NATIONAL CHEMICAL LABORATORY

POONA

1950—1960

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COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH

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COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH

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DIRECTOR GENERAL

FOREWORD

The National Chemical Laboratory, Poona, is among the first in the chain of research laboratories set up by the Council of Scientific & Industrial Research. The Laboratory was declared open on January 3, 1950 and has over a decade of useful work to its credit. I am happy that a report on the scientific achievements of the Laboratory for the decade up to 1960 has been prepared.

The record will interest not only scientists pursuing research in the various disciplines covered by the Laboratory but also all connected with different chemical industries. Chemistry embraces a wide variety of subjects and techniques of research. I notice that a careful selection has been made of the work carried out in the Laboratory during the period under review and only projects which led to worthwhile results have been dealt with. The fact that nearly 500 research papers have been published and 95 patents have been taken is a measure of the contribution of the Laboratory to the growth of chemistry and chemical technology in the country. In the choice of projects, the Laboratory has been guided by two main considerations: the needs of the country, and the specialised knowledge, experience and interest of the scientists in the Laboratory.

Much credit goes to Professor J. W. McBain, F.R.S., and Professor G. I. Finch, F.R.S., the past Directors of the National Chemical, Laboratory for planning and organising the activities of the Laboratory in its early years. And I would like to take this opportunity of paying my meed of tribute to them and the present Director, Dr. K. Venkataraman, who, as head of this institution since 1957, has extended the scope and given further impetus to the research programme of the Laboratory. I am sure that, under his able guidance, the Laboratory will continue to forge ahead and play a significant role in the promotion of chemical technology and the growth of chemical industry in the country.

(M. S. Thacker)

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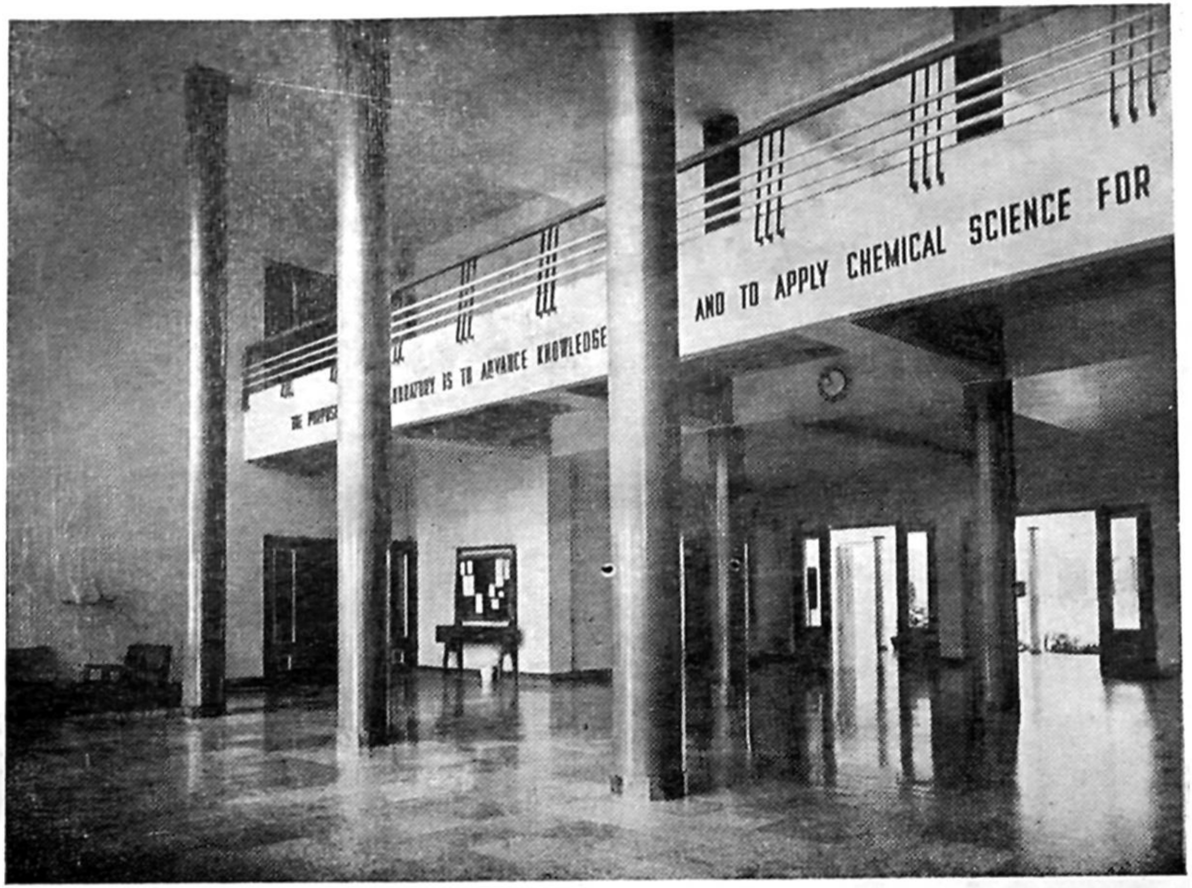


ENTRANCE HALL

REPORT OF THE
NATIONAL CHEMICAL LABORATORY
for the period 1950 - 1960

In September 1941 the late Sir Shanti Swarup Bhatnagar submitted proposals to the Government of India for the establishment of a National Physical Laboratory and a National Chemical Laboratory. Plans for the NCL were completed in 1945, and on the 6th April, 1947, the foundation-stone was laid by the late Shri B. G. Kher, Chief Minister of Bombay, on a breezy plateau five miles north of Poona between Government House and the village of Pashan. The Laboratory, which is situated in a campus of 475 acres and has a residential colony now providing accommodation for about a third of its staff, was declared open on the 3rd January, 1950, by Shri Jawaharlal Nehru, Prime Minister of India, being the first among the national laboratories which now number 25. The late Professor J. W. McBain was appointed the first Director in October 1949, and was followed by Professor G. I. Finch in September 1952. The present Director took charge in August 1957.

The purpose of the NCL, as inscribed in the entrance hall, is "to advance knowledge and to apply chemical science for the good of the people." Like its counterpart in physics, the National Physical Laboratory, the NCL is wide in its scope and seeks to embrace the whole field of chemical research, supplementing the work of the national laboratories which are concerned with specialized branches of chemical technology, such as leather, glass, salt, fuels, metals, food and drugs. There is no line of demarcation between fundamental and applied research, and the NCL endeavours to advance the frontiers of chemical knowledge, to make a scientific as distinct from an empirical approach to the solution of chemical problems with immediate or long-range practical objectives, and to undertake the more direct task of assisting the chemical industry in the investigation of technical problems. These broad aims are not easy to achieve; nevertheless it is generally recognized that the National Physical Laboratory and the NCL have to be charged with such responsibilities, and they remain the two bedrocks on which is built the national research structure of the Council of Scientific and Industrial Research.

The laboratories at present cover a floor area of about 200,000 sq. ft. and possess modern equipment for chemical research in all its aspects and for process development. For administrative and organizational convenience the NCL is divided into nine Divisions of Physical Chemistry, Inorganic Chemistry, Organic Chemistry, Essential Oils, Biochemistry, Polymer Chemistry, Organic Intermediates and Dyes, Chemical Engineering, and Survey and

Information. The Division of Survey and Information makes literature surveys, maintains data on raw materials, chemicals and equipment manufactured in India or abroad, assesses the commercial possibilities of products and processes developed in the NCL, deals with outside enquiries, and looks after visitors. Work on essential oils was initiated in 1951 as a part of the programme of the Division of Organic Chemistry; the results achieved have justified the creation of a separate Division which will shortly be housed in new laboratories. The Division of Organic Intermediates and Dyes is a recent addition, created on the recommendation of the Development Council for Drugs, Dyes and Intermediates; it has made a modest beginning in the laboratories of the Chemical Engineering Division, but a separate wing is under construction. In addition to the nine Divisions, the NCL has a well-equipped workshop and a glass-blowing section, in which many items of plant and apparatus are fabricated. A new pilot-plant building for the Chemical Engineering Division to house most of the heavy equipment for unit operations and a new workshop building were constructed in 1960 and were formally opened by Professor M. S. Thacker, Director General of Scientific and Industrial Research, on the 26th December 1960. An isolated building for the Radiation and Nuclear Chemistry Section of the Physical Chemistry Division comprising a floor area of 7,000 sq. ft. is under construction.

The choice of problems and projects undertaken by the NCL is guided mainly by two considerations: the country's needs of chemical products and the specialized knowledge, experience and interests of members of the staff. Data available from Government sources, especially the Development Wing of the Ministry of Commerce and Industry, recommendations of the national Five–Year Plans, and the industrial contacts of the staff, have been useful in formulating programmes of research on problems of potential technical importance; it is hoped that in future the Indian chemical industry will take a more direct and active interest and utilize to a much greater extent the facilities offered by the Laboratory. Schemes of research sponsored by industry are being undertaken in steadily increasing numbers. The contribution made by the NCL to chemistry and chemical technology in the first decade of its existence can be judged by over 500 papers which have appeared in Indian and foreign journals. On the technical side the work of the NCL has led to over 95 patents; a list of processes, which have been taken over by industry and are in various stages of commercial exploitation, is given in Appendix V. The progress of research in the NCL during 1950–1960 is briefly reviewed in the sequel, and an annual report will be published in future.

SURFACE AND COLLOID CHEMISTRY

Some properties of surface – active compounds

Far below saturation of a soap solution with solubilized benzene or toluene there is a zone of maximum turbidity caused by the formation of acid soap which is largely resorbed in the micelle before true saturation is reached. The phenomenon has been observed with potassium laurate, dodecylamine hydrochloride, and cetyl pyridinium chloride, all of which are hydrolyzable, but not with sodium lauryl sulphate, where hydrolysis is absent. This intermediate turbid region tends to be suppressed by all factors that reduce hydrolysis or solubilize the products of hydrolysis. These are high alkalinity in the case of ordinary soaps, high concentration of soap or detergent, and high temperature, where the amount of solubilization is greatly increased. A mechanism has been suggested which might cover the known facts.

