5-androstene (CLXXXII). It is argued that the conversion of Heard's imidoyl ester (CLXXV) by alcohol to an oil followed by base hydrolysis to an imide (CLXXVI), soluble in aqueous sodium carbonate, is actually the cleavage of the diacetate of the type (CLXXVIII; 5,6-dihydro, R=R'=Ac) to the nitrile ester (CLXXIX; 5,6-dihydro, R=Ac, R'=CH₃) followed by hydrolysis to the carbonate soluble acid (CLXXIX; 5,6-dihydro, R=R'=H). The hypsochromic shift in the UV absorption observed in going from the α -oximino ketone (CLXXIII) and (CLXXVII; λ_{\max} , 238 μ) to its acetate (CLXXIV) and (CLXXVIII; R=R'=Ac) (λ_{\max} , 223 μ) respectively seems to be general for a system where the oximino hydroxyl is *anti* to the keto group. An explanation for the latter phenomenon lies in the recognition that the chromophore in α -oximino ketones is polarized in a different direction as the chromophore in α,β -unsaturated oximes:



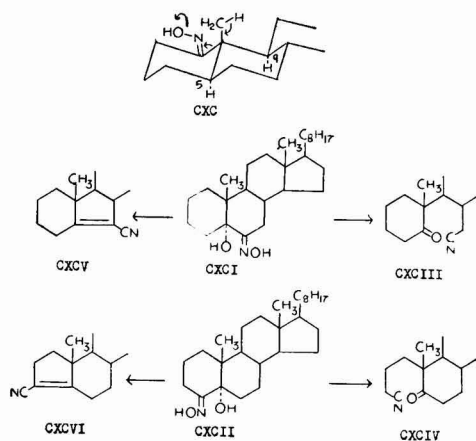
The conclusion of Hassner and Pomerantz⁹² was that esters of α -oximino ketones undergo facile cleavage with water or ethanol rather than Beckmann rearrangement, but later Hassner *et al.*⁹³ observed to their surprise that free oxime *anti*-16-oximino-5-androsten-3 β -ol-17-one (CLXXVII)

with boiling acetic acid and acetic anhydride gives mainly the imide (CLXXXIII), a Beckmann rearrangement product. It appears that cleavage of the oxime acetate (CLXXVIII; R=R'=Ac) to nitrile acid (CLXXIX; R=Ac, R'=H) precedes the formation of the Beckmann rearrangement product (CLXXXIII).

The earlier postulation that oxime (CLXXVIII; R=Ac, R'=H) undergoes rearrangement with SOCl₂ to yield imide (CLXXXIII) has been shown to be incorrect, because of mistaken identity of the starting material which has actually been nitrile acid (CLXXIX; R=Ac, R'=H) rather than oxime (CLXXVIII; R=Ac, R'=H). The formation of imide (CLXXXIII) evidently takes place via an acid chloride of (CLXXIX; R=Ac, R'=H) followed by ring closure and hydrolysis and this inference is borne out by the experimental work of Hassner and Pomerantz⁹².

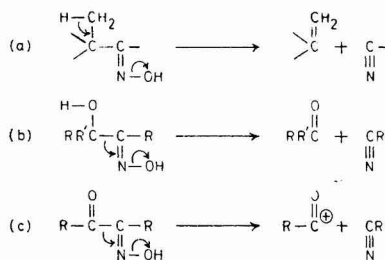
Rearrangement of α -Hydroxy Ketoximes

Continuing with their work on azasteroids, Shoppee *et al.*⁹⁴ conducted the rearrangement with 5 α -cholestan-1-one oxime (CLXXXIV), A-nor-5 α -cholestan-1-one oxime (CLXXXV), 5 α -cholestan-2-one oxime, 5 α -cholestan-4-one oxime, and 5 α -cholestan-6-one oxime. The Beckmann rearrangement of these five steroid ketoximes gave the expected lactams but only in cases of (CLXXXIV) and (CLXXXV) there were formed in addition abnormal products, ω -cyano-olefins. Thus, it was observed that compound (CLXXXIV) on treatment with SOCl₂ at 20° gave approximately equal amounts of the normal rearrangement product, 1-aza-A-homo-5 α -cholestan-2-one (CLXXXVI) and the abnormal 'second-order' Beckmann cleavage product (CLXXXVII). Similarly, compound (CLXXXV) furnished 1-aza-5 α -cholestan-2-one (CLXXXVIII) and 1-cyano-1,10-*seco*-compound (CLXXXIX) in 35 and 50 per cent yields respectively. The formation of ω -cyano-olefins in addition to lactams by rearrangement of alicyclic ketoximes, like cyclopentanone oxime and cyclohexanone oxime⁹⁵ is known, but the cause of the production of ω -cyano-olefins from 1-ketoximes (CLXXXIV) and (CLXXXV), in contrast with the 4-ketoxime and 6-ketoxime, is not very clear. Considering that the displacements are concerted, the formation of 1-cyano-1,10-*seco*-compounds (CLXXXVII and



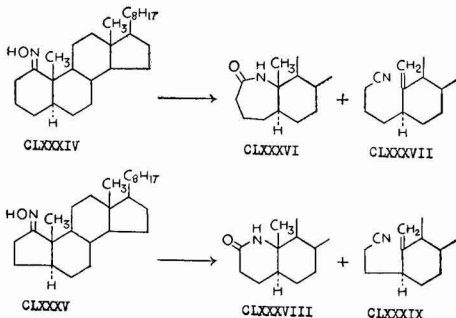
CLXXXIX) is explained on the basis of stereoelectronic requirement for a *trans*-coplanar arrangement (CXC) of the C-H bond broken to the C-C bond cleaved.

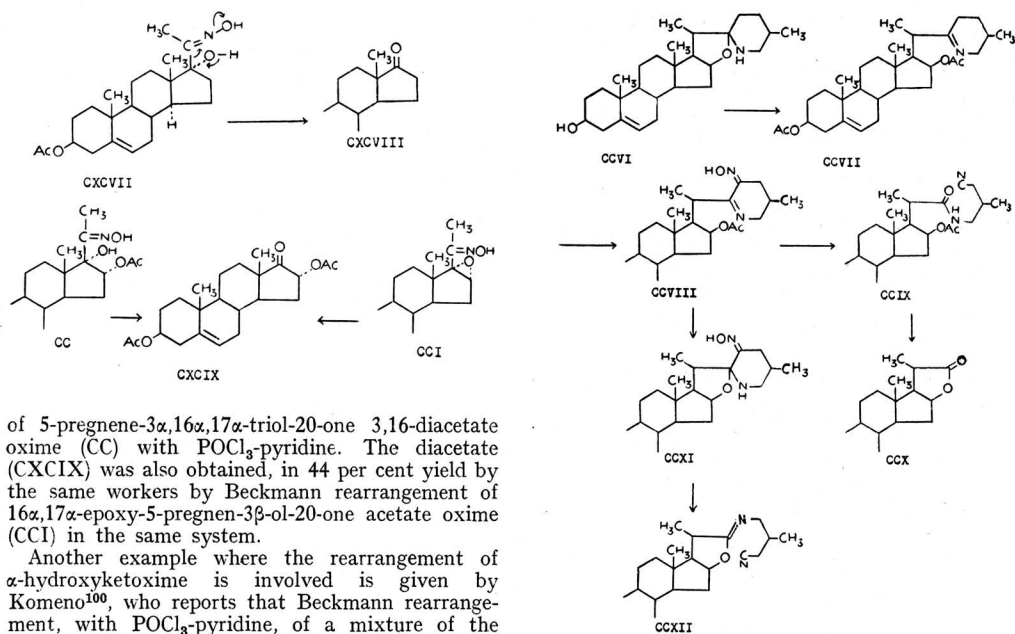
The close analogy of the formation of ω -cyano-olefins (a) for the ready production of aldehydes or ketones and cyanides from *anti*- α -hydroxy-oximes; (b) of acylium cations and cyanides from *anti*- α -ketoximes; and (c) by 'second type' Beckmann change⁹⁶, led Shoppee and Roy⁹⁷ to examining the behaviour of some steroid α -hydroxy ketoximes. They found that 5-hydroxy-5 α -cholestan-6-one oxime (CXCI) and 5-hydroxy-5 α -cholestan-4-one oxime (CXCVI) on treatment with SOCl₂ at -20° or with hydrogen chloride in ether at 15° gave nearly quantitative yields of the ω -cyano-olefins (CXCIV) and (CXCV) respectively. The oximes (CXCI) and (CXCVI) are converted into 6-cyano- β -norcholest-5-ene (CXCIV) and 3-cyano-A-norcholest-3-ene (CXCV) respectively.



On the basis of these findings Shoppee and Roy⁹⁷ argue that the conversion of 3 β -acetoxy-17 α -hydroxypregn-5-en-20-one oxime (CXCXVII) by POCl₃-pyridine at 0° in 98 per cent yield into androst-16-en-3-one (CXCXVIII) may proceed by elimination of methyl cyanide rather than by normal rearrangement to the 17 β -acetamido-17 α -alcohol and loss of acetamide.

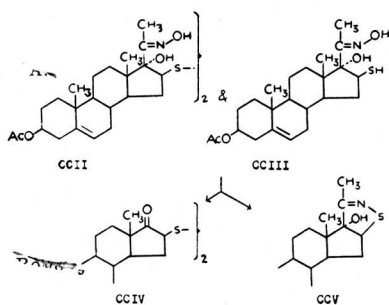
In the same context may be mentioned the work of Klimova and Suvorov⁹⁹ who prepared 5-androst-16-en-3-one diacetate (CXCXIX), in 71 per cent yield, by Beckmann rearrangement





of 5-pregnene-3 α ,16 α ,17 α -triol-20-one 3,16-diacetate oxime (CC) with POCl₃-pyridine. The diacetate (CXCIX) was also obtained, in 44 per cent yield by the same workers by Beckmann rearrangement of 16 α ,17 α -epoxy-5-pregnen-3 β -ol-20-one acetate oxime (CCI) in the same system.

Another example where the rearrangement of α -hydroxyketoxime is involved is given by Komeno¹⁰⁰, who reports that Beckmann rearrangement, with POCl₃-pyridine, of a mixture of the oxime disulphide (CCII) and the oximethiol (CCIII), which were obtained from 3 β ,17 α -dihydroxy-16 β -thiocyanato-5-pregnen-20-one 3-acetate by the action of hydroxylamine afforded 16 β ,16 β' -dithiobis-(3 β -hydroxy-5-androsten-17-one acetate) (CCIV) and a byproduct C₂₃H₃₃NO₃S, which has now¹⁰¹ been identified to be 3'-methyl-16,17-dihydro-isothiazolo-(1,2-*b*)-16,17)androst-5-ene-3 β ,17-diol 3-acetate (CCV).