Changes in volume with temperature were studied for a number of sodium laurate-water systems and sodium laurate-salt-water systems containing from 50 to 75 per cent by weight of soap. For some of these ternary systems, the volume-temperature curves exhibited discontinuities at 80–90°, which were attributed to the appearance of a homogeneous phase termed the 'kettle-wax'. The importance of this phase in determining various equilibria in soap boiling has been discussed.

The change in transference number with concentration of potassium laurate and lauryl sulphonic acid was measured at 0.5° with a Tiselius electrophoresis apparatus using concentration boundaries. The anion transference number increment was found to show a sharp rise when the concentration exceeded the critical concentration for micelle formation. Differences in the Schlieren patterns of the ascending and descending limbs were observed.

Metal–protein interactions

An extensive study of the interaction of Cu^{2+}, Zn^{2+}, Cd^{2+} with bovine serum albumin in buffered and unbuffered solutions was made, using electrophoretic, polarographic, equilibrium dialysis, and potentiometric titration techniques. The polarographic method gave binding data comparable in accuracy with those obtained by the equilibrium dialysis technique. From a polarographic study of competition between Zn and Cd ions for interaction with albumin it was concluded that this protein contains two sites, more reactive than imidazole, which combine with Zn in preference to Cd.

It was shown that the binding data supplemented by electrophoretic mobility data could be used to treat the interaction in buffered solutions as a competition between the metal and hydrogen ions for combination with a given set of sites.

At *p*H 6.5 in an acetate buffer, the interaction of Cu^{2+}, Zn^{2+} and Cd^{2+} with albumin was found to occur through 2 to 3 compound sites involving, presumably, an imidazole site and a neighbouring peptide N or peptide O. Peptide N appeared to be involved in interactions with Cu^{2+} and peptide O with Zn^{2+} and Cd^{2+}. In the higher binding regions the interaction was found to be governed by competition between imidazole and carboxyl sites for the metal ions and may be accompanied by configurational changes in the protein molecule. The interaction of Co^{2+}, however, occurs through combination with the free carboxyl groups.

The interaction of Cu^{2+} and Co^{2+} in unbuffered nitrate medium was followed by tritation and equilibrium dialysis methods. The existence of compound sites through which the first two cupric ions are bound was confirmed. Co^{2+} is, however, bound to the imidazole groups of the albumin molecule in the nitrate medium, unlike the acetate medium. The intrinsic constant for Co^{2+}–albumin interaction has a value of $\log k^{\circ}=2.5$.

The interaction of Co^{2+} with simple imidazole at 2° and 25° was studied by the potentiometric titration method. The first association constant for Co^{2+}–imidazole interaction is $\log k^{\circ}=2.49$ at 25° and 2.80 at 2°.

The molecular weight, size, shape and depolarization characteristics of several synthetic and natural polymers have been studied in a number of solvents using the light scattering technique. The importance of carefully measuring dn/dc in this connection has been demonstrated.

A theoretical study of light scattering by anisotropic inhomogeneities in thin polymer films under strain (simple elongation) was made. These inhomogeneities were assumed to have two different polarizabilities, one longitudinal and the other transverse. A distribution function for orientation of these anisotropic inhomogeneities was obtained as a function of degree of strain and utilized to obtain average scattered intensity. Expressions for differential cross sections of scattering were derived.

A theoretical expression for the relative viscosity η_r of dilute solutions of long chain polymer molecules was obtained on the basis of the theory of rate processes, using an 'equivalent sphere' model which relates η_r to parameters such as the shearing force, absolute temperature and the volume fraction of the polymer. In the limit of zero rate of shear, a relation between the Flory constant ϕ and Huggins constant k'' was derived.

Water dispersible DDT formulaions

This project was sponsored by the Indian Council of Medical Research. Since DDT is a contact poison the diffusion of DDT into the insect body should be facilitated by covering the DDT particles by a non-volatile, lipoid-dissolving oil layer; further, needle-shaped DDT particles (diameter 1-2 μ, length 5–25 μ) should have much larger covering power than spherical particles (diameter 5–25μ). With these considerations in view a number of paste formulations incorporating a non-volatile oil and thin needle-shaped particles were developed. Preliminary experiments were also made to obtain wettable DDT powders by spray crystallization of molten DDT containing a suitable wetting agent. In this connection extensive basic data were obtained experimentally. For example, contact angle and sedimentation volume measurements were carried out with a number of wetting agent—DDT systems in order to find the most effective wetting and dispersing agent for DDT.

The experiments to spray-crystallize molten DDT required a large amount of fundamental work on the linear rate of crystallization of DDT particles as a function of supercooling temperature and in the presence of potential nucleating agents. It was found that additives such as (i) stearic acid and stearamide enhanced the rate of crystallization, (ii) γ–HCH retarded the rate, and (iii) clays and solid anionic type of wetting agents insoluble in molten DDT had no effect. Further, this rate was profoundly influenced by the degree of supercooling. The apparent activation energy for crystallization of DDT was evaluated. This was found to decrease rapidly on adding substances which enhanced the rate of crystallization. Substances such as stearic acid and stearamide were also found to enhance the rate of nucleation of supercooled DDT to some extent. The phase equlibria of p,p'–DDT with the above substances were also studied. These binary systems were found to form simple eutectic mixtures (e. g. ideal with γ–HCH and non–ideal with stearic acid). The latent heats of fusion of p,p'–DDT and γ–HCH were 6.7 and 5.8 Kcal/mole, as evaluated from these measurements.

Water dispersible DDT as an oil–bound paste formulation has been found to form a stable suspension with a suspensibility of 80–90 per cent (WHO specification requires 60 per cent and ISI 50 per cent). This process has been released to industry for commercial exploitation.

Monolayer properties of long chain compounds

This project was undertaken mainly to study the water evaporation retarding ability of the monolayers of various long chain compounds and to correlate this with the other physical properties of such monolayers. It was thought that better evaporation retardation than that due to monolayers of cetyl and stearyl alcohols could be achieved if the polar group of the fatty alcohols was suitably modified so as to reduce the melting point and increase the interaction with the water subphase, without introducing side chain, bending, stiffness, or instability on water. It was felt that this would be best achieved by condensing the long chain alcohol with an ethylene oxide molecule. Such glycol mono–*n*–alkyl ethers were indeed found to be much more effective water evaporation retardants than the parent fatty alcohols in laboratory as well as open air evaporimeter pan and small brick–lined tank experiments. Thus the percentage water evaporation reduction for C_n–OCH_2CH_2OH was always higher than for the corresponding C_n–OH, when $n = 16$, 18 or 22. Again, the glycol mono–*n*–alkyl ethers exhibited much greater ease of spreading than the alcohols. A mixture of the C_{18} and C_{22} ethers showed a remarkably high retardation (more than 80 per cent) over a temperature range of 20–35°. The C_{18} ether was found to be effective (more than 70 per cent reduction) in evaporimeter pan experiments while C_{18} alcohol had difficulties in spreading under comparable conditions.

THERMISTOR BRIDGE

The construction and operation of a simple A.C. thermistor bridge, used for measuring the osmotic activities of aqueous solutions, have been described. Osmotic coefficients of potassium laurate, Aerosol MA and potassium salts of dibasic acids were thus determined. The high values for potassium sebacate in dilute solutions were attributed to the cumulative effect of hydration and interionic attraction. A pair of matched thermistors can also serve as a sensitive element for determination of molecular weights of compounds in organic solvents.

ELECTRON AND OPTICAL MICROSCOPY

Texture of vacuum deposited films

The texture of Zn and Cd films condensed from vapour *in vacuo* on to plastic substrates was examined electron microscopically. Stray grease-like contaminants were found to promote their deposition. Pre-coats of metals such as Ag, Cu, and Al had a very pronounced promoting effect on the deposition. A surface sensitized with a silver deposit of the order of 1 Å average thickness collected uniform deposit of Cd or Zn of a small uniform crystal size which completly covered the surface. In further studies the texture of Zn and Cd films condensed *in vacuo* at low vapour beam flux densities on to plastic substrates having Ag pre-coats of 10^{-3}–10^{-1} Å average thickness was examined. It was concluded that each silver crystallite in the pre-coat nucleated either Zn or Cd equally well. It was found that the rate of deposition of the Ag pre-coat did not appreciably influence the Zn and Cd film textures.

Optical microscopic studies of Zn and Cd films condensed from vapour *in vacuo* on the two sides of mica cleavage exhibited well-matched textures. In those cases where the deposition was due to the Frenkel mechanism of homogeneous nucleation, there was a similarity in the gross features only. On the other hand, when the condensation was due to the presence of "active" centres of nucleation on the mica surface, there was generally a "one-to-one" correspondence between the crystallites on the two faces.

During a further optical microscopic investigation, Zn films, deposited on to stearic acid single crystals, exhibited remarkable textures in that the smooth surfaces between the well-known growth spiral steps on the crystal face became heavily metallized, while the spiral step edges and the regions in their immediate neighbourhood remained virtually free of the deposit. As a result, the growth spirals starting from screw-dislocations could be strikingly revealed with an optical microscope.

Epitaxy

In a series of experiments carried out to investigate the nature of epitaxial crystal growth, the growth of methylene blue and ammonium iodide crystals from solution on mica cleavage surface was studied. It was found that the orientation of a crystal growing from a nucleus formed on one side of some of the cleavage steps differed from that of a crystal seeded on the other side. Despite this, a growing crystal retained its original orientation on and after crossing such a step; growth was then no longer epitaxial. When sodium nitrate crystals growing epitaxially on a calcite cleavage surface were mechanically disordered, the crystals continued to grow in their deranged orientations uninfluenced by the substrate surface structure. It was thus concluded that in epitaxial crystal growth the substrate only determines the orientation of the first few atoms and that further growth of this nucleus is no longer influenced by the substrate surface structure.

Micro-organisms

Electron and optical microscopic studies of a variety of industrially important micro-organisms were conducted. Seven species of useful lactic acid bacteria were studied by electron microscopy with regard to cell size, shape and mode of division. Of the two cocci forms, *Streptococcus faecalis* divided only in one plane, thus forming long straight chains, while *Leuconostoc mesenteroides* divided successively in mutually perpendicular planes to form zigzag bunches. Among the rod-like forms, *Lactobacillus brevis,* which consisted of rather shorter rods as compared with the others, also appeared to divide successively in mutually perpendicular planes. In the remaining four types, extensive bud formation and asymmetric cell division seemed to be characteristic of *Lactobacillus arabinosus* and *L. acidophilus,* but not of *L. casei* or *L. delbrueckii.*

Comparative electron microscopic studies of sulphur oxidizing bacteria grown in sulphite, sulphide, thiosulphate and thiourea media were also carried out. It was observed that the isolate under study revealed the presence of flagella which were asymmetric in some and polar in others. The sulphur globules were prominently seen in dividing cells; there were variations in shape and size of the cells due to changes in physiological conditions, and there were indications of budding in the sulphite medium.

A comparative study of shaken and rest cultures of Baker's yeast (*S. cerevisiae*) was made, using various techniques such as colorimetric estimation of dye absorption, optical microscopy of stained cultures, phase contrast microscopy of unstained cultures, and electron microscopy of thin sections. The absorption of Lugol's iodine and toluidine blue started at 1½ hours with shaken culture and at 3 hours with rest cultures indicating an earlier glycogen build-up in the former. Cells from the rest culture showed large vacuoles, usually single, while those from the shaken cultures exhibited a large number of smaller vacuoles. The results also confirmed the laminated nature of the cell wall and the internal structure of the yeast cell.

ELECTRON DIFFRACTION

Corrosion of copper and iron

The thermal oxidation of Cu (110), (111) and (100) faces was carried out in undried air at temperatures varying between 125° and 300°. On (110), the resulting oxide films consisted of cuprous oxide, or a mixture of cuprous and cupric oxides, though cuprous oxide was always formed initially. With increasing film thickness, and depending on the temperature, the oxide crystals grew epitaxially or at random or exhibited a preferred orientation in both cuprous and cupric oxides. On Cu (111) and (100) faces, the initial oxide layer consisted of Cu_2O having an amorphous or a fine–grained structure. Increase in film thickness with temperature and/or time of oxidation resulted in (111) epitaxial growth of Cu_2O on both the faces. With further oxidation, mixed two–degree (111) + (100) orientated Cu_2O was formed presumably as a result of multiple twinning and finally the top layers consisted of randomly disposed or one–degree orientated Cu_2O. Above 200°, with sufficient time of heating and depending on the temperature, two–degree orientated, randomly disposed or one–degree orientated CuO layers were observed over the initial Cu_2O layers.

If a Cu surface, after oxidation in air at 100 mm Hg pressure, was cooled *in vacuo*, the oxide layer consisted solely of Cu_2O, a result which suggested that dissociation of CuO had taken place during cooling. Also, Cu (110) faces were anodically oxidized in caustic soda solution at 75–80° at different current densities and concentrations of the alkali. The oxidation products were either Cu_2O or a mixture of Cu_2O and CuO. The initial Cu_2O grew epitaxially and under certain conditions these were covered with layers of CuO, either random or one–degree orientated, depending upon the film thickness. The initial CuO film also appeared to grow epitaxially, but with a slight distortion of lattices (pseudomorphism). A mechanism of the growth of oxide crystals in the anodic oxidation process was suggested.

A dissolution rate study of iron from mild steel disks coated with electrodeposited nickel having (100), $(10\bar{1}0)+(211)$, (110) and (210) orientations revealed that, in hydrochloric acid solutions, coating with (211) orientation gave the best protection to the iron. On the other hand, in neutral sodium chloride solution no appreciable difference in the dissolution rate was observed. These results have been discussed with reference to the precipitation of basic iron salts, the electrochemical anisotropy of different faces of a single crystal, and also the texture of the deposits.

Electrodeposition

A study was made on the crystal orientation and mode of growth of deposits on polycrystalline substrates in sulphate-boric acid, sulphate-chloride-boric acid and chloride-boric acid baths. The effect of *p*H, temperature, concentration, current density, mechanical stirring, etc., and of various organic and inorganic additives was investigated. Depending on bath conditions various preferred orientations (one–degree), mixed or otherwise, were observed and their causes were discussed. It was found that the addition agents producing a high cathode polarization resulted in outward growth deposits. Chloride ions generally favoured lateral growth and counterbalanced the outgrowth condition due to impurities; this was attributed to the deposition of the impurities on the cathode, which facilitated the electron transfer process.

FACADE, MAIN BUILDING

Electrodeposited nickel was found to develop one-degree (100) orientation within a pH range 4 to 5.7 from a sulphate bath, whereas at a slightly higher pH value (5.85) mixed and equivalent (211) and $(10\bar{1}0)$ orientations were observed.

Iron and chromium, electrodeposited on Cu (110), (100) and (111) faces, initially grew in (211) or (100) orientation, but with different azimuthal orientations, and later they developed one-degree orientation depending on the bath conditions.

New structures

The following new structures were observed during the studies of thin films prepared under various conditions.

Substance	Structure	Preparation condition
Nickel	b. c. cubic $a_0 = 2.78$ Å	Vapour phase deposition on rock salt, mica and glass substrates
Molybdenum	f. c. cubic $a_0 = 4.16$ Å	Vapour phase deposition for 10–30 min at 10^{-4} to 10^{-5} mm Hg on rock salt or glass
Bismuth oxide	Cubic $a_0 = 5.65$ Å	Oxidation of Bi film in air
Bismuth oxide	Cubic $a_0 = 7.02$ Å	– do –
Nickel oxide	Metal excess spinel $a = 8.12$ Å (space group Fd3m)	Ni electrodeposited on Pt and heated in air at 500° for 2 hr and cooled slowly
β–Iron oxide	Unit cell same as Fe_2O_3, some cations located at tetrahedral interstices	On heating α–Fe_2O_3 in air at 500°
Copper sulphide	Cubic $a_0 = 33.51$ Å	Reaction with sulphur vapour at 300°
Copper sulphide	Cubic $a_0 = 4.34$ Å	– do –

SOLID STATE CHEMISTRY

The behaviour of monovalent impurities incorporated in alkali halides has been studied by the radioactive tracer technique. The trace impurity ions having sizes larger than the host ions were found to evaporate from the matrix of the host lattice at fairly low temperatures. Furthermore, the impurity concentration was found to have major effects on the rate of its evaporation, and there were optimum concentrations where evaporation was most rapid. The observed effects due to the size and the concentration of the impurity ions on its rate of evaporation can be explained in terms of overlap forces, Johnson's hypothesis of preferential diffusion impurities, and the concepts of quantum rate theory.

Using the concept of phonon-lattice interaction, an expression is derived for the minimum temperature, T_g, of a solid at which it may enter into solid state reaction. The reaction between two different solids was re-examined in the light of their physical and crystallographic properties. The rate law for such an additive reaction has been deduced from the quantum rate theory. It is concluded that crystallographic phase transformations and the formation of transitional superstructures constitute phase boundary processes and that the kinetics are governed by the dynamics of the diffusion process.

It was concluded that the $\alpha - \gamma$ transformation is a necessary step preceding the solid state spinelization reaction involved in the formation of a ferro-spinel. The nature of the vacancy distribution in the defect spinel type oxides γ-Fe_2O_3, γ-Mn_2O_3, γ-Cr_2O_3, and γ-Al_2O_3 was analysed in the light of available magnetic and structural data. All vacancies lie in octahedral sites, their distribution being ordered in γ-Fe_2O_3, but random in the other oxides. The tetragonal symmetry of γ-Mn_2O_3 which was attributed to dsp^2 square bonding of the cations in octahedral sites is stabilized by d^2sp irregular tetrahedral bonding in tetrahedral sites.

The origin of the distortion of spinels from cubic to tetragonal symmetry was examined in the case of copper ferrites and a series of manganite–ferrite systems. The degree of distortion was found to depend on the temperature and the net fraction of cations forming appropriately orientated dsp^2 bonds in octahedral sites. Expressions derived for the degree of distortion as a function of temperature and concentration agreed satisfactorily with the experimental data.

The tetragonal distortion of octahedral (ML_6) systems having two-fold orbital degeneracy was studied on the basis of a molecular orbital scheme which takes into account the covalency and essential features of the Jahn–Teller effects.

A number of new oxidic semiconductors having the spinel or hausmannite type symmetries were formed by solid state reactions and their structures, electrical conductivity and magnetic properties were studied at different temperatures.

Some compositions giving a high temperature coefficient of resistance were used for developing "thermistors" with various time and dissipation constants, room temperature resistivities and temperature coefficients of resistance. The process has been released to industry for commercial exploitation.

The superexchange interaction for the spin coupling in certain magnetic compounds was treated taking into account the deformation of the electron orbitals of the intervening ions. Quantitative calculations were carried out for a three centre-four electron system. A rough numerical estimate gave a reasonable agreement with the oberved Néel temperature for MnO. The conditions which lead to ferromagnetic and antiferromagntic spin coupling between magnetic ions were then explored. It turned out that the coupling is antiferromagnetic if the semilocalized orbitals of the anion show "angular" correlation effect and ferromagnetic if they suffer from "in–out" effect.