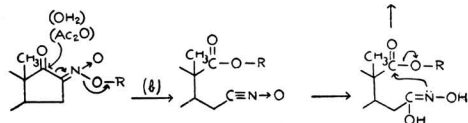
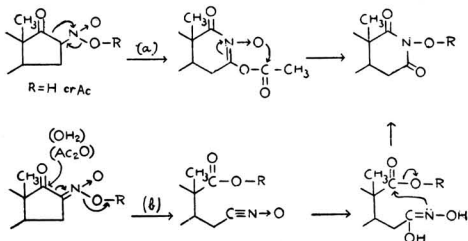
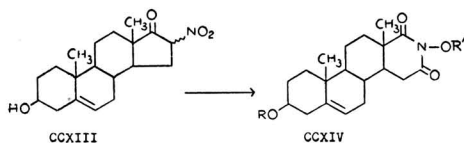


Other Rearrangements

Under this head we discuss two papers which could not be put under any of the classifications. Sato and Ikekawa¹⁰² reported rearrangement of certain oximes in the course of their work on the so-called *pseudo* derivatives 'B' of solasodine and tomatidine, of the type (CCVII; from solasodine), formed from the respective steroid alkaloids by treatment with a solution of ZnCl₂ in acetic anhydride-acetic acid mixture. The *pseudo* diacetate derivative 'B' (CCVII) obtained from solasodine (CCVI) was found to form with nitrous acid, the C₂₃ oximino derivative (CCVIII), $\lambda_{\text{max}}^{\text{EtOH}}$ 232.5 μ .

(log ϵ 4.08), which on treatment with TsCl in pyridine readily suffered rupture of the C₂₂-C₂₃ bond affording, after treatment with water, the cyanamide derivative (CCIX). The same rearrangement was observed with SOCl₂ in benzene-chloroform as medium. This cleavage is another example of the so-called abnormal Beckmann rearrangement—references to different cases of the kind are listed by Sato and Ikekawa¹⁰². That compound (CCIX) is a cyanamide derivative was confirmed by its conversion to the lactone, 3 β ,16 β -dihydroxy-5-bisnor-cholenic-22 \rightarrow 16-lactone (CCX) by solvolysis with methanolic alkali, and this provided a convenient route for the preparation of (CCX), rather an inaccessible lactone. The treatment of the oximino derivative (CCVIII) with methanolic alkali affects cyclization in the *pseudo* compounds 'B' to yield the C₂₃ oximes of solasodine (CCXI). Interestingly, acetylation (acetic anhydride-pyridine, 5°) of the oxime (CCXI) resulted in a concomitant occurrence of rearrangement to produce the imino nitrile (CCXII). The same type of studies were carried out in tomatidine series.

Lastly, a reference may be made to the work of Hassner and Larkin¹⁰³ who report that steroidal α -nitroketones undergo a novel type of rearrangement to N-hydroxyimides under acid conditions. Thus, 16-nitro-5-androsten-3 β -ol-17-one (CCXIII) is converted by treatment with acid in 75 per cent yield to the N-hydroxyimide (CCXIV; R=R'=H) or by treatment with acetic anhydride at room temperature in 80 per cent yield to the corresponding diacetate (CCXIV; R=R'=Ac). Two paths are considered to explain the rearrangement of the α -nitroketones to N-hydroxy or N-acetoxyimides. Path (a) represents a Beckmann rearrangement



of an *aci* form of a nitro compound analogous to that of a conventional oxime. Path (b) represents a cleavage reaction and is analogous to the cleavage of cyclic α -oximinoketones or of their acetates to nitrile acids followed by ring closure to an imide^{92,93}.

Summary

Azasteroids are of potential medicinal interest. This and the mechanistic and stereochemical implications involved in the synthesis and structure elucidation motivate the study of the synthetic nitrogenous steroids.

Various types of biological activity are encountered among azasteroids. There are now known azasteroids with anabolic, antihormonal, antihypercholesterolaemic, vasodilatory, anticancer, anti-inflammatory, or antimicrobial activity, etc.

There are various routes to the synthesis of steroid analogues and this review mainly deals with detailed study of the use of Beckmann rearrangement and Schmidt reactions. After a brief introduction to the mechanisms of these reactions, their application to the steroid field has been taken account of. For the sake of convenience, the discussions have been classified into different sub-heads. First, there are discussed the reactions as applied to steroid ketones, resulting in the introduction of N into rings A, B, C, D, and A and B, and position 5. Next, application of the reactions to α, β -unsaturated ketones is reviewed. The use of the UV and NMR spectroscopy in the study of oximes and the respective lactams has been briefly dealt with. The other sub-classifications pertain to the rearrangement of α -oximinoketones, the rearrangement of α -hydroxyketoximes, and certain other miscellaneous rearrangements. During all these discussions a stress has been laid on the basis of the structure elucidation of the azasteroids resulting from the application of Beckmann rearrangement and Schmidt reaction, and related reaction conditions.

Acknowledgement

This review has been compiled during the literature survey for the research project 'Synthesis

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REVIEWS

FUNDAMENTALS OF SEMICONDUCTOR DEVICES by Joseph Lindmayer & Charles Y. Wrigley (D. Van Nostrand Co. Inc., New York), 1965. Pp. ix+486. Price \$ 11.95

At present, semiconductor electronics is a vital and evergrowing part of the already well-established electronics industry. In addition, the theoretical as well as technological aspects of semiconductor devices have increased several-fold in the last few years. It is thus necessary that well and clearly written books giving the latest on the subject should be available. The book under review does fulfil such a need. This book is written both for the student who is trying to understand the fundamentals of semiconductor devices and the working engineer. The latter category would particularly benefit as the semiconductor courses have only recently been introduced in the colleges. Thus, the many engineers, physicists and chemists working in the transistor field and being handicapped because of lack of basic understanding of the subject would find the book quite helpful.

The presentation of the subject material in the book takes a fresh and rather unconventional approach. A slow introduction of semiconductor theory with an early treatment of transistor operation is followed by a detailed and extensive study of various semiconductor devices and semiconductor theory. The book is divided into twelve chapters. The first five chapters acquaint the reader with a thorough knowledge of diode and transistor operation. The second half of the book discusses a new range of semiconductor devices, additional modes of transistor operation coupled with extensive semiconductor physics. Among the new devices included are four-layer devices, tunnel diodes, integrated microcircuits, metal interface amplifiers, metal base transistor and metal gate transistor.

The book is certainly an excellent addition to several other texts available on the subject. The book is bound to be of great help to both the students as well as the engineers in the field of semiconductor technology.

K. S. BALAIN

THE DYNAMICS OF CONDUCTION ELECTRONS by A. B. Pippard (Gordon & Breach Science Publishers, New York), 1965. Pp. 150. Price \$ 1.95 (paper); \$ 4.95 (cloth)

This book which comprises lecture notes given by Prof. Pippard in 1961 is to be welcomed by workers in the field of dynamics of conduction electrons and the Fermi surface of metals. It gives a brief and clear account of the work done up to that time in this field. The presentation is concerned more with the elucidation of physical processes involved in electronic phenomena and so only simple mathematics has been used. This book may be read with great profit in combination with a book like Kittel's *Quantum theory of solids* which certainly is more concerned with the mathematical details and methods.

The book starts with a brief description of electrons in the periodic field of crystal lattices and then gives an account of transport phenomena in the presence of boundary and size effects and of electric and magnetic fields. It then discusses as to how the experimental information obtained by studying magnetoresistance, de Haas-Van Alphen effect, anomalous skin effect, magnetoacoustic effect and cyclotron resonance can be utilized in different crystalline materials to get information about their Fermi surface without recourse to analytical methods.

The work in this field, done after these lecture notes were written, has been indicated in references given at the end of the book. Some very interesting topics like acoustic amplification, existence of helicon magnetoplasma modes and their coupling with the acoustic waves are indicated in these references. It is felt that materials having solid state plasma and with strong electron-phonon coupling will play a great role in revolutionizing the field of microwave devices. The research workers in this field and anyone interested in learning about this field will profit considerably from Prof. Pippard's book.

R. P. SINGH

OPTICAL MODEL OF THE ATOMIC NUCLEUS by Ivan Uhlhela, Ledislav Gomolcak & Zolenek Pluhar (Academic Press Inc., New York), 1965. Pp. 147. Price \$ 7.75

It is well known that nuclear theory has not kept pace with the accumulation of experimental data on nuclear structure, i.e. nuclear energy levels and nuclear reactions. In the absence of a single comprehensive theory which could explain the general laws governing the structure and behaviour of atomic nuclei, different models have been proposed to explain different aspects of nuclear properties.

This book, which runs into some 140 pages, gives an admirable account of the optical model, which successfully explains the phenomena in the process of nuclear reactions. This model in which the atomic nucleus is considered as resembling a semi-transparent medium is, like the shell model, a model of independent particles. However, in contrast to the shell model, the average potential in which the incident particle is moving is a complex quantity.

The book is divided into six chapters. The first two chapters give the theory of elastic scattering and inelastic scattering (and reactions) respectively, followed by three chapters on the optical model as applied to scattering and reactions in low, medium and high energy ranges. The three energy ranges concerned are low energies up to 10 MeV.; medium energies between 10 and 100 MeV. and high energies above 100 MeV.

Knowledge of quantum mechanics on the part of the reader is assumed, though intricate mathematical computations are avoided in the text. As the authors point out, the book is intended to be useful to both groups of physicists, "those who

measure and those who endeavour to work out the basic theory". This objective has been well achieved.

B. V. THOSAR

STATISTICAL THEORIES OF SPECTRA: FLUCTUATIONS edited by Charles E. Porter (Academic Press Inc., New York), 1965. Pp. xv+576. Price \$ 5.95 (paper bound); \$ 9.50 (cloth bound)

This publication will provide the students coming out of the university with the necessary background for starting research in the highly developing field of statistical spectroscopy. This is a collection of reprints and original papers with a learned introductory review article by the late Dr C. E. Porter.

Illustrating the application of statistical concepts to the complex sequence of prime numbers, Dr Porter has lucidly discussed how the statistical approach may prove useful for the study of the spectroscopy of a quantum system on account of the high complexity of the system. He has discussed the invariance of the Hamiltonian under various types of transformations explaining in detail the time reversal invariance and its connection with the three groups of compatible canonical transformations — orthogonal, unitary and symplectic. The average properties of spectroscopic quantities of major importance have been enumerated and discussed; these averages form the scale for the fluctuation effects. The basic distribution functions have been developed for the Gaussian ensembles and for the three possible symmetry situations — orthogonal, unitary and symplectic. For the actual Hamiltonian matrix of infinite dimension a model Hamiltonian matrix of finite dimension has been considered, the model is expected to retain the essential features of the actual one. The distribution functions have been derived on the assumption that they have to be invariant function of the Hamiltonian and independent in the matrix elements. In the calculation of the volume element, the symmetry property of the Hamiltonian plays an important role; expressions for the volume element have been derived for all three symmetry situations. Distributions of eigen values and eigen vectors can now be easily determined and these in turn can be utilized to obtain the distributions of important physical quantities like energy level spacings, level widths and expectation values, etc.

The research papers collected include important contributions of E. P. Wigner, C. E. Porter, A. Bhor, L. Landau, F. J. Dyson, M. L. Mehta, N. Ullah, R. G. Thomas and many others. The publication will be highly useful to those who intend to pursue research in statistical spectroscopy.

N. C. SIL

tutions in the USSR. In this context it would be helpful if some information is given about the authors and the classes in which the book is intended to be used in the USSR. In the present case no such information is given.