CRYSTAL GROWTH

Sapphire and ruby single crystals were grown in the form of boules and thin rods by the Verneuil technique with the object of determining the technical details of the process.

The colour problem in ruby was analysed in the light of the available structural and magnetic data. It was suggested that the anomalous colour effect, below a critical concentration of the Cr^{3+} ions, is due to the squeezing out of the 3d electrons from these ions into new satellite orbits.

X–RAY DIFFRACTION

The crystal structure of Ni_3Se_2 and $CdIn_2O_4$ was determined from the X–ray powder diffraction data. For Ni_3Se_2, the unit cell is rhombohedral with $a = 4.24$Å, $\alpha = 90°38.6'$, $z = 1$, and the space group R32, D_3^7 with the ionic arrangement – 2 Se at $\frac{1}{4}\frac{1}{4}\frac{1}{4}$, $\frac{3}{4}\frac{3}{4}\frac{3}{4}$ and 3 Ni at $\frac{1}{2}\frac{1}{4}\frac{3}{4}$, $\frac{1}{4}\frac{3}{4}\frac{1}{2}$. $CdIn_2O_4$ is cubic with $a = 9.11$ Å, the space group *Fd3m* and the ionic arrangement corresponds to that in an inverse spinel structure.

The decomposition of manganese carbonate was studied up to 1000° by the differential thermal analysis and X–ray diffraction techniques at different heating rates, in the presence of graphite and in various gas atmospheres, and also by heating in an external furnace under similar conditions. The formation of specific oxides of manganese at different stages was reported.

A homologous series of *n*–alkyl malonic acids, from methyl to octadecyl, was synthesized. Tables of X–ray powder diffraction data for the acids were presented in a form applicable for analytical purposes. The results were discussed with reference to those of the *n*–alkanoic acids and their derivatives.

RADIOACTIVE TRACER TECHNIQUE

A Radioactive Tracer Laboratory which will be soon provided with a separate new building has been organized.

Studies were carried out in the fields of solid and liquid state diffusion; C^{14} was used for studying the mechanism of fermentation of carbohydrates to citric acid and for the biosynthesis of simple carbon compounds; tracers were used for testing paint and varnish films; and investigations were made on the effect of radiation on polymerization reactions and on the production of mutants in micro-organisms and seeds.

In collaboration with the Armed Forces Medical College, the causes of anaemia were investigated. Seven patients out of fifteen showed a low vitamin B_{12} (labelled with Co^{58}) absorption as judged by urinary excretion. No obvious cause, such as steatorrhoca, seemed to be responsible for this malabsorption. This was unexpected and is at variance with the current views on anaemia in such cases. In a similar series of experiments radioactive iodine was used in connection with diagnostic studies of thyroid disorder.

Solutions of crepe rubber in carbon tetrachloride, tetrahydrofuran, benzene and ethylene dichloride were exposed to radiations from a 100 mc/Ra – Be source. Changes in colour, viscosity, infrared absorption spectra, which occurred in the carbon tetrachloride solutions, have been followed. Cyclization was observed in the final irradiated product which resembled that obtained by heat treatment of rubber with sulphuric acid.

INFRARED SPECTROSCOPY

The infrared spectroscopic technique was used extensively for structure elucidation and quantitative estimation of organic compounds, such as the isomers of kamlolenic acid, staphisine, compounds containing a cyclopropane ring, sterculic acid, geraniol, aconitine, and iso-oleic acid in hydrogenated fats.

The natural, aged, chlorinated, cyclized and cyclized-chlorinated rubbers were studied. In the infrared absorption spectra of solutions of natural rubber in carbon tetrachloride two new absorption bands were observed at 1538 and 1220 cm^{-1}. These bands disappeared when films from the solution were examined. It was suggested that the absorption at 1538 cm^{-1} might be due to a perturbed double bond in a loose complex formed between the rubber molecule and carbon tetrachloride. Rubber aged in solution also showed these absorptions. However, in the film from aged rubber the 1220 cm^{-1} absorption is found to persist.

n-Alkyl malonic acids and their diethyl esters showed a strong and broad absorption in the region 3400–3000 cm^{-1} indicating the presence of molecular hydrogen bonding in the acids. Furthermore, the frequency of C–O stretching vibration in the acid was found to alternate in odd and even numbered carbon chains.

From the infrared spectra of nickel and copper manganites formed by reaction in the solid state, the optical activation energy and the force constants for the stretching of bonds between the oxygen ions and the octahedral or tetrahedral cations have been calculated. The calculated force constants were used to evaluate the compressibility and the Debye temperature for these compounds.

PHOTOGRAPHIC EMULSIONS

Gelatine was prepared from hides and bones and then sensitized for the preparation of negative and positive emulsions.

Impurities such as Ag_2S in silver halides were suggested as producing complexes which act as effective electron traps, and this provides a rational view of the latent image formation mechanism in photographic processes.

INSTRUMENTATION

A special section for the maintenance, repair and fabrication of instruments has been organized as a part of the Division of Physical Chemistry. Equipment fabricated in the section with the active help of the workshop included an electron diffraction camera, X-ray unit, stabilized high tension supply, and automatic recording differential thermal analysis unit. In addition some developmental work was carried out: (i) The McBain-Bakr method was modified to obtain silica springs which were comparable to those prepared by mechanical devices. (ii) An inexpensive X-ray diffraction heating camera was designed for studying samples containing a volatile component. Both heating of the sample and determination of temperature were accomplished by the condensation of vapours principle. The camera was used successfully up to 250°. (iii) An electron diffraction specimen carrier having a fore vacuum stage between a high vacuum and the atmosphere was designed and constructed.

CHEMISTRY OF RARE ELEMENTS

India enjoys an advantageous position in the minerals of some "rare" or minor metals, such as titanium, zirconium, thorium, niobium and the rare earths, which have become important in recent years. The programme of work in this field included (a) the development of suitable chemical methods for the extraction of these elements from Indian raw materials, (b) isolation of the individual components of mixtures in high grades of purity, and (c) evolution of rapid methods for the estimation of minute quantities.

The light rare earths

In the processing of monazite for thorium and uranium oxides, the Indian Rare Earths Factory obtains mixed rare earth chlorides (hydrated) as a by-product, which contains nearly half its weight of rare earth oxides, principally of cerium, lanthanum, praseodymium, neodymium and samarium. Collectively they are usable in the manufacture of pyrophoric alloys such as mischmetal, but individually they are much more valuable in the ceramic, glass and leather industries, as catalysts, and for their nuclear properties.

The methods available for the separation of this extremely similar group of elements generally depend on fractionation of some kind, based on precipitation, crystallization, thermal decomposition, liquid–liquid extraction or ion exchange. Occasionally, where a different valency state of some stability exists, a chemical separation is possible which is much more effective. Different fractionation methods vary in the time involved, somewhat counterbalanced by corresponding separation factors, and a scheme of maximum efficiency is often the result of combination of more than one method at different stages and dependent on the degree of purification desired.

The separation procedure developed so far is as follows: Ceria, the major constituent in the mixture, is precipitated by oxidation–cum–hydrolysis from a fairly strong solution of the mixed chlorides with alkaline hypochlorite at a steady pH of 4.0–4.3. The 85 per cent crude is purified to over 99.9 per cent ceria by a single basic nitrate hydrolysis. Heavy earths are then removed as sodium double sulphate (with some light earths), and the precipitate of light earth sodium double sulphates converted to chloride through hydroxide. Fractional precipitation by air–diluted ammonia leaves 80 per cent lanthana in solution (recovery 80 per cent) in a single stage, which is then purified by loading a cation–exchange column to near capacity and running a short elution to remove the constituents other than lanthanum. The precipitated fraction, a quaternary mixture of Pr, Nd, Sm and La was earlier fractionated into simple binaries first, then resolved, but later a direct process of elution by sodium tripolyphosphate has been found which eliminates the intermediate step of isolation of the binaries. The scheme operates on single–stage separations under the most favourable set of conditions, and in such a way as to leave no idle fractions.

Zirconium and hafnium oxides from Indian zircon

Essentially, there were two important but somewhat separate problems involved in this investigation. The first was to develop a rapid method of isolating a pure zirconium compound having Hf/Zr weight ratio less then 0.01 per cent; the second, the relatively less studied problem of isolating the hafnium in a purity of 99 per cent or over, a problem particularly difficult of solution on account of the low hafnia content ($\approx$1.5% HfO_2) of Indian zircon.

Zircon mineral is usually opened up either through chlorination of the carbide or by sodium hydroxide fusion. For ease of processing, the latter method was preferred and details of preparation of the 8–hydrated zirconium-hafnium oxychloride were worked out. Using this stable salt as the raw material, a method of obtaining the hafnium–free zirconium oxychloride was developed by running an acidic methanolic solution of the mixture down an activated silica gel column. Three main fractions were separated: pure fraction ($<$0.01% Hf/Zr, 70–75% of input), low–Hf fraction ($\approx$0.5% Hf/Zr, 25–20% of input), and high–Hf fracton ($>$20% Hf/Zr, 5% of input). The silica gel and methanol were reusable after treatment. Recycling the second and third fractions increased their hafnium contents considerably.

A new anion–exchange procedure was developed for the preparation of pure or high–grade hafnium oxide. The hafnium concentrate ($Hf/Zr > 20\%$ w/w) from the silica gel column was used as such or further upgraded by a second run over silica gel. The oxychloride was converted to the potassium hexafluoro salt and impregnated on a strongly basic ion exchanger. Elution with 0.2 N sulphuric acid broke up the anionic complex of the more electropositive element, hafnium, which was collected in pure state in the effluent. Under specific conditions, the separation of the two constituents is quantitative.

Systematic data on solvent extraction were collected on the differential extractability of zirconium and hafnium. Several oxygenated solvents were found to extract zirconium or hafnium preferentially, and offered possibilities of separation in a multistage technique, especially in presence of a salting–out agent.

Niobium and tantalum oxides from Indian sources

The use of tantalum as an acid–resistant metal for fabrication work has been known for some time, but some of the applications of niobium metal, e. g., in cladding cores of fast reactors, are expanding. Niobium and tantalum are co–existent in all minerals and their separation in quantity remained a major problem till the introduction of the liquid–liquid extraction technique. Indian tantalo–columbite ores, obtained through the Department of Atomic Energy, were studied from several aspects. A laboratory method of obtaining the binary oxide mixture, low in other impurities, was first worked out. A liquid-liquid extraction method, using a solvent now manufactured in the country, was developed, which could be operated at low concentrations of hydrofluoric acid in glass containers without visible corrosion. A complete flow–sheet of the process of extraction from the ore was worked out for the Department of Atomic Energy.

Germanium

Germanite ore is not found in India, but the reported germanium contents (0.04–0.08 per cent) of some flue dust samples, especially those obtained from burning Assam coals, make the latter an interesting source for extraction of this truly rare element. Initial difficulties of a quick chemical method of estimating germanium were overcome by developing a new colorimetric reagent (quinalizarin acetate). Interferences were studied and a method for estimating germanium in flue dusts was worked out in detail. Several alternative methods of extraction of the germanium were studied, including hydrochloric acid distillation and extraction as sodium thiogermanate. An ammonium oxalate-oxalic acid mixture was very effective in the hydro-extraction of germanium from flue dust samples.

Co–deposition of germanium with several metals (Cu, Ag, Ni, Co, Sn) was studied from cyanide, citrate, tartrate, pyrophosphate and oxalate solutions. Its quantitative co-deposition with nickel from an ammoniacal oxalate solution was a new observation, and is the first successful attempt at complete recovery of germanium by electrolysis from very dilute solutions.

The reduction of germanium at the dropping mercury cathode was studied in buffered media. The reduction scheme is representable by $HGeO_3^- + 4e + 2H_2O \rightarrow Ge + 5OH^-$, but it appears that the reducible species is a poly-germanic acid, probably $H_2Ge_5O_{11}$, which undergoes depolymerization at higher *p*H values to a more difficultly reducible form.

TITANIUM COMPOUNDS

Titanium tetrachloride is a well-known defence chemical, and is an important raw material for the manufacture of titanium metal, rutile titanium dioxide pigment and organic titanium compounds. Large deposits of ilmenite occur in the form of black beach sands of Kerala, and form one of the world's most readily accessible natural sources of titanium. Also, the sludges left over in the Bayer process of extraction of alumina from some Indian bauxites contain 20-30 per cent of TiO_2, and are of interest as a source of titanium.

The customary sulphate process, as applied to ilmenite, needs modification in working with bauxite sludge to impart satisfactory pigment properties to the product. Complete laboratory conditions were worked out and published. Simple titrimetric procedures, which were fairly accurate and could replace the customary gravimetric methods, were developed for the estimation of active and total acids in a mixed sulphate solution of Ti, Fe and Al. In a systematic investigation on the chlorination (in presence of carbon) of minerals, such as monazite, ilmenite and rutile, etc., the laboratory data on experiments with bauxite sludge showed that (a) the yield of tetrachloride was satisfactory when the TiO_2 content of a sludge was above 35% w/w, (b) ferric oxide was chlorinated simultaneously with titania, and (c) in working with a partly upgraded sludge, the chlorination could be so adjusted as to leave the greater part of the alumina in the material unreacted with chlorine.

Trial experiments were carried out on the electrodeposition of titanium metal from titanium-alumina sponge. KCl–NaCl eutectic, KCl–LiCl eutectic and calcium chloride were used as fused electrolytes. The powdered thermit product was added to the fused electrolyte under a blanket gas of purified argon. Cathode materials tried were platinum and tungsten foils, stainless steel or graphite rods, the anode being usually a lump of the thermit product itself. In some cases, black adherent deposits giving positive tests for titanium were observed, but the chief difficulty was the insolubility of the crude material in the electrolyte.

Among the titanium organics, butyl titanate is well known as a constituent in coating compositions. In the absence of an indigenous industry producing butyl alcohol, fusel oil, which is available from the distilleries, was used as a substitute in preparing a number of esters including the titanate. One application of the latter was in the incorporation of titanium into a CNSL–formaldehyde varnish to impart moisture repellency and superior electrical insulation.

Limestone

In view of the importance of limestone as a raw material for the chemical industry a survey was undertaken in 1953 of higher grade limestones in terms of their significant chemical and physical properties. A monograph entitled "High–Calcium Limestones of India", incorporating data on fifty limestones forming the first phase of the study, has been published, and it is hoped that it will help the chemical industry to select the limestones most suitable for specific purposes.

Inorganic fertilizers

In the First Five-Year Plan of Industrial Development, the National Planning Commission made recommendations on investigating possible new fertilizers which could be made from readily available raw materials. Specific recommendations included dicalcium phosphate, ammonium phosphate and potash in any suitable form. The first process whose optimum conditions were worked out was for dicalcium phosphate, using Kossier phosphate rock as the raw phosphatic material. Simultaneously, the process was worked out for nodular phosphate from Tiruchirapalli. The latter product contained some calcium fluoride, but showed no loss of citrate-solubility on long storage. A Finnish process of making semi-acidulated phosphate (a mixture of water-soluble and citrate-soluble P_2O_5) was examined and worked on the basis of information obtained from Rikkhihappo Ja Superfosfaattitehtaat Oy, Helsinki. The process, which is similar to superphosphate, consumes less sulphuric acid per unit weight of phosphate rock. This work was extended to the synthesis of a new N–P compound fertilizer by acidulating phosphate rock with hydrochloric acid in presence of ammonium sulphate. Further studies showed that, by substituting ammonium sulphate with sulphate-nitrate, non-caking products of compositions 8–13, 11–11 and 15–8 ($N-P_2O_5$) could also be prepared. Surprisingly, ammonium carbonate and urea were found unsuitable for addition. Pot culture experiments carried out by the Indian Agricultural Research Institute indicated highly significant increases in the yields of wheat and *marwa* crops with high absorption of both nitrogen and phosphorus. From the ferruginous apatite rock of Singhbhum, which is considered unsuitable for superphosphate production, a new process of selective extraction of P_2O_5 by curing with ammonium bisulphate was worked out, the final product being a water-soluble mixture of mono-ammonium phosphate and ammonium sulphate (Ammophos II).

By the simultaneous action of ammonia and carbon dioxide on marine bitterns, a 22–0–4 water-soluble product was isolated, expected to be suitable for coconut plantation. Pure light carbonate of magnesia was a valuable by-product of the process. Two gallons of 34.5° Be bittern yielded 3.5 kg of the nitro–potash fertilizer and 1 kg of light magnesium oxide.

Barium compounds

The barium mineral, barytes, occurs in quantity in Andhra and Rajasthan. In spite of the fact that much of these deposits is of a good chemical grade, large quantities of barium chemicals including the simple salts are imported. Laboratory experiments showed that some off-colour grades of the mineral from Andhra could be made suitable for incorporation into rubber and for use as extender. High temperature chlorination in presence of carbon converted the mineral directly to the chloride with little loss of chlorine, the sulphur being largely removed as a mixture of volatiles including sulphur dioxide, hydrogen sulphide and elemental sulphur. By injecting steam into the chlorine, hydrogen sulphide became the principal gaseous product of the reaction.

Synthetic cryolite

Owing to the relatively high silica content of Indian fluorspars and the absence of a hydrofluoric acid industry in the country, a method of fluorine extraction by solutions of salts of tri- and tetra-valent metals was investigated. Aluminium sulphate is known to form a soluble complex of the type $Al_2SO_4F_4$. Experiments showed that 80 per cent of the fluorine in fluorspar samples was extractable with aluminium sulphate, the solution obtained after processing being practically free from silica. However, the solution of the fluorosulphate complex was found unsuitable for direct precipitation of cryolite as it invariably gave mixtures of cryolite with large proportions of alumina, which were difficult to separate later. The fluorosulphate complex formed syrupy solutions on concentration, solidifying to a heavily hydrated mass which gave up its fluorine as hydrofluoric acid at about 300°.

PHASE RULE STUDIES

The bitterns of Sambhar and Didwana lakes in Rajasthan consist chiefly of sodium chloride, sodium sulphate and sodium carbonate (normal and acid). Various suggestions had been put forward from time to time for the economic recovery of the constituents from the approximately 50 million gallons of bittern discharged every year. Equilibrium data at low temperatures of the quaternary system $NaCl–Na_2SO_4–Na_2CO_3–H_2O$ were not available in the literature. Equilibrium relationships of the system at 10°, 5° and 0° were obtained and plotted in a three-coordinate system according to the method used by Teeple. These data and the phase diagrams indicated how the constituents could be separated by chilling and fractional crystallization.

A study of the precipitation of sodium bicarbonate from the reciprocal salt pair $Na_3PO_4 + 3NH_4HCO_3 \rightleftharpoons (NH_4)_3PO_4 + 3NaHCO_3$ was made, including solubility determinations of tri-ammonium phosphate in water and trisodium phosphate in ammonia solutions. Under established conditions, the precipitation of triammonium phosphate from the above system was preferable to that of sodium bicarbonate. In a similar system with tripotassium phosphate, it was found necessary to carry out the precipitation of triammonium phosphate from not too concentrated solutions to prevent contamination of the triammonium phosphate by potassium bicarbonate. Thus the greater solubility of tripotassium phosphate gave it no special advantage over the sodium salt in the ammono–carbonation reaction.