The book is divided into 25 chapters which deal with the basic principles of organic chemistry in more or less conventional manner. The standard of treatment is approximately up to the Honours level of the Indian universities. Two useful features of the book may be mentioned: (i) there are extensive references to the contributions of the Russian chemists and brief life sketches of a number of them (not easily available elsewhere) are given, (ii) Russian industrial processes (e.g. petroleum industry) are frequently referred to and useful information is given.

There are relatively few printing mistakes, e.g. rybose (p. 280), butyryl (p. 547), ozone (instead of osone, p. 279), potassium nitre (instead of nitrate, p. 348). One striking omission is that there are no questions or problems anywhere in the book.

The printing and the get-up of the publication are good.

V.V.S.

ORGANIC REACTIONS: Vol. 14, edited by Arthur C. Cope (John Wiley & Sons Inc., New York), 1965. Pp. vi+498

Organic reactions constitute a well-known series of volumes presenting important synthetic organic chemical reactions that are frequently used in research and constantly used by students as well as by teachers. The present volume includes three important reactions: (1) the Chapman rearrangement, (2) α -amido-alkylations at carbon, and (3) the Wittig reaction. In keeping with the traditions of previous volumes, each chapter provides an authoritative and critical discussion of a reaction giving full details of its mechanism, scope and limitations, experimental conditions and typical important experimental procedures and is completed by a tabular survey. The literature references are extensive.

The Chapman rearrangement covers about 52 pages and deals with material contained in about 100 publications. The rest of the book is almost equally divided between the two other reactions. Wittig reaction has become quite famous for its effectiveness and versatility. Though the reaction is little more than 10 years old, the literature is voluminous and the material collected in the present article is derived from about 400 publications. The account is quite attractive and useful. We can with full justification say that this volume ranks equally with the earlier numbers with regard to its utility and importance and will be a valuable acquisition to any library.

T. R. SESHADRI

THEORETICAL BASIS OF INORGANIC CHEMISTRY by A. K. Barnard (McGraw-Hill Book Co. Inc., New York), 1965. Pp. x+372

With the advent of nuclear age ushered in two decades ago, there has been a marked resurgence

ORGANIC CHEMISTRY by B. Pavlov & A. Terentyev; translated from the Russian by Bores Belitzky (Gordon & Breach Science Publishers, New York), 1965. Pp. 568. Price \$ 16.00

In recent years there has been considerable activity in translating scientific literature from Russian into English in order to bring within the reach of the English-speaking teachers and students science text-books which are in use in the academic insti-

of interest in inorganic chemistry. The impact of quantum mechanics and of modern physical methods of attack to the solution of chemical problems has mainly contributed for the renaissance of inorganic chemistry. The rapid strides made in research in the field of inorganic chemistry and the consequent systematization of the subject have profound implications on the teaching programme, in this branch of chemistry. A reorientation in the method of teaching the theoretical principles and training in practical experiments for undergraduate course has been going on in all the advanced centres of teaching and research.

New kinds of text-books are being published by enlightened teachers to meet this growing demand. The book under review is one important publication which is expected to serve as a basic text for undergraduate students. The book starts with atomic systems and is followed by non-electrostatic and then electrostatic valence forces. Fourth chapter covers non-valence cohesive forces, including metallic bond and semiconductors. Elements of nuclear chemistry forms the fifth chapter. Crystal and molecular structure determination is described in the sixth chapter. The chemistry of the transition elements, the physical principles of inorganic chemistry and the preparation of pure elements and compounds form the subject matter of chapters seven, eight and nine respectively. The book is provided with an index and every chapter ends with references for further reading. It is important to point out that there is no single best way of presenting modern inorganic chemistry and as such there is bound to be some differences of approach to this problem. The present author has made a successful, bold and admirable attempt to fulfil the vital needs of upgrading and updating undergraduate chemistry curriculum for the benefit of both teachers and students.

A.R.V.

INSTRUMENTAL METHODS OF ANALYSIS by H. H. Willard, L. L. Mersitt & J. A. Dean (D. Van Nostrand Co. Inc., New York), 1965. Pp. xviii+784. Price \$ 10.75

This book has all along been a popular choice with those making their initial acquaintance with a new instrumental technique of analysis. The authors have now brought out the fourth edition of the book in a substantially revised and enlarged form, in the light of the growing importance of certain techniques in analytical and pure chemistry. Two new useful chapters on electronic circuitry have been added. Since instrumentation in analysis is getting more and more complicated in view of the necessity for improved limits of detection, greater precision and inline methods of analysis, it is all the more necessary that the analytical chemist knows enough about electronics that goes behind instruments. A unique addition is the inclusion of a separate chapter on 'Process instruments and automatic analysis'. Some of the techniques like NMR and mass spectrometry, infrared and UV absorption spectra are now increasingly used for structural identification. This slant is evident in the new presentation. All but four chapters have been

completely rewritten. Some of the chapters in the previous editions have been split up and subjects treated in separate chapters like the one on ESR and Raman spectroscopy. Thermoanalytical methods are discussed together in a separate chapter.

While the laboratory experiments will prove useful to teachers in planning the laboratory work, the series of numerical problems will help students in understanding the theoretical basis behind the various techniques. The book also gives a listing of abbreviations and symbols to avoid confusion which arises at times due to overlapping use of certain symbols in diverse techniques.

It can be said that the utility of the book has increased manifold by this new edition.

V. T. ATHAVALA

BRIDGED AROMATIC COMPOUNDS by Brandes H. Smith (Academic Press Inc., New York), 1965. Pp. xi+553. Price \$ 14.00

Interest in the chemistry of bridged aromatic compounds originated mainly from considerations of stereochemistry and optical resolution based on restricted rotation. Beginning with the introduction of ANSA compounds by Luttringhaus in the 1930's, greater interest and attention have been paid to this class of compounds from other angles as well. Cram's classical work on the chemistry of cyclophanes investigated the electronic consequences of aromatic rings interacting across space. With the development of ferrocene chemistry, a variety of bridged ferrocenes have also been studied and reported in the literature. Consequent upon such prolific growth of the chemical literature in this direction, a monograph like the one under review is thus most timely.

Brandes Smith has done a commendable piece of work in collating the chemical research in this area. In an introductory chapter, the author has presented a lucid codification of the systematic nomenclature and numbering of the different types of bridged compounds. The ending -phane has been made common to the entire class of compounds. Thus one may easily visualize the systems indicated by such names as acridinophane, thiophenophane, ferrocenophane, etc.

The largest chapter deals with the numerous methods of synthesis leading to bridged aromatic compounds. Among these are the now well-known acyloin synthesis, the Ziegler cyclization at high dilution, the halo-amine and the halo-ether cyclization methods and the cyclization by oxidative coupling. The author has done well to bring out the erroneous structure assignments made in the earlier literature in the course of this discussion. Nearly 160 pages are devoted to the different preparative methods.

The chemical reactions of the aromatic nuclei and those of the bridges are discussed in considerable detail in the two subsequent sections. Brief but useful accounts are also given of the spectral characteristics of the bridged systems. Ultraviolet, infrared and nuclear and electron magnetic resonance spectroscopy are touched upon briefly. Extensive tables running to some seventy pages cover the

physical properties of the bridged compounds reported so far in the literature.

The book is well written in general, although occasional lapses in language make for strained and involved reading.

A volume like this may well stimulate further interest in the area of bridged aromatic compounds and is to be welcomed enthusiastically.

B. S. THYAGARAJAN

METHODS IN CARBOHYDRATE CHEMISTRY: Vol. 5 — GENERAL POLYSACCHARIDES by R. L. Whistler (Academic Press Inc., New York), 1965. Pp. xxii + 463. Price \$ 16.50

Methods in the study of carbohydrates are numerous but they are distributed widely in literature. It would, therefore, be a great advantage to the research worker if these various methods, at least a good majority of them, are found consolidated in a single book. The author of this book has attempted to achieve this colossal task.

The present volume, which is the fifth and the last in the series, deals with general polysaccharides, other than starch and cellulose. It comprises five sections. The first two sections deal with the general methods of isolation and the preparation of the various polysaccharides, while the next two describe their chemical and physical analyses. Section five presents the methods of determining the molecular weights, while the next section deals with the structural methods commonly employed. Section seven gives an account of the derivatives, while section eight contains some selected methods found in other collections, glossary and the index. In each section the subject matter is presented in the form of articles, written by authorities in the field. As in journals, each article is divided into introduction, procedure and references. The book is, therefore, a compilation of articles contributed by various authorities in the field. Since the authors of most of the articles are the actual investigators who originated the methods, the procedures described are certainly authoritative, descriptive and comprehensive, and they are, therefore, of much help to fresh workers in the field. The volume is, so to say, a practical text-book to the routine and research worker in the field of polysaccharides; it is a valuable addition, which facilitates their work.

However, the book is not free from blemishes. Right through the text, expressions like 'is ground 3 hr', 'shaken 1 hr', 'refluxed 2 hr' appear, wherein a noun is unorthodoxly used as adverb, and it is jarring to those who are used to the normal usages like 'is ground for 2 hr', 'is shaken for 1 hr', etc. Even here no consistency has been observed, since similar expressions appear with the preposition 'for' in some places and without it in other places.

Another unhappy feature is the non-syllabic splitting of the words at the end of lines and there are at least 30 such cases. Interesting examples are 'homog-enizer' (p. 7), 'centrif-ugation' (pp. 8 and 172), 'metaperi-odate' (p. 123), 'meas-urement' (p. 253) and 'spectrophotom-eter' (pp. 359 and 376). Although the splitting of 'disodium'

is syllabic (p. 251), it would have been better had the splitting been 'di-sodium' instead of 'diso-dium'.

On going through the book one comes across instances of wrong usage of words and words having been wrongly spelt. There are also some printer's devils. It is hoped that all these will be taken care of in the next edition.

P. S. RAO

TENTH SYMPOSIUM (INTERNATIONAL) ON COMBUSTION organized by The Combustion Institute (The Combustion Institute, Pittsburgh), 1965. Pp. xxvi + 1488. Price \$ 22.00 (members); \$ 35.00 (individuals)

The publication under review is based on papers presented in the Tenth Symposium (International) on Combustion held at the University of Cambridge, England, from 17 to 21 August 1964. One hundred and thirty papers based on different aspects of combustion and flame research have been included. The book begins with an introduction by R. Friedman and the plenary lecture by R. G. W. Norrish, FRS, of the Department of Physical Chemistry, University of Cambridge, a pioneer in the field of combustion kinetics.

The papers presented at the conference have been grouped into a number of sections, such as flame chemistry (13 papers), flame spectroscopy (6 papers), general papers (7 papers), reaction kinetics (12 papers), elementary combustion reactions (24 papers), electrical properties of flame (12 papers), detonation in gas and condensed phase (12 papers), fire research (6 papers), aerodynamics in combustion (22 papers), combustion and flow (10 papers), and solid propellant combustion (6 papers).

Brief mention may be made of some of the interesting papers. Two papers on the effect of additive (methyl bromide as an additive for methane-oxygen flame and carbon tetrachloride for hydrogen-chlorine flame) and two others on cool flames of aldehydes and hydrocarbons are worth mentioning. The section on flame spectroscopy presents papers on important topics like mass spectrometric sampling of flames and a band ratio technique for determining temperatures and concentration of hot combustion gases. While the session on electrical properties of flames includes a few papers on the augmentation of flames by applying high voltage electrical discharge across the reaction zone, the papers on free burning and forest fires are highly interesting and of much practical value. The other important papers are on solid and liquid rocket instability, combustion in supersonic flow, problems in gas turbine combustion, oscillatory combustion in tunnel burners, mechanism of combustion instability and burning of solid propellents.