INORGANIC ANALYSIS

This somewhat flexible line of activity derived its orientation of research-cum-service from other more specific projects where an evaluation of attempted methods became necessary. Modern techniques of spectrophotometry, emission spectrography, polarography, etc., were used in addition to classical analytical methods. Published methods were first tested and adapted where necessary to suit any special requirement. Some new methods also resulted from such critical studies.

Benzohydroxamic and oxalohydroxamic acids were found to be good analytical reagents for the estimation of vanadium, and of calcium, zirconium and thorium respesectively. The insolubility of bismuth phosphate in dilute acid solutions and the stability of the orange–red complex of bismuth diallyl dithiocarbamido–hydrazine together formed the basis of an indirect titrimetric method of estimation of phosphate in rocks and synthetic fertilizers and also of a phosphate titrimetric procedure for the estimation of Mg, Zn, Mn, and Cd. A quick and comparative method of assay of edible salt, based on the physical comparison of a barium sulphate precipitate and of a calcium–magnesium phosphate precipitate from the solution of the salt, was developed to suit limited facilities available with small-scale salt manufacturers.

A micro–gravimetric method of estimating germanium, silicon and phosphorus as their acridine heteropolymolybdates was worked out. The wet precipitate could be dissolved in a measured excess of alkali followed by back titration with acid in presence of phenolphthalein or thymol blue indicator. Alternatively, the acridine content of the precipitate could be estimated by fluorescence or ultraviolet absorption measurement. New spectrophotometric methods were developed for titanium with the reagent phenylfluorone, for niobium with sulphosalicylic acid, and for cerium, vanadium, uranium and molybdenum with pyrocatechol disulphonic acid. Mapping of the absorption spectrum of yellow molybdo-germanic acid showed that the optical density at 350 mμ was 40 times as large as at 440 mμ, and remained unchanged for a long time.

Intensity analysis of similar emission line pairs due to zirconium and hafnium led to the choice of suitable pairs for the estimation of low amounts of zirconium in hafnium and *vice versa.* Similar studies on binary mixtures of niobium and tantalum oxides led to the selection of suitable pairs for the estimation of each as an impurity in the other.

A fundamental study of the formation and interconversion of the nitro and other acido-complexes of niobium and reduction of the former at d.m.c. established conditions for linear proportionality between the limiting current and sub-millimolar concentration. Similar reduction studies of uranium in an alkaline sodium tripolyphosphate buffer indicated a one-electron irreversible reduction process, on the basis of which a polarographic estimation of uranium in presence of certain other elements was developed. At *p*H 9, the half-wave potential for uranium was sufficiently separated from most elements which interfere in its conventional polarographic analysis, e.g.. Ti, Ce, As and Mo. A general polarographic study of the complexes of a number of metals with sodium tripolyphosphate showed their similarity with the pyrophosphate complexes, but indicated greater stability. The reduction waves were generally irreversible and were distorted, even totally suppressed, by the presence of small amounts of surface-active agents such as camphor and gelatine.

NATURAL ORGANIC PRODUCTS

Constituents of chana (Cicer arietinum *Linn.)*

Three crystalline products, biochanin A, B and C were isolated from the fresh germ of *chana,* one of the major foodgrains of India. On the basis of chemical evidence biochanin A and B were assigned the structures of 5,7-dihydroxy-4′-methoxyisoflavone and 7-hydroxy-4′-methoxyisoflavone (formononetin) respectively. The structure of biochanin A was confirmed by a direct comparison with a synthetic sample kindly supplied by Professor Wilson Baker. Pratensol isolated from red clover flowers by Power and Salway is probably identical with biochanin A. Formononetin was synthesized in good yield from 2,4-dihydroxyphenyl 4-methoxybenzyl ketone by the ethyl orthoformate method and its identity with biochanin B was confirmed. Biochanin C proved to be identical with *l*-asparagine.

Biochanin A was found to be oestrogenic and its activity was 1.2 ($\pm$ 0.3) x 10^{-4} of oestrone as determined by the Allen-Doisy procedure using ovariectomized albino rats. 5-*O*-Methyl biochanin A prepared through partial benzylation of biochanin A, followed by prolonged methylation and debenzylation, was oestrogenically inactive in doses up to 5 mg/rat, indicating that the presence of a hydroxyl group in 5-position is necessary for enhanced oestrogenic activity. *Chana* oil was also found to have slight oestrogenic activity and a fraction having fairly high oestrogenic activity was obtained.

Three crystalline glycosides were isolated from alcoholic extractive of defatted *chana* meal. The aglycones from two of them belong to the group of triterpene alcohols.

Synthesis of geijerin

Geijerin, 6-isovaleroyl-7-methoxycoumarin, isolated by Lahey and Wluka (1955) from the bark of *Geijera salicifolia,* has been synthesized. The Fries reaction on 7-isovaleroxycoumarin gave 8-isovaleroyl- or 6-isovaleroyl-7-hydroxycoumarin according to conditions, and methylation of the latter yielded geijerin.

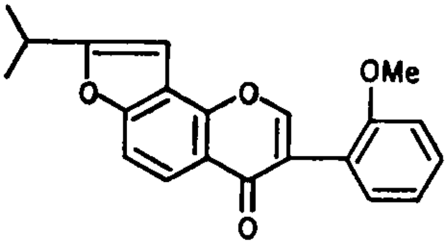
Synthesis of flavan-3,4-diols

The leucoanthocyanins are of great biogenetic interest because they are possible precursors of flavonoids, catechins and the condensed tannins. Some leucoanthocyanins have been shown by F. E. King and others to be flavan-3,4-diols. Attempts were made to synthesize all the four possible racemates of a few flavan-3,4-diols. It was found that the reduction of the dihydroflavonols with lithium aluminium hydride and Adams catalyst was stereospecific, and two racemates of 6-methyl-4′-methoxyflavan-3,4-diol were synthesized. Stereochemical conformations were further deduced by introducing for the first time the concept of conformational analysis in this field. Failure to synthesize the epimeric dihydroflavonols (the obvious starting material for the synthesis of the remaining two racemates of the flavan-3,4-diol) led to the investigation of other routes. Two isomeric 3-bromoflavan-4-ols were then synthesized from the epimeric 3-bromo-6-methyl-4′-methoxyflavanone, and their reactions studied. The third racemate of the flavan-3,4-diol was then synthesized by replacement of bromine by the acetoxyl group in one of the 3-bromoflavan-4-ols. Thus, three out of the four possible racemates of 6-methyl-4′-methoxyflavan-3,4-diol and two racemates of melacacidin (7,8,3′,4′-tetramethoxyflavan-3,4-diol) different from the natural product were synthesized.

The fourth racemate of 6-methyl-4′-methoxyflavan-3,4-diol was subsequently synthesized in the Institute of Science, Bombay, where this work was continued.

Munetone

Several crystalline compounds have been isolated from the root bark of ***Mundulea suberosa,*** well known as an effective fish poison. From degradative experiments coupled with infrared and ultraviolet spectral studies, the constitution of the principal compound, munetone, was established as 2′-methoxy-7,8(2-isopropyl-4,5-furano)-isoflavone. Munetone has been found to be highly toxic to fish.



Munetone

Colchicine and gloriosine

The rhizomes of *Gloriosa superba,* available in India, may be a useful raw material for colchicine used in medicine and polyploidy. A process for the preparation of pure colchicine from the mature tubers was worked out.

A new alkaloid, gloriosine, was also isolated and assigned the structure of *N*-formyl-iso-desacetyl colchicine.

Transformations of kurchi alkaloids

The Indian kurchi *(Holarrhena antidysenterica)* contains 24 or more alkaloids with the pregnane nucleus. Four of these, holarrhimine, conessine, isoconessimine and conessimine, have been converted to 20 new 18-substituted steroids, some of which can be considered to be potential intermediates in the synthesis of aldosterone and other steroid hormones. Holarrhimine was converted to 18-hydroxyprogesterone in a one-stage procedure in about 50 per cent yield. 18-Hydroxyprogesterone was converted to 11,18-dihydroxyprogesterone by a microbiological method. On treatment with nitrous acid holarrhimine yielded a number of products, some of which were converted by acetylation with boron trifluoride and acetic anhydride to 3β,18,20α-triacetoxy-5-pregnene. Progressive deacetylation of triacetyl holarrhimine led to the formation of the neutral diacetyl and the basic 3-monoacetyl holarrhimine. The proximity relationship of the hydroxyl group at position 18 and the amino acid group at position 20α in 3-monoacetylholarrhimine was studied through the behaviour of this compound in different reactions.

Conessimine was synthesized for the first time from conessine through its mono-*N*-oxide. Both conessimine and conessine were converted to 18-hydroxyprogesterone, and isoconessimine to 3-oxo-4-conenine.

Constituents of the fruit of Capparis moonii

From the fruits of *Capparis moonii,* for which antitubercular properties have been claimed, four crystalline products were isolated, three of which have been identified as *l*-stachydrine, rutin and β-sitosterol. *In vitro* tests against *M. tuberculosis* and tests in experimental tuberculosis of guinea-pigs, carried out at the Central Drug Research Institute, Lucknow, and elsewhere have given negative results.

Lipids

(a) Nim *(*Melia indica*) oil and its constituents.* The *nim* tree is extensively grown throughout the country on account of the medicinal properties attributed to its various parts. The dried seeds contain 45 per cent of an oil, but owing to its bitter taste and unpleasant smell only an insignificant portion of the available seeds is processed for oil, which is mainly used for medicinal purposes. A process for the extraction of the bitter principles as well as the malodorous component of the oil with ethanol was developed, which resulted in simultaneous purification of the oil. The fatty acid composition of the resulting refined oil, which has potential industrial uses, was determined. From the alcohol extract two crystalline bitter principles, nimbin and nimbinin, and an amorphous bitter principle, nimbidin, were isolated. Clinical trials indicated the efficacy of nimbidin in the treatment of skin affections. Alkaline hydrolysis of nimbidin under certain conditions yielded amorphous nimbidinic acid, the sodium salt of which stimulated uterine contraction and had diuretic action.