It may be concluded that the Tenth Symposium (International) on Combustion has in its traditional way dealt with the problems on combustion and flame in a wide spectrum and will be much appreciated like the previous ones by the researchers in this field. However, addition of a few papers on turbulent flame and burning of coal dust would have supplemented the volume.

D. BHADURI

THE SORBY CENTENNIAL SYMPOSIUM ON THE HISTORY OF METALLURGY by Cyril Stanley Smith (Gordon & Breach Science Publishers, New York), 1965. Pp. 580. Price \$ 12.50 (professional edition); \$ 29.00 (reference edition)

This publication, sponsored by the Society for the History of Technology, American Society for Metals, and the Metallurgical Society, American Institute of Mining, Metallurgical and Petroleum Engineers, is probably the only book of its kind on the history of metallurgy, the latter's growth and development across the span of the nineteenth and twentieth centuries. The publication is a memorial to Sorby, called the father of microscopical petrography, and the most brilliant metallurgist of his times. The initial pages of the text furnish the reminiscences— Sorby, the man and his background, followed by the history of metallurgy as shaped by world metallurgists across the decades. The publication has drawn upon the lifetime experiences of the world's most distinguished scientists and metallurgists in compiling different chapters on the growth of science of metallurgy in the background of their purposeful achievements and chance discoveries.

The publication has given most comprehensive references of the researches and metallurgical developments in different parts of the world on various aspects of physical metallurgy, extraction and process metallurgy, heat treatment, phase transformations, experimental methods and research techniques developed, etc. Practical subjects like the processing and production of different types of steels, including the high alloyed steels, magnetic materials, special cast iron, etc., have equally well received due treatment. The developments and growth of specialized research methods and techniques in different countries have been highlighted in special chapters devoted to the subject. While the publication gives an authentic record of the symposium papers presented at the Sorby Centennial Symposium on the History of Metallurgy, the historical sequence of their orderly presentation presents admirable and interesting reading, for which the editor and the sponsors of the publication deserve due compliments of all those engaged in metallurgical profession in one form or the other.

Undoubtedly, the publication will be of immense value to the students, research workers and the metallurgical historians throughout the world. The publication has been profusely illustrated and exceedingly well printed, reminiscing the older metallurgists of their heydays gone by and stimulating the younger of the vast virgin fields and pastures new across the metallurgical horizons confronting them.

The references given at the end of each chapter and the comprehensive index including the dates of births and deaths of leading scientists and metallurgists the world over would be noticed for the first time in any book or text of metallurgical interest and enhance the value of the publication. It is only to be hoped that similar publications on the history of different specialized scientific disciplines, including the science of metallurgy apart from epoch-making industrial revolutionary eras would be highlighted in similar texts of significant histori-

cal value and scientific interest for future scientists and metallurgists of the world in the decades to come.

B. R. NIJHAWAN

IMPROVING THE EFFECTIVENESS OF RESEARCH AND DEVELOPMENT by Robert E. Seiler (McGraw-Hill Book Co. Inc., New York), 1965. Pp. x+210

The book is divided into 14 chapters, each chapter dealing with a specific problem in the field of organization of research. The book is attractively brought out and well produced.

The book is based on survey of 116 American industries and gives some extremely useful information about the size of the company and its research attitudes. The book could, in fact, be divided into three sections. Section one could be the implicit assumptions of the author which though not explicitly stated in the book are an evidence in abundance throughout the discussion of the subject matter. The second section is the opinions of the author which are expressed in very clear, often categorical statements, about the various aspects of research and its organization in an industrial laboratory. The third section deals with the results of the survey of the industries, based on the questionnaire given in appendix, and the correlations made and the inferences drawn.

It would have been very useful and interesting if the assumptions, opinions of the author and the conclusions of the survey were discussed at some length.

After reading the book the reviewer was reminded of a story about operational research. An operational research worker took up a problem about the best shape for the handle of axe. After intensive study of the various facets of the problem for a decade he came to the conclusion: the best shape for the handle of an axe is some sort of a cylinder.

A.R.

ELEMENTS OF STRUCTURAL GEOLOGY by E. S. HALL (Asia Publishing House, Bombay), 1965. Pp. xi+483. Price Rs 16

This book is an enlarged version of the author's earlier book *Outlines of structural geology*. The new book has 14 chapters: The domain and contents of structural geology; Depositional textures and structures; Nondiastrophic structures; Physics of deformation; Environment, time and material; Planar and linear structures and jointing; Faults; Folds; Tectonic analysis of folds; Cleavage; Major structures and tectonics; Igneous rocks; Structural petrology; Geomorphology and structure morpho-tectonics.

The first chapter is a sort of an introduction to the subject in general and to the book in particular. In Chapters II and III primary structures of sediments, penecontemporaneous structures and structures which are secondary, but not caused by diastrophism, are treated. The physical principles governing rock deformation are excellently summarized in Chapters IV and V. Mohr diagrams are appended to Chapter V. The planar and linear structure elements, their statistical examination and a detailed consideration of secondary structures are presented in Chapters VI to X. A broad-based consideration of the structures on a regional scale

and the geotectonics is made in Chapter XI. Structures originated in the fluid phase and other structures characteristic of igneous rocks are dealt with in Chapter XII. Chapter XIII on the structural petrology is a contribution of Prof. E. den Tex, where a very good account of the micro-tectonics is given. The last chapter of the book is devoted to the consideration of the influence of structures on geomorphology.

The subject has been treated in a very logical and coherent way, as can be seen from the division of the book into chapters and their sequence. The book, as a text, scores a point over other texts on this subject, in having a full chapter on morpho-tectonics and in giving a reasonably thorough qualitative coverage of the physics of deformation. The book is full of illustrations and examples of structures from all over the world. However, the quality of reproduction of some of the photographs could have been improved. The author has somehow preferred the footnote reference system though it is inconsistent with the system followed nowadays.

In so far as the book makes a thorough coverage of the fundamental topics of structural geology, with adequate attention to illustrations and definitions, I am confident that it would be one of the best text-books available both for the undergraduate and the postgraduate students.

C. NAGANNA

THE RATIONAL USE OF DYES IN BIOLOGY by Edward Gurr (Leonard Hill Books Ltd, London), 1965. Pp. xii+422. Price 105s.

This book is a companion volume to Dr Gurr's previous work, *Staining animal tissues, practical and theoretical*. The title is appropriate since most often the use of dyes by biologists tends to be quite irrational. Dr Gurr has eminently succeeded in bringing home the point that synthetic dyes are merely agents for imparting colour but are also highly reactive chemical substances. To encourage the rational, as opposed to the empirical, use of dyes, it is necessary to have some knowledge of the principal physical and chemical characteristics of the dyes used. Since most biologists may not possess enough knowledge of the organic chemistry of dyes, this book contains a very valuable compendium of such information. After introducing the reader to the physical and chemical properties of the biologically useful dyes, the book deals with the practical applications of dyes and the experimental procedures which should be followed for getting good staining. Information on absorption maxima, molecular weights, solubility, structural formulae and pH of the important stains is given in the appendix. The methods of preparing the stains are also indicated in the appendix. Thus, this is a book which any research worker using dyes in his experimental programme would benefit by having.

M. S. SWAMINATHAN

METABOLISM OF STEROID HORMONES by R. I. Dorfman & F. Ungar (Academic Press Inc., New York), 1965. Pp. viii+716. Price \$ 32.00

The first crystalline steroid hormone, estrone, was isolated thirty-six years ago, in 1929, at a time when

the structure of the basic steroid nucleus was unknown. Nobody could have perhaps predicted at that time that the field of steroids would undergo such an expansion as to constitute a single major field of activity broad enough to retain the attention of a sizeable section of organic chemists, biochemists, physiologists and endocrinologists, as well as the medical students of several disciplines throughout the world. The literature in steroids has grown to such an extent that it is not even possible for a single worker working in the field to keep track of all the facets of different developments in steroids. Further, with the advent of techniques such as gas liquid chromatography and thin-layer chromatography, new steroids are being isolated almost every day. From urine alone some 200 steroids have been isolated so far. In this background a novice feels quite lost, unless a more effective documentation procedure is evolved. The volume under review will fulfil this need to a great extent.

The magnitude of the task in compiling a volume of steroid metabolism can be appreciated from the fact that some 2000 primary references had to be screened before selecting the data for presentation. The monograph has covered all pertinent literature from early 1934 through the later part of 1964. Mere collection of this information and logical organization of this massive data in eight chapters constitutes a stupendous achievement.

Chapter I deals with introductory material and a brief but lucid treatment of nomenclature.

Chapters II and III give a very concise account of the isolation of steroids from natural sources as well as their biosynthetic relationships. Most of the steps involved in these biosynthetic relations are presented in a series of tables. The information in these tables has been translated into figures with many structural formulae which present the best available and most lucid schemes of biosynthesis.

Chapter IV dealing with the microbiological transformations of steroids and Chapter VI pertaining to enzymes and mechanism of reactions have been presented very elegantly with many examples. For convenience, the steroidal enzymes have been classified into different categories and each type has been discussed in detail giving plausible mechanisms for enzymatic reactions, wherever possible.

Chapters V and VII cover catabolic reactions of the steroids and formulate a system of steroid metabolism. Although the authors have dealt with these topics with great care giving much informative material in the form of tables and figures, it would have been of greater assistance if these outlines had been more completely supplemented with textual material. The reader may get somewhat confused by the tremendous array of various types of tables and figures.

In Chapter VIII the relationships between tissue steroids and metabolites in blood and urine have been covered very systematically and a considerable effort has been made in relating the steroid hormones formed in the tissues with their corresponding metabolites in blood and urine.

The monograph concludes with a brief but critical chapter on steroid hormone production rates.

Although the volume supplies an excellent review of the various aspects of steroid metabolism, it is certainly not intended as a text-book but is welcome as a very informative handbook. More information on individual topics can be obtained from reference to the original literature.

A few typographical errors have crept in, especially in figures where formulae are given. Also, the authors could have shown, at least in the introductory chapter, the existence of aldosterone and 18-hydroxy corticoids in tautomeric forms as hemiacetals and hemiketals respectively.

The book is highly recommended as an acceptable and agreeable reference book which will bring the workers in the steroid field up to date in a reasonably objective fashion. The reviewers are in complete agreement with the statement made by the authors in the preface that this edition will be very useful for the clinician, endocrinologist, gynaecologist, obstetrician, pediatrician and others who may desire a firm background in steroid biochemistry.