Work on the structure of nimbin is in progress.

(b) Kamala *seed oil. Kamala* oil, obtained from the seeds of *Mallotus philippinensis* Muell. Arg., contains α–kamlolenic acid to the extent of 55–60 per cent of the total fatty acids. On the basis of chemical studies and physical evidence, α–kamlolenic acid was assigned the structure of ω–hydroxy–*cis*–9,*trans*–11,*trans*–13–octadecatrienoic acid. On irradiation with ultraviolet light this acid is converted to β–kamlolenic acid which was assigned the structure of ω–hydroxy–*trans*–9,*trans*–11,*trans*–13–octadecatrienoic acid.

Owing to the size of the molecule and the presence of an ω–hydroxyl group, kamlolenic acid has attracted attention as a suitable intermediate for the production of macrocyclic compounds of value in perfumery.

The presence of kamlolenic acid imparts quick–drying properties to the oil. Varnishes prepared from *kamala* seed oil modified alkyd resins form films which, on baking, set to wrinkle–finish patterns resistant to water, alkali and organic solvents.

(c) *Inedible vegetable oils.* Inedible oils, such as *Karanja (Pongamia glabra* Vent.), *Nageswar (Mesua ferrea)* and *Polang* (*Calophyllum inophyllum* Linn.), which cannot be refined by conventional methods, were refined by the ethanol extraction method used earlier for *nim* oil. The fatty acid compositions of refined oils, which should be suitable for hydrogenation and soap manufacture, were determined.

From the non–glyceride portion of the nut oil of *Calophyllum inophyllum*, calophyllolide, calophyllic acid, and a new polyene acid, inophyllic acid, were isolated. On the basis of chemical and other evidence, inophyllic acid was assigned the structure of ω–phenyl–2,7–dimethyl–5–one–6,8–nonadienoic acid.

(d) *Composition and utilization of sugar–cane wax and sisal wax.* The gelling property of carnauba wax which makes it suitable for use in polishing compositions is attributed to the presence of high molecular weight long chain polyesters, which are synthesized in nature mainly from long chain diols and ω–hydroxy acids. Investigations on the composition of sugar–cane wax showed it to contain long chain alcohols, acids and some sterols. It has no gelling properties, owing probably to the absence of ω–hydroxy acids. Amide and ester waxes prepared from oxidized sugar–cane wax could be used to give fairly hard creams which, however, do not stand high summer temperatures.

Saponified sisal wax (from the waste of *Agave cantala*) was found to contain long chain acids, alcohols, diols and ω–hydroxy acids, being comparable in composition to carnauba wax. Sisal wax has high gelling properties and some of the cream samples prepared from 'it stood higher summer temperatures better than carnauba wax.

The component acids and alcohols of sugar–cane and sisal waxes were determined by paper chromatography after Kaufmann and Pollerberg.

A process for the extraction of wax from sisal waste has been patented in India and leased to industry.

Saponins

While considerable work on the aglycone part of a saponin from Indian saponin-bearing plants has been reported, few references to work on the carbohydrate part are available. In the course of a systematic study of the composition of the saponin glycosides, a number of easily available plants was investigated for the identification of the sapogenins as well as the sugar moiety of the pure saponins. The sugars from the hydrolysates of pure saponins, isolated from *Entada scandens, Balanites roxburghii, Sapindus trifoliatus* and *Bassia latifolia* were identified by paper chromatography. A new triterpene aglycone, entagenic acid, was isolated from the seeds of *Entada scandens.* The course of the separation of pure saponins by chemical methods was followed by a paper partition chromatographic technique in which sodium periodate in alkaline permanganate solution was used as a spray reagent giving permanent brown spots against a white background.

NATURAL COLOURING MATTERS

Flavonoids of Artocarpus *species*

The heartwood of *Artocarpus integrifolia* contains several flavones and a flavanone with the unique 2′,4′-orientation of hydroxyl groups in the B-ring. Two of them, artocarpin and isoartocarpin, are also unique because the former contains an isopentenyl substituent in the 3-position and the latter an isopentenyl ether group. Other *Artocarpus* species are under investigation.

New methods for the synthesis of naturally occurring anthraquinones

Naturally occurring anthraquinone derivatives with one or two possible exceptions are no longer of interest as colouring matters, but their physiological properties justify further study. With the object of examining a series of naturally occurring anthraquinones and their analogues for their antitubercular and other physiological properties, routes for the synthesis of hydroxyanthraquinones, anthraquinone carboxylic acids and anthraquinone carbinols, starting from common anthraquinonoid dye intermediates, have been developed.

The colouring matters of lac

The yellow pigment, erythrolaccin, isolated by Tschirch and Lüdy in 1923, has been shown to have the structure 1,2,5,7-tetrahydroxy-3-methylanthraquinone. Dimroth's laccaic acid has proved to be a complex mixture, some of the constituents containing a primary amino group in a side-chain; but all are derivatives of purpurin. Work is in progress to isolate all the constituents in the pure state and determine their complete structures.

Anhydrous zinc chloride and phosphorus oxychloride mixure – a new reagent

A mixture of anhydrous zinc chloride and phosphorus oxychloride in certain proportions has been found to be very effective in a number of condensations in which the individual reagents are practically ineffective.

(a) *Synthesis of hydroxybenzophenones and hydroxyxanthones.* Hydroxybenzophenones and hydroxyxanthones have been prepared in good yields from phenols and *o*-hydroxybenzoic acids in the presence of the new reagent. Hydroxyxanthones were obtained directly when the acid component was a 2,6–dihydroxybenzoic acid or when the phenolic component was either orcinol or phoroglucinol; other phenols gave hydroxybenzophenones. Twenty-three xanthones and nine hydroxybenzophenones were prepared by this method. 2,4,2′,4′–Tetrahydroxybenzophenone, which has recently been found to be a very effective ultraviolet absorber, appears to be produced commercially in the U.S.A. under the name Uvinul D–50, following the same procedure with slight modifications.

(b) *Aryl alkyl ketones and deoxybenzoins.* Phenols, free or protected, reacted with aliphatic or arylacetic acids in the presence of the new reagent under mild conditions, providing good yields of the corresponding aryl alkyl ketones and deoxybenzoins. Thirteen aryl alkyl ketones, including 2,4-dihydroxy-*n*-caprophenone useful for reduction to hexylresorcinol (a well–known antiseptic and anthelminthic) and nine deoxybenzoins including deoxyanisoin, a key intermediate for the synthesis of diethylstilbestrol, have been prepared in good yields by this method. A specific advantage of this method is the almost exclusive formation of the *p*-isomer in most cases. These processes have been patented in India, and have been leased to Unichem Laboratories, Bombay, for the production of hexylresorcinol and diethylstilbestrol.

Synthesis of 4-hydroxycoumarins

One of the most useful applications of the new reagent is for a new synthesis of 4-hydroxycoumarin and its derivatives by a direct condensation of phenols with malonic acid under mild reaction conditions. Other agents, such as stannic, aluminium or ferric chloride, individually or in conjunction with phosphorus oxychloride, were almost ineffective in this reaction. Seventeen 4-hydroxycoumarins have been prepared in good yields, including the parent compound 4–hydroxycoumarin, which is the key intermediate for the synthesis of important anticoagulant drugs, such as warfarin sodium, sintrom, and tromexan, and the modern rodenticides, warfarin, tomorin and fumarin.

This process has been patented in India and England and patents in Germany, Switzerland and the U. S. A. are pending. The process covered by the Indian patent has been leased to Unichem Laboratories, Bombay.

It was also found that diaryl malonates, when treated with one mole of the corresponding malonic acids in the presence of the new reagent, also gave 4-hydroxycoumarin in good yields. Nine 4-hydroxycoumarins including the parent compound were prepared by this method. This process was also covered by an Indian patent and leased to Unichem Laboratories, Bombay.

2, 4-Dihydroxyquinolines

An aromatic primary amine and malonic acid react in the presence of the new reagent giving fairly good yields of 2,4-dihydroxyquinolines. Thirteen 2,4-dihydroxyquinolines were prepared by this method. This reaction can also be effected by phosphorus oxychloride alone, but the products were difficult to purify. The process has been covered by an Indian patent.

Diethylstilbestrol

A new process was developed for the stereospecific dehydration of the stereoisomeric forms of 3,4-dianisylhexan-3-ol to *trans*-diethylstilbestrol dimethyl ether in almost quantitative yield by using phosphorus oxychloride under certain conditions as the dehydrating agent. *trans*-Diethylstilbestrol was then prepared by the usual procedure of alkali demethylation of the dimethyl ether under pressure. The process has been patented in India and has been leased to Unichem Laboratories, Bombay.

Metal hydride reductions

A new route to anthraquinone carbinols has become available through the selective reduction of anthraquinone carboxylic acids with diborane in diglyme. Thus aloe-emodin (1,8-dihydroxy-3-hydroxymethylanthraquinone) and citreorosein (1,6,8-trihydroxy-3-hydroxymethylanthraquinone) were prepared in good yield from 1,8-diacetoxyanthraquinone-3-carboxylic acid and emodic acid triacetate respectively.

A new general method has been developed for reducing anthraquinones to the corresponding anthracenes, using a mixture of sodium borohydride and boron trifluoride-etherate in diglyme.