P. K. BHATTACHARYYA & PREMA B. RAMAN

ANIMAL GAMETES (MALE): A MORPHOLOGICAL AND CYTOCHEMICAL ACCOUNT OF SPERMATOGENESIS by Vishwa Nath (Asia Publishing House, Bombay), 1965. Pp. xvi+162 Plates 184. Price Rs 50.00

In the current scientific interest in the reproductive processes and their control, studies on gametogenesis and gametes occupy an outstanding position. Dr Vishwa Nath's pioneering researches on animal gametes at the Panjab University are indeed a landmark in the history of zoology in this country. It is, therefore, in the fitness of things that he should record his long sustained experience of research in this area in the compass of a monograph. This is what he has done primarily, but most judiciously he has attempted to do so over an integrated scaffolding of the present-day knowledge on cytology, cytochemistry and electron microscopy of gametogenesis and gametes.

The panorama of spermatozoan organization unfolds with a detailed consideration of the mammalian form and proceeds in the phylogenetic sequence to the invertebrates with their breath-taking nuances. The form and function of spermatozoa, their structure and chemistry have been dealt with meticulously, and viewed through the mental telescope of a mature biologist. The impact on the reader's mind is most pleasant and profitable.

It is, however, felt that the section on mammalian spermatozoa could have been enriched further by a reference to the recent significant studies on the chemistry of acrosome, maturation process in the epididymis, and the phenomenon of 'capacitation' in certain species. A brief mention about the immunochemical make-up of the spermatozoa, the nature of their association with the Sertoli cells, and the enzymatic mechanism of the spermiation process would have been pertinent.

The monograph is well presented and the illustrations are superb. To all those interested in the study of spermatogenesis and spermatozoa this excellent monograph will be most valuable.

Dr Vishwa Nath deserves our commendation and gratitude for this timely publication.

A. B. KAR

PUBLICATIONS RECEIVED

ENERGY, ITS PRODUCTION, CONVERSION AND USE IN THE SERVICE OF MAN by Philip Scorn (Pergamon Press Ltd, Oxford), 1966. Pp. xiv+69. Price 7s. 6d.

LIBRARIES IN THE MODERN WORLD by G. Chandler (Pergamon Press Ltd, Oxford), 1965. Pp. vii+164. Price 17s. 6d.

HIGH SPEED PULSE TECHNOLOGY: Vol. I — CAPACITOR DISCHARGES MAGNETOHYDRODYNAMICS-X-RAYS-ULTRASONICS by Frank Früngel (Academic Press Inc., New York), 1965. Pp. xiii+620. Price \$ 24.00

THE BIOCHEMISTRY OF ANIMAL DEVELOPMENT: Vol. I — DESCRIPTIVE BIOCHEMISTRY OF ANIMAL DEVELOPMENT edited by Rudolf Weber (Academic Press Inc., New York), 1965. Pp. xiii+648. Price \$ 23.00

INTRODUCTION TO QUANTITATIVE ULTRAMICRO-ANALYSIS by I. M. Korenman (Academic Press Inc., New York), 1965. Pp. ix+234. Price \$ 9.50

PROGRESS IN NUCLEIC ACID RESEARCH AND MOLECULAR BIOLOGY: Vol. 3, edited by J. N. Davidson & Waldo E. Cohn (Academic Press Inc., New York), 1965. Pp. xiv+363. Price \$ 11.50

HANDBOOK OF MICROWAVE FERRITE MATERIALS edited by Wilhelm H. von Anlock (Academic Press Inc., New York), 1965. Pp. xxiv+518. Price \$ 12.00

QUANTUM MECHANICS AND PATH INTEGRALS by R. P. Feynman & A. R. Hibbs (McGraw-Hill Book Co. Inc., New York), 1965. Pp. xiv+365. Price \$ 12.50

CHEMICAL STUDY OF SOME INDIAN ARCHAEOLOGICAL ANTIQUITIES by Satya Prakash & N. R. Rawat (Asia Publishing House, Bombay), 1965. Pp. 85. Price Rs 6.00

VACUUM SCIENCE AND ENGINEERING: PROPERTIES OF GASES AT LOW PRESSURE; VACUUM MEASUREMENTS; DESIGN AND OPERATING FEATURES OF VACUUM PUMPS AND SYSTEMS by C. M. Van Atta (McGraw-Hill Book Co. Inc., New York), 1965. Pp. xiv+459. Price \$ 15.00

AN INTRODUCTION TO ELECTRONIC COMPUTERS by Gordon B. Davis (McGraw-Hill Book Co. Inc., New York), 1965. Pp. 541

NEW LIGHT ON SPACE AND TIME by Dewey B. Larson (North Pacific Publishers, Portland), 1965. Pp. 264. Price \$ 6.00

DIGITAL COMPUTERS DESIGN FUNDAMENTALS by Yaohan Chu (McGraw-Hill Book Co. Inc., New York), 1962. Pp. xiv+481

FISHERY BYPRODUCTS by Julius Brody (AVI Publishing Co. Inc., Westport, Connecticut), 1965. Pp. xiii+232. Price \$ 12.50 (US); \$ 13.50 (foreign)

PRINCIPLES OF PHYSICS by F. Bueche (McGraw-Hill Book Co. Inc., New York), 1965. Pp. xiv+660

Pion with spin (spion)

Recent observations made at the Istituto Nazionale di Fisica Nucleare, sezione di Torino and Istituto di Fisica dell' Università di Torino, Italy, with a view to finding the validity of a recent hypothesis postulating a pion with a spin have failed to find evidence in favour of the hypothesis. The existence of a pion with a spin (spion) was postulated by Cvijanovich *et al.* [Cvijanovich, G. B., Jeannot, E. R. & Sudarshan, E. C. G., *Phys. Rev. Lett.*, **14** (1965), 117] in their studies of $K_S^0 \rightarrow \pi^+ + \pi^-$ decay since the existence of such a spion could explain a long-standing asymmetry in the $\pi \rightarrow \mu$ decay angular distribution for the lower part of the π^+ spectrum in τ decay. Two consequences that follow the postulation of the spion are: (i) the ratio of the decay rates for electronic and muonic modes should be comparable for the charged spion, and (ii) if the spin of the spion is 1 and neutral spions exist, the preferred decay mode would be into an electron-positron pair plus a photon. Assuming a very short lifetime ($\leq 10^{-12}$ sec.) one expects to observe in a bubble chamber an anomalous number of Dalitz pairs in K^+ decays. The validity of the hypothesis was checked by searching for evidence according to the above two consequences predicted by the hypothesis. Consequence (i) above was checked in τ and τ' decay and consequence (ii) in τ' , $K_{\mu 3}$ and K^+ decays involving a π^0 . No evidence substantiating the above two consequences could be found and all the results observed were compatible with a normal behaviour of the pions from K^+ decays [*Phys. Rev. Lett.*, **14** (1965), 761].

Plasma arc spraying

Coatings with layers only a few thousandths of an inch thick can be sprayed quickly and conveniently on surfaces of plastics, wood, ceramics and metals with 'plasma arc spraying', a technique recently rediscovered at Bell Telephone Laboratories. The technique employs a plasma arc spray gun consisting of a water-cooled tungsten cathode, a copper anode and a copper nozzle. To

NOTES & NEWS

start the plasma, a suitable gas or mixture of gases is fed into the gun, where a high frequency spark initiates ionization; the d.c. arc sustains the plasma. The net effect of the electric field is to increase the velocity of the electrons which by a collision mechanism transfers the energy to the ions which are speeded up. High ion velocity produces correspondingly high temperature, the arc space often reaching to higher than 15000°C. The material to be sprayed is introduced into the plasma near the nozzle as a powder. The gun propels the gas at a few pounds per square inch through the arc, where it violently expands, catches the powder introduced downstream and propels it at high speeds. The powder provides the surface necessary for recombination of ionized gas molecules and electrons and in the process takes the enormous energy released and gets molten. The rapidly expanding gas stream then carries the molten particles to the target where they coalesce and form a coating.

The coating may be formed either by chemical bonding between the surface and the hot particles or by mechanical bonds formed by the interlocking of the molten particles at the anchor points on the surface produced by grit blasting and other preparative methods.

The parameters critical in this operation are substrate temperature, power of the gun and particle size. It is now possible to spray powders 0.5-60 microns in diameter [*Mech. Engng.*, **87** (1965) (No. 10), 52].

1,4-Dihydropyridine

Although several N-alkyl or N-aryl-substituted dihydropyridines are known, none of the five isomeric unsubstituted dihydropyridines has ever been reported. Recently, the preparation of 1,4-dihydropyridine has been achieved. In the actual synthesis 1-trimethylsilyl-1,4-dihydropyri-

dine (5 ml.) was treated with methanol (1.25 ml.) containing 0.1 per cent potassium hydroxide. The product obtained immediately was separated into fractions by vacuum transfer at -20° to 30°C . The distillate was collected in liquid nitrogen-cooled traps, approximately 0.5 ml. of 1,4-dihydropyridine being collected.

The dihydropyridine is a colourless viscous liquid and contains about 5 per cent impurities as revealed by VPC analysis. That the compound is 1,4-dihydropyridine is supported by ultraviolet, infrared and NMR spectra and elemental analysis. Dihydropyridine solutions in deuteriobenzene and cyclohexane are stable for several weeks when air is excluded, but the neat material solidifies and turns orange rapidly [*J. Amer. chem. Soc.*, **87** (1965), 3283].

A novel acetylation

Boron-catalysed acetylation of 9-chloro-1-decalone has been shown to give, for the first time, two isomeric 6-methyl-*m*-dioxenones instead of the expected 2-acetyl-9-chloro-1-decalone. The two isomeric *m*-dioxenones have been found to incorporate, without rearrangement, all of the atoms of the starting material. That the compounds are not enol acetate of 2-acetyl-9-chloro-1-decalone is shown by the stability of the compounds towards acid hydrolysis and non-resemblance of the infrared spectra with the enol acetate prepared from 2-acetyl-1-decalone.

NMR spectra of the two isomeric compounds are similar to that observed for 2-methoxy-6-methyl-4-pyrone and 4-methoxy-6-methyl-2-pyrone. A mass spectrum showed major peaks at 150 and 68 mass units consistent with the presence of an octalone and a methacryloyl or crotonoyl unit. Treatment with 2,4-DNP gave after several hours the 2,4-DNP of Δ^9 -octal-1-one. A series of reactions confirmed the similarity of the compounds with another dioxenone derivative reported earlier by Carrol and Bader [*J. Amer.*

chem. Soc., **95** (1953), 5400]. The formation of the two isomeric 6-methyl-*m*-dioxenones is shown to result from formation of *gem*-diacetate and subsequent cyclization. That 1-decalone did not show the behaviour may be due to the inductometric effect of the chlorine. This is the first example of an acid-catalysed acylation furnishing *meta*-dioxenones [*J. chem. Soc.*, (1965), 4456].

Memory fixation in goldfish

It has been commonly suggested in recent years that nucleic acids or proteins may be connected with learning and memory. Memory can be disrupted by various physical or chemical agents administered shortly after training, but within minutes to hours it becomes insusceptible to these agents. Correlations between changes in brain metabolism and deficits in fixed memory produced by various agents may provide an insight into the biochemical basis of memory fixation.

Puromycin, an antibiotic inhibiting protein synthesis, blocks memory in mice [Flexner, J. B., Glexner, L. B. & Stellar, E., *Science*, **145** (1963), 57] and goldfish [Agranoff, B. W. & Klinger, P. D., *Science*, **146** (1964), 95] when given after training. Further investigations on memory fixation in goldfish have been carried out by B. W. Agranoff, R. E. Davis and J. J. Brink [*Proc. nat. Acad. Sci., Wash.*, **54** (1965) 788] by measuring the memory deficits obtained by injecting different amounts of puromycin at different times after training. The training procedures were as follows: Goldfish were placed in individual shuttle boxes in which light was paired with repetitive electrical shock. To avoid the shock, fish had to swim over the hurdle from the light to the dark end of the box. A correct response was scored when the fish swam over the hurdle before the onset of the shock. All fish were given 20 trials on the first day of an experiment and 10 trials on the fourth day. Puromycin dihydrochloride in 10 μ l. of saline was injected into the cranial cavity over the tectum at specified times on the first day. Responses were scored by direct observation.

Memory was inferred from an increase in correct responses between blocks of trials.

The results obtained indicate that the memory forming during the 20 trials on the first day is different from the memory detected on the fourth day. Memory on the first day is apparently not disrupted by puromycin. Memory on the fourth day, on the other hand, can be completely or partially blocked by administering puromycin at different times during the hour following trial 20 on the first day. The change in susceptibility suggests that memory during the trials on the first day is temporary or 'short term', and that it becomes fixed into 'long-term' memory detected on the fourth day. The insusceptibility of the short-term memory to puromycin indicates that the drug acts specifically on the fixation process, in which long-term memory is formed. The magnitude of the deficit in 'long-term' memory of the response varies with the amount of puromycin injected; 170 μ g. effectively obliterates the long-term memory.

The concept of short- and long-term memory arose primarily from studies on learning in man. It has been proposed that short-term memory, like the initial input, is bioelectrical. The above findings about the goldfish are compatible with this premise and indicate that long-term memory is chemical. Thus, these studies indicate that puromycin specifically disrupts the formation but not the maintenance of long-term memory, while the temporary or 'short-term' memory is not susceptible to the action of puromycin.—
B. S. ACHAR

Active centre of phosphoglucomutase

The structure and function relationship of macromolecules has been a fascinating subject in biological chemistry. Many investigators have been interested in the study of catalytic sites of various functional macromolecules like phosphoglucomutase (PGM). The mechanism of action of this enzyme which catalyses the reversible conversion of glucose-1-phosphate (G-1-P) to glucose-6-phosphate (G-6-P) has been elucidated by

Najjar and Pullman. The enzyme contains a single phosphate group per molecule of the enzyme and can be reversibly transferred to the substrate. Phosphoglucomutase is particularly suited for studies on the active centre because of the ease with which radioactive ^{32}P can be incorporated into the enzyme molecule.

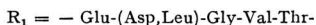
The presence of phosphate group in the enzyme as well as its exchangeability with phosphates in the substrates also has been demonstrated. The phosphate group is not cleaved even under strong acid conditions and serine phosphate (SerP) could be isolated. This prompted studies on the amino acid sequence around this point of the molecule. Koshland and Erwin have reported the sequence around this point to be Asp-SerP-Gly-Glu-Ala-Val- and observed similarity to the active site of chymotrypsin.

Recently, G. Milstein and F. Sanger [*Biochem. J.*, **79** (1961), 456] reported a sequence at the active site of the molecule which does not agree with the sequence proposed by Koshland and Erwin. Milstein and Sanger have obtained Thr-Ala-SerP-His-Asp- as the sequence at the active centre. Yet another group [Harshman, S. & Najjar, V. A., *Fed. Proc.*, **21** (1962), 233] has obtained a different sequence, viz. Asp-Gly-Glu-SerP-Ala-Gly. More recent studies of S. Harshman and V. A. Najjar [*Biochemistry*, **4** (1965), 2526] have indicated that the catalytic site of PGM displays two active serine residues cooperatively involved in phosphate transfer to the substrate. These authors have confirmed the presence of two different phosphoserine peptides proposed by Milstein and Sanger and themselves. However, they could not obtain evidence to support the Koshland and Erwin's sequence.

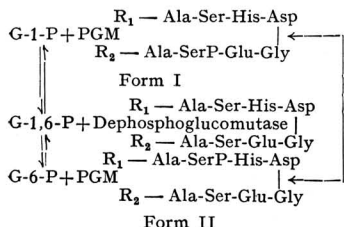
These authors have found evidence for the presence of two active serine residues at the catalytic site, only one or the other carrying the phosphoryl group and proposed that the active site may exist in one of the following forms:

- (I) $\text{R}_1 - \text{Ala-Ser-His-Asp-Gly-Glu-SerP-Ala-R}_2$
 (II) $\text{R}_1 - \text{Ala-SerP-His-Asp-Gly-Glu-Ser-Ala-R}_2$

where



They have represented the mechanism of catalysis which involves both the serine residues as follows:



In this mechanism, it is important that form II of the enzyme is converted back to form I. No preferential fit of phosphate on either serine residue is obvious.—
L. V. S. SASTRY

Codons for termination and release of polypeptide chains from ribosomes

The intriguing puzzle regarding the mechanism of release of completed polypeptide chains from ribosomes has elicited a spate of speculations, particularly after the description of polycistronic messenger RNA (mRNA). When bacteriophage f2 RNA was introduced into the cell-free system of *Esch. coli*, as a source of mRNA, the synthesized protein was released into the supernatant, after a short time lag. In contrast, with synthetic polyribonucleotides as mRNA the peptide chains were firmly attached to the ribosomes and were bound with tenacity to the 50s subunit of the ribosomes even after the 30s subunits were dissociated from them. However, addition of puromycin or ribonuclease digests of aminoacyl-s-RNA resulted in the release of polypeptides from the ribosomes. This evoked the suggestion that synthetic polynucleotides in the above-mentioned experiments probably lacked specific nucleotide sequences, indicating termination of message and the release of polypeptide chains.

In their interesting experiments, M. Takanami and Yonhon Yan

[*Proc. nat. Acad. Sci., Wash., 54* (1965), 1451] have investigated the polypeptide releasing activity of the various synthetic polyribonucleotides. Using polyribonucleotides of a variety of base compositions and ratios they found that maximum amount of protein release (as indicated by the ¹⁴C activity) to the supernatant was observed with UA, UAI, UAC and UACI polyribonucleotides, while with other polyribonucleotides of a larger number, very little protein was detected in the supernatant. Contrary to previous suggestions, the presence of methylated bases did not increase the radioactivity in the supernatant, showing they do not specify termination of peptide chains. Thus Takanami and Yan conclude that polynucleotides with U and A must contain some code message indicative of termination and release of peptide chains.

Extending their studies further, the authors noted that in copolymers containing U and A, increasing the proportion of A to U resulted in an increase of radioactivity in the supernatant. Possibly a 2AIU codon is the codon for termination. Also, poly UAI was more effective in releasing radioactivity to the supernatant than what was expected from its calculated abundance of 2AIU triplets, and was more effective in this respect than poly UAC which had the same content of U and A as poly UAI. AI and UI copolymers were ineffective. The above facts clearly indicate that poly UAI is more effective since it contains another termination codon, viz. UAI (IU, IA, II). This is in agreement with the fact that in several other polynucleotides like poly CUAI, poly UAI, the chain releasing potencies were identical when the calculated content of UAA plus UAI (sequence is not specified) was the same.

M. Nirenberg, P. Leder, M. Bernfield, R. Brimacombe, J. Trupin, F. Rottman and C. O'Neal [*Proc. nat. Acad. Sci., Wash., 53* (1965), 1161] have reported that trinucleotides UAA, UAG and UGA had no activity in binding aminoacyl-s-RNA's to ribosomes and that these may be 'nonsense' triplets. This is in remarkable agreement with the

above findings that UAA and UAI (I is treated as equivalent to G) have high chain terminating activity.

The mechanism of recognition of chain terminating triplets is still not fully understood. A suggestion by S. Brenner, A. O. W. Stretton and S. Kaplan [*Nature, Lond., 206* (1965), 994] is that an RNA molecule similar to s-RNA recognizes the chain terminating triplets and releases the polypeptide chain. This is amply supported by the findings that the polypeptide chain is attached to ribosomes solely by the s-RNA binding site of ribosomes.—S. MUTHU KRISHNAN

RNA-dependent RNA-polymerase

The existence of DNA polymerase and DNA-dependent RNA-polymerases led to the question of information transfer in organisms with an RNA genome as in most of the RNA viruses. Almost all the bacteriophages, except a few, contain single stranded RNA as their genome. R. H. Døi and S. Spiegelman [*Science, 138* (1962), 1270] using *Esch. coli* Hfr strain (K₁₆ and K₁₀) infected with f2 bacteriophage, showed that DNA is not used as an informed intermediary in the synthesis of virus particles in the host organism during infection. This led to the working hypothesis of the existence of a specific enzyme exhibiting RNA polymerizing activity with a unique dependence on viral RNA as template. Later, I. Haruna, K. Nozu, Y. Ohtaka and S. Spiegelman [*Proc. nat. Acad. Sci., Wash., 50* (1963), 905] isolated bacteriophage from *Esch. coli* organisms infected with MS φ2 bacteriophage an enzyme, RNA-dependent RNA-polymerase (also called replicase), showing a specific requirement of a homologous RNA of the virus as template for activity. It was also shown by the same authors that the newly synthesized RNA was identical to the template RNA on comparison of the nearest neighbour sequence studies. Hence, for the first time, the existence of RNA-dependent RNA-polymerase was shown which ignores the pre-existing cellular RNA, obviously conferring an

advantage on the virus for multiplication during infection.

However, in all such studies of replicating mechanisms it is necessary to show, in addition to the nearest neighbour sequence analysis, that the newly synthesized RNA preserves the message for the production of viral particles possessing the same effective infectivity as that of the template RNA particle. In such type of experimentation, it is essential to show that the enzyme used, RNA-dependent RNA-polymerase, is free from RNases, DNA-dependent RNA-polymerase activity and DNase, since any such contamination, however trivial chemically, may greatly hamper the biological assay. Secondly, the enzyme should also show a total dependence on added RNA for activity with a low level of saturation. By employing such an enzyme system, obtained from a mutant Hfr strain of *Esch. coli* (Q 13) infected with a bacterial virus QB, S. Spiegelman, I. Haruna, I. B. Holland, G. Beaudreau and D. Mills [*Proc. nat. Acad. Sci., Wash.*, **54** (1965), 919] have shown that the replicase synthesized new RNA using viral RNA as template and that the newly synthesized RNA has the same infectivity as that of the template RNA. To avoid the ambiguity that the observed increased infectivity may be due to the activation of the input template RNA, the authors used a serial dilution method. Even after fifteen transfers when the input RNA is diluted to a very great extent, there is a seventy-five-fold increased RNA synthesis with a corresponding increase in the total infectivity. It has also been confirmed by a unique serological reaction that the newly synthesized RNA after fifteen transfers resembled the template RNA. Hence for the first time, a simple system with required ions and heterologous nucleotide triphosphates with a template RNA is provided, opening the door for the study of the exact mechanism of replication of single stranded viral RNA.—A. S. PERUMAL

Linkages in glycoproteins

A considerable amount of information has become available

in recent years, on the nature of linkage of the carbohydrate prosthetic groups with the polypeptide in glycoproteins. The types of covalent bonds that link the carbohydrates to polypeptides in naturally occurring polymers are glycosylamine bonds and O-glycosidic ester bonds. The former type of bond which is comparatively stable to acid and alkali is found to occur in a wide variety of glycoproteins. A definite proof for the presence of such a bond in hen ovalbumin has been provided by A. Neuberger and R. D. Marshall [*Biochemistry*, **3** (1964), 1596]. The linkage involves the amide group of asparagine and the reducing group of N-acetylglucosamine.

The linkage between carbohydrate and protein in a salivary glycoprotein or mucin from ovine and bovine submaxillary glands is that of the glycosidic type. Ovine submaxillary glycoprotein (OSM) is composed of about 800 α -D-N-acetylneuraminy-N-acetylglucosamine disaccharide units joined at intervals covalently to a polypeptide chain. Studies of A. Gottschalk [*Ann. N.Y. Acad. Sci.*, **106** (1963), 168] on OSM and on similar bovine submaxillary glycoprotein (BSM) suggested that most of these prosthetic disaccharide units were linked to polypeptides by ester bonds involving the reducing hydroxyl groups of N-acetylgalactosamine and the ω -carboxyl group of dicarboxylic acids. The kinetics of the reaction of OSM with dilute alkali containing NH_4OH , under carefully controlled conditions, did not, however, support this suggestion. Moreover, after digestion of BSM and OSM with proteolytic enzymes, a glycopeptide fraction was isolated which contained galactosamine, dicarboxylic acids and hydroxy acids in the molar ratio of 1.0:0.1:1.2. This led to the suggestion that at least a part of the hexosamine in these glycoproteins is linked by glycosidic bonds involving the β -hydroxy groups of serine or threonine.

The involvement of serine or threonine residues in the glycosidic ester linkage was, however, confirmed independently by V. P. Bhavanandan, E. Buddecke, R. Cambelli and A. Gottschalk [*Biochem. biophys. Res. Commun.*, **16** (1964), 353], K. Tanaka, M.

Bartolini and W. Pigman [*Biochem. biophys. Res. Commun.*, **16** (1964), 404] and S. Harbon, G. Herman, B. Rossignol, P. Jolles and H. Clanser [*Biochem. biophys. Res. Commun.*, **17** (1964), 57]. The β -elimination of O-substituent readily occurs when O-substituted seryl or threonyl derivatives in proteins are treated with dilute alkali at room temperature. Treatment of OSM and BSM with dilute alkali or with dilute alkali containing borohydride released the carbohydrate components and effected extensive destruction of the hydroxy amino acids in the protein suggesting the presence of such a glycosidic linkage in OSM and BSM and also the involvement of β -elimination reaction.

J. S. Best, V. P. Bhavanandan and A. Gottschalk [*Biochim. biophys. Acta*, **101** (1965), 141] tried to find out whether or not in addition to the O-glycosidic linkages to serine and threonine known to be present, another type of alkali-labile linkage, in particular a glycosidic-ester linkage, occurs in the OSM preparation from German sheep. In their first set of experiments it was found that the release of 35.2 per cent of N-acetylgalactosamine, when OSM was kept at pH 8.0 and 42°C. for 12 hr was matched by the loss of an equimolar amount of serine and threonine without a significant change in the other amino acids. In the second set of experiments it was shown that on treating OSM with 0.1N sodium hydroxide at 4° the decrease of bound hexosamine with time was linear. Finally, in the third set of experiments glycopeptides prepared from OSM by trypsin or pronase digestion were treated with LiBH_4 in tetrahydrofuran and the dicarboxylic acid content of the protein moiety was determined before and after this treatment. No significant difference was observed after 6 hr LiBH_4 treatment. Only traces of homoserine and α -amino- δ -hydroxy-*n*-valeric acid were detectable after LiBH_4 reduction.

The LiBH_4 treatment of trypsinized OSM prepared from Australian sheep resulted in the loss of 35 per cent of the dicarboxylic acid content, whereas similar treatment of OSM prepared from

German sheep resulted in insignificant loss of dicarboxylic acid. The results obtained with Australian OSM and European OSM are consistent in themselves, but differ from each other. The suggestion is made that the difference in age of the sheep used for the material may be a contributing factor, because in Australia lambs (4 months) are slaughtered and in Germany adult sheep (4 years) are killed.—M. K. C. SRIDHAR

Binding of amino acids and peptides to gelatin

Amino acids and short peptides occur as physically associated non-protein nitrogen (NPN) in highly purified acetic acid-soluble collagenous proteins. The NPN fraction influences the chemical determination of the average chain weight (C_n value) of collagenous proteins and the removal of NPN by acetone precipitation from a good quality gelatin has resulted in an increase in the C_n value from 50,000 to 100,000, which is consistent with the weight-average molecular weight of gelatin.

A simple ion-exchange technique employing Amberlite CG-50 for the separation of NPN fraction from commercial gelatin has been reported [*Biochim. biophys. Acta*, **104** (1965), 189]. NPN is selectively eluted from the protein bound to the resin with 1M acetic acid; the protein and the resin remain irreversibly associated under these conditions. The nitrogen content of the eluates is determined by micro-Kjeldahl analysis. A plot of total nitrogen eluted against the equilibration time of the protein with the resin gives a constant minimum level of nitrogen approx. 1.7 per cent of the total nitrogen after 8-10 hr, which is in good agreement with the percentage NPN found in other collagenous proteins.

The time lag in the binding of the protein to Amberlite CG-50 suggests a non-ionic bonding between the two, as this requires time to orientate the polypeptide chain in such a manner that they are firmly bound to the resin particles. A quick release of peptides and acids from the protein and the resin in acetic acid indicates a purely ionic interaction.

Preparation of specifically labelled milk proteins

A simple method for preparing specifically labelled milk proteins, using cultures *in vitro* of bovine mammary secretory cells, has been reported from the Department of Dairy Sciences, University of Illinois. The method, which involves the addition of skim milk to the culture flasks after an incubation period with the labelled precursor, followed by the isolation of the labelled protein from the milk-medium mixture, can be used for preparing milk proteins of high specific activity on a large scale without encountering the problems of large precursor pools and the waste isotope disposal problems inherent in the *in vivo* methods.

Bovine mammary cell cultures, prepared by placing the dispersed mammary tissue from newly lactating dairy cow in a cultivation medium, are incubated with labelled amino acids for 19 hr. Cultures harvested at the end of each isotope pulse period are centrifuged at 3000 g for 10 min. The media and cell homogenates obtained are stored at -10°C . if not analysed immediately.

For isolation of the labelled milk proteins, the medium is mixed with an equal volume of mixed herd skim milk which had been previously assayed immunologically for both β -lactoglobulin and α -lactalbumin. Crystalline β -lactoglobulin is isolated from the mixture by scaled down isolation procedure involving crystallization from the 43-80 per cent $(\text{NH}_4)_2\text{SO}_4$ fraction. Before preparation, the solutions are dialysed for 8-10 hr, first against a large excess of 0.1 per cent solution of the labelled L-leucine and then exhaustively against distilled water. The crystals are then lyophilized with a minimum of distilled water in a tared vial. Cellulose acetate strip electrophoresis and immunological assay have shown the crystals obtained to be of pure β -lactoglobulin.

For the isolation of α -lactalbumin, the supernatant solution from the β -lactoglobulin crystallization is adjusted to pH 2.0. α -Lactalbumin is estimated from the areas of protein spots developed by cellulose acetate strip

electrophoresis [*Biochim. biophys. Acta*, **104** (1965), 462].

Progress Reports

Ahmedabad Textile Industry's Research Association

The research and liaison work conducted by the research institute of the association during 1964-65 is presented in its annual report for the year (Pp. 56). At the end of the year the membership of the association stood at 113, with a total installed spindleage of 38,24,441 and 74,144 looms. A number of projects are in hand dealing with cottons of modified crystallinity. A study of tensile properties of cotton fibres containing formaldehyde cross-links established in the dry and wet states has shown a series of interesting power-law type relationships among bundle tensile strength, elongation at break, test length of the bundle and amount of bound formaldehyde.

A theory has been worked out and experimentally verified which shows that a twisted uniaxial filament behaves like a combination of an optical 'rotator' and retarder. A new apparatus designed for measuring fibre refractive indices over a range of wavelengths is based on 'fibre-refractometer' principle, incorporating a comparison between the transmitted and scattered light by a fibre pad immersed in a liquid and illuminated by parallel white light. A new type of self-resonating circuit has been evolved for measuring dynamic Young's modulus of cotton fibres at audio-frequencies.

On the basis of studies on oligosaccharides and methylated fragments obtained through enzymic degradation and methylation, many structural features of tamarind kernel polysaccharide have been established. It has been shown to be composed of a backbone of β -1,4-linked D-glucose residues from which subtend branches of L-arabinose, D-xylose, 2-O- β -D-galactopyranosyl-D-xylose and 4-O- β -D-galactopyranosyl-D-glucose.

A realistic model based on renewal theory for fibre assembly structures in sliver has been proposed. Fibre configuration in card sliver and its effect on the

technology of spinning have been investigated. At constant production rates, lighter slivers have been seen to result in a decrease in the incidence of major hoots and to lead to significantly better yarn quality, while the bulk at feed has very little effect on either of these characteristics.

A wide range of investigations have been carried out both on the nature of end breaks at ring frames and on the effect of various factors on the incidence of such breaks. It has been observed that the linear density of the yarn immediately at the point of break is almost invariably less than the average density at other points and that a break in the yarn is very often accompanied by a preceding heavier length of yarn (30-50 cm. of length).

Extensive trials have been conducted on the feasibility of mixing up to 15 per cent of indigenous viscose staple with different types of cottons. It has been observed that in the coarse and medium count range, for fabrics which are not mercerized, such blending can be generally done without any significant loss in tensile strength of yarn or fabrics, although wet strengths are slightly lower than for all-cotton fabrics.

A small cylinder drying range has been fabricated with necessary instrumentation for measuring steam quality.

Dr S. R. Valluri

Dr S. R. Valluri has been appointed Director, National Aeronautical Laboratory, Bangalore.

Dr Valluri (b. 25 June 1924) took degree in Mechanical and Electrical Engineering from the Banaras Hindu University in 1946, diploma in Aeronautics from the Indian Institute of Science, Bangalore, in 1948 and received the Douglas Pauley award for higher studies in the United States. He obtained Master's Degree and Ph.D. in Aeronautics from the California Institute of Technology in 1950 and 1954 respectively. He served in the California Institute of Technology for 10 years, specializing in the

field of material behaviour, including fracture, fatigue and creep. He was a consultant for the Missiles and Space Systems Division of the Douglas Aircraft Company, the United States Air Force, the Northrop Aircraft Corporation and others. Returning to India in 1964, he joined the Indian Institute of Technology, Madras, as Senior Professor and Head of the Department of Aeronautics and Applied Mechanics and continued in that post till he took up the present position.

Dr Valluri was awarded the Wright Brothers' medal for the year 1963 by the Society of Automotive Engineers for his outstanding achievements in the field of aeronautics. He is the first Indian to have received this medal. He was invited to present papers at international conferences on 'Fracture' held in the United States and Japan. Dr Valluri has to his credit a number of scientific research papers in the general field of fracture.

Dr Valluri is an Associate Fellow of the American Institute of Aeronautics and Member of the American Society of Metals, Society of Experimental Analysis, and Society of Sigma XI.

New Periodical

NPL Technical Bulletin

This bulletin, to be issued quarterly, is intended to highlight those aspects of the work of the National Physical Laboratory, New Delhi, which have a bearing on industry. The first number (January 1966, Pp. 12) carries write-ups on (1) the objectives, functions and main achievements of the laboratory, and (2) the first Open Day and Sir K. S. Memorial Lecture arranged on 21 January 1965. An illustrated semi-technical article describes the various stages in the reconditioning of mercury arc rectifiers. Also included are lists of lectures delivered in recent months by visiting scientists at NPL and by NPL scientists in other institutions. Papers published by scientific staff of NPL since January 1965 are listed.

Announcements

■ *Indian Science Congress Association* — Prof. T. R. Seshadri, FRS, has been elected to preside over the next session of the Indian Science Congress Association to be held during January 1967. The following have been elected presidents of the various sections: Prof. U. N. Singh, Baroda (*Mathematics*); Dr V. S. Huzurbazar, Poona (*Statistics*); Prof. F. C. Auluck, Delhi (*Physics*); Prof. R. C. Mehrotra, Jaipur (*Chemistry*); Dr R. L. Singh, Varanasi (*Geology and Geography*); Prof. R. N. Tandon, Allahabad (*Botany*); Prof. Sivatosh Mookerjee, Calcutta (*Zoology and Entomology*); Dr A. K. Mitra, Delhi (*Anthropology and Archaeology*); Prof. A. B. Chowdhury, Calcutta (*Medical and Veterinary Sciences*); Dr B. N. Sahu, Bhubaneswar (*Agricultural Sciences*); Dr S. R. Maitra, Calcutta (*Physiology*); Dr H. C. Ganguli, New Delhi (*Psychology and Educational Sciences*); and Prof. D. Banerjee, Howrah (*Engineering and Metallurgy*).

Prof. P. Maheshwari, FRS, has been elected General President for 1967-68.

■ *The Third Symposium on Earthquake Engineering*, sponsored by the University of Roorkee, will be held during November 1966. The discussion at the symposium will cover the following fields: Analysis of structural response and design of structures for earthquake forces; Design of dams and other appurtenant works in seismic zones; Soil and foundation behaviour during vibrations; Seismicity, wave propagation and ground motion; Instruments for earthquake engineering and seismological studies; Geological studies of tectonic features influencing occurrence of the earthquakes; Recent strong earthquakes and resultant damage; and Housing in seismic zones. Correspondence regarding the symposium should be addressed to Dr Jai Krishna, Professor and Director, School of Research and Training in Earthquake Engineering, University of Roorkee, Roorkee, UP, India.

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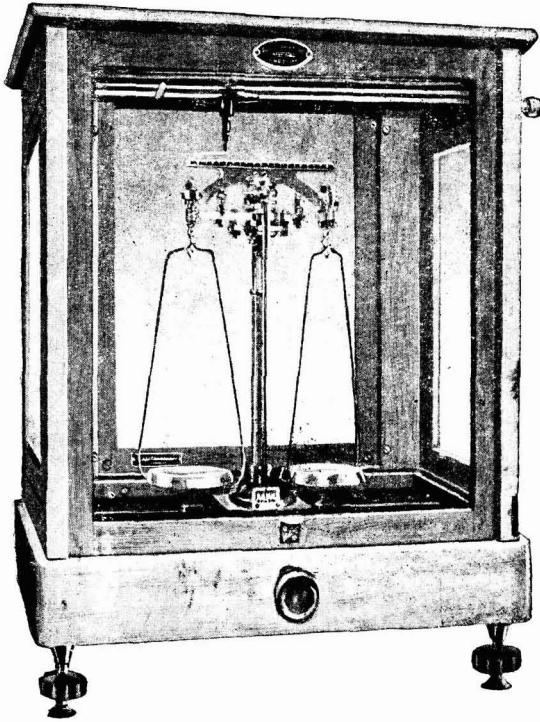
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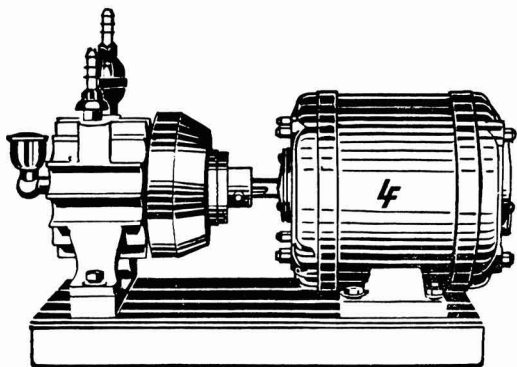
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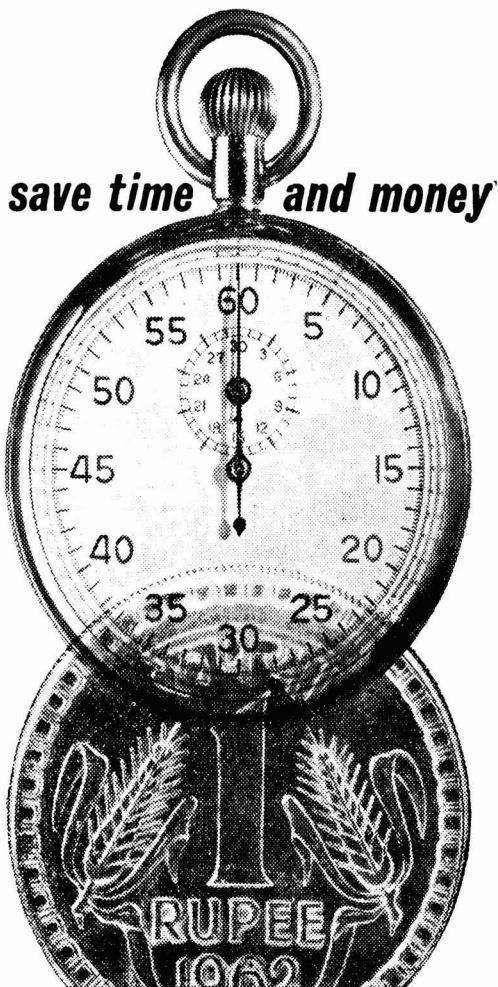
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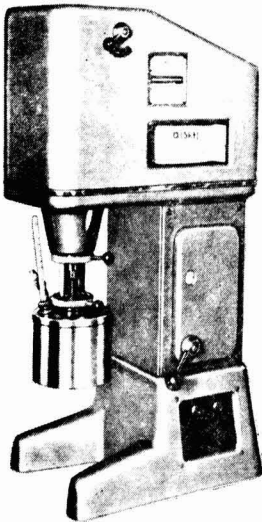
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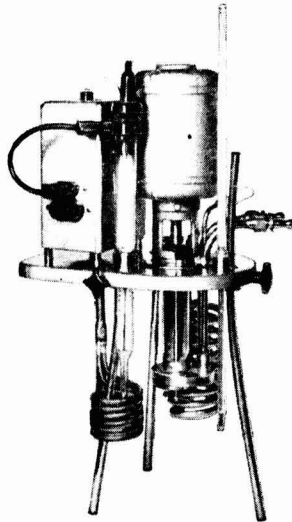
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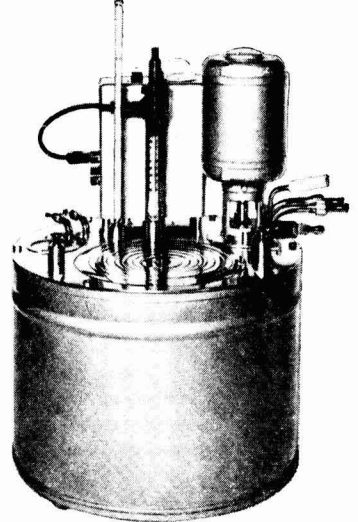
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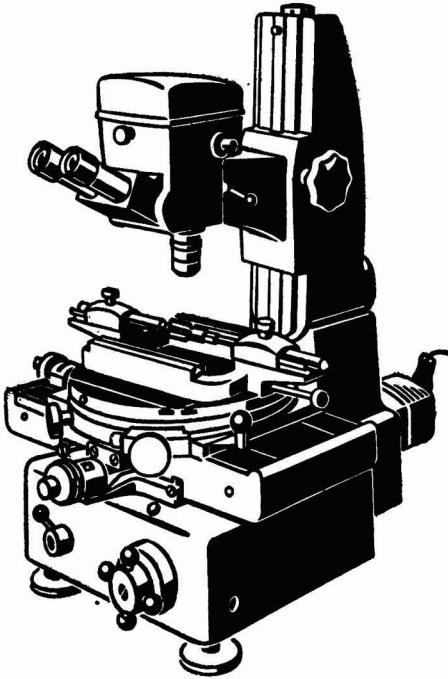
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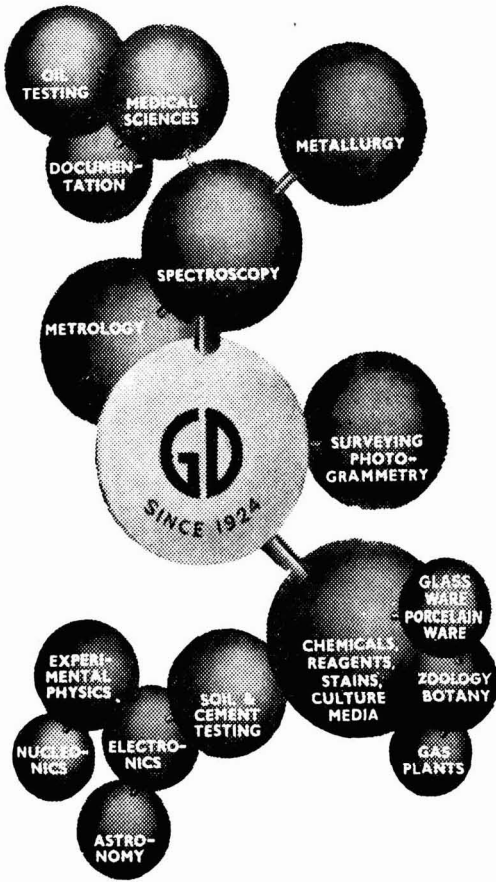
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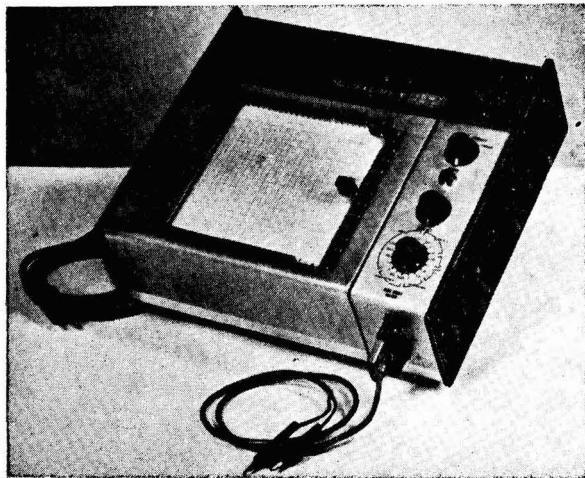
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