The germicidal activity of some pyridinium salts

Cetyl pyridinium bromide and similar *N*-alkyl pyridinium salts are known to have germicidal properties. Stearyl, oleyl, linoleyl and linolenyl pyridinium bromides were synthesized and assayed for germicidal activity. The bactericidal potency of these compounds increases with the degree of unsaturation in the hydrocarbon chain of the cation, maximal activity being shown by linolenyl (phenol coeff. 650) and the minimal activity by the stearyl derivative (phenol coeff. 100). Pyridinium bromides were also prepared from the mixed fatty acids of two naturally occurring fats, hydnocarpus oil and shark-liver oil, which contain a high proportion of unsaturated acids. The mixed pyridinium bromides possess high bactericidal activity (phenol coeff. 475 and 550).

Cyanuric chloride condensations

Cyanuric chloride provides a convenient method for the synthesis of analogues of isonicotinic acid hydrazide and of *s*–triazine derivatives of *p*–aminosalicylic acid, and diaminodiphenylsulphone. By the stepwise replacement of the reactive chlorine atoms in cyanuric chloride it becomes possible to combine antitubercular agents of two or three types and to modify the solubility, acid or basic character and other properties of a given type of antitubercular agent. Other types of chemotherapeutic agents built on cyanuric chloride are under investigation. The synthesis of reagents useful for the identification of alcohols, phenols, aldehydes, ketones, amines, and amino acids is another aspect of this programme.

SYNTHETIC DYES

Constitutional problems concerning anthraquinonoid vat dyes

Anthraquinonoid vat dyes are of interest to the organic chemist, because they include many complex homocyclic and heterocyclic compounds and offer numerous problems for investigation from the point of view of structure determination, degradation methods, synthetical reactions, and relationships between chemical constitution and colour, affinity for cellulose, and fastness properties. Work carried out in this field in the Department of Chemical Technology in Bombay is being continued. Mayvat Brilliant Red AF has been identified as a halogenated derivative of 3,4,9,10–dibenzopyrene–5,8–quinone (I), the latter being obtained by dehalogenation of this dye or Indanthrene Scarlet 4G with Raney nickel, aqueous sodium hydroxide and hydrogen. Reduction of (I) by Coffey's method, using aluminium tricyclohexoxide or reduction of the sulphuric ester of the leuco compound by Raney nickel, yielded 3,4,9,10–dibenzopyrene, a potent carcinogen isolated by Bonnet and Neukomm from tobacco smoke. The general method for the reduction of anthraquinones to the corresponding anthracene derivatives by the action of Raney nickel on the sulphuric esters of the leuco derivatives has been applied to several 1,2–benzanthracenes.

An apparatus and procedure for the chromatographic adsorption analysis of vat dyes on alumina at temperatures between, 100^0 and 200^0 using solvents such as *o*–dichlorobenzene, trichlorobenzene, phenol, *o*–chlorophenol, and *m*–cresol for adsorption, development and elution, have been described. The constitution of the essential constituent of nitrated dibenzanthrone, used for producing black shades on cotton by application as a vat dye and subsequent hypochlorite oxidation on the fibre, has been conclusively proved to be 16–nitrodibenzanthrone. Crystalline 16–nitrodibenzanthrone was prepared by nitration of dibenzanthrone and also isolated from commercial nitrated dibenzanthrone by chromatography on alumina at 110°. Chromic acid oxidation followed by decarboxylation gave 2,2′–dianthraquinonyl. The constitution was confirmed by reduction to

16-aminodibenzanthrone, synthesized by alkali fusion of an equimolar mixture of benzanthrone and 2-aminobenzanthrone; the resultant mixture of dibenzanthrone, 16-aminodibenzanthrone, and 16,17-diaminodibenzanthrone was separated by hot chromatography. 16,17-Diaminodibenzanthrone was prepared by alkali fusion of 2-aminobenzanthrone. 16-Nitrodibenzanthrone or 16, 17-dimethoxydibenzanthrone with chromic and sulphuric acids under controlled conditions gave 1,2,7,8-diphthaloylphenanthraquinone, a new triquinone; further oxidation then yielded the known 2,2'-dianthraquinonyl-1,1'-dicarboxylic acid.

Condensation of 2,4-dinitrochlorobenzene with o-hydroxyarylamides

o-Hydroxyarylamides react readily with 2,4-dinitrochlorobenzene in presence of bases to give the *N*-2,4-dinitrophenyl derivatives. The reaction appears to proceed by a direct displacement of the halogen by the amide nitrogen involving intramolecular nucleophilic catalysis by the phenoxide oxygen; but a Smiles rearrangement is not completely excluded. The *N*-dinitrophenyl derivatives are stable to boiling hydrochloric acid, but are hydrolyzed easily by aqueous sodium carbonate or hydroxide. Condensation with dinitrochlorobenzene, followed by mild alkaline hydrolysis, is a useful procedure for determining the constitution of azoic coupling components of the Naphtol AS type. Thus Naphtol AS-RS and AS-KN have been shown to be the 4-chloro-2-methoxy-5-methylanilide of 2-hydroxy-3-naphthoic acid and the α-naphthylamide of 3-hydroxydibenzofuran-2-carboxylic acid respectively.

Derivatives of 1,3,5-triazine

The ready availability of cyanuric chloride and the discovery of the Procion and Cibacron type of dyes capable of direct chemical combination with cellulose have created new interest in 1,3,5-triazine derivatives. Some work in this field initiated in 1946 in the Department of Chemical Technology, University of Bombay, has been resumed. The condensation of cyanuric chloride with *o*-hydroxybenzanilides takes place on the amide nitrogen. Treatment of the products with acetic anhydride and pyridine opens the triazine ring and yields benzoxazine derivatives. When one mole of cyanuric chloride is condensed successively with one mole of an azoic coupling component of the Naphtol AS type and 2 moles of dimethylamine, the product undergoes smooth hydrolysis under mild conditions to an *o*-hydroxycarboxylic acid, from which the constitution of the Naphtol can be deduced.

Attempts are being made to prepare new types of reactive dyes based on cyanuric chloride.

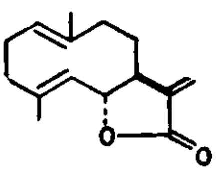
ESSENTIAL OILS AND PERFUMERY MATERIALS

India has large resources of essential oil-bearing plants, but these have mainly been exported, either as crude raw materials or after preliminary processing, and the country has been importing at the same time several crores of rupees worth of perfumery products and aromatic chemicals. A small research group was therefore organized in 1952, and was expanded in 1960 into a Division. The programme includes a systematic chemical investigation of the constituents of essential oils of potential commercial importance, as well as a study of methods of synthesis of perfumery materials.

Costus root oil

A low-temperature solvent extraction procedure has been developed for extraction of costus root (*Saussurea lappa*) in high yields. The oil thus extracted is free from denaturing and polymerization and is obtained in the form in which it exists in the roots, containing about 50 per cent by weight of crystalline lactones. The delactonized costus root oil obtained after partial removal of the lactones is more stable, retains the perfumery properties of the whole oil, and has found favour with perfumery houses abroad.

In addition to the compounds obtained from the oil by previous workers, three new lactones, costunolide (I), dihydrocostunolide (II), and methoxy-costunolide (III), have been isolated by column chromatography. These lactones contain a somewhat uncommon 10-membered carbocyclic system; their structures and stereochemistry have been completely elucidated. Costunolide is possibly a biogenetic precursor of lactones of the santonin group and easily cyclizes to bicyclic systems. Methoxy-costunolide is probably an artefact, and has been synthesized from costunolide by treatment with methanolic alkali. Saussurea lactone (IV) has been obtained by the pyrolysis of dihydrocostunolide (II). Its structure and absolute configuration were elucidated by converting it to santanolide 'C' (V). The lactone fractions also contain a large amount of dehydrocostus lactone (VI) and some dihydro-dehydrocostus lactone (VII). Other constituents of the oil are costol (VIII), aplotaxene (IX), β-sitosterol, lupeol, and α- and β-ionones.



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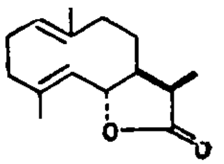

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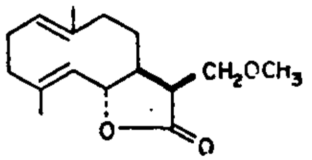

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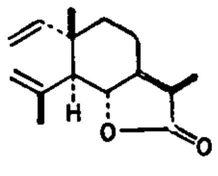

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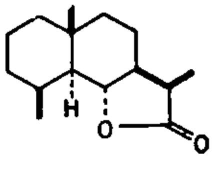

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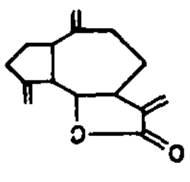

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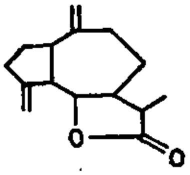

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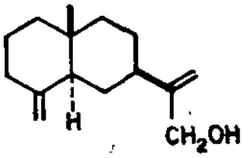

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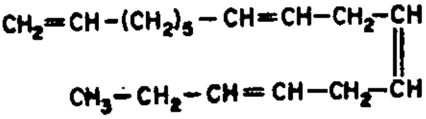
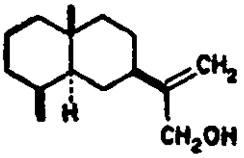


$$\begin{array}{l} \mathrm{CH_2{=}CH{-}(CH_2)_5{-}CH{=}CH{-}CH_2{-}CH} \\ \hfill \| \\ \mathrm{CH_3{-}CH_2{-}CH{=}CH{-}CH_2{-}CH} \end{array}$$

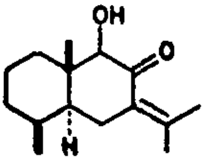
(IX)

Agar-*wood oil*

The constituents of *agar*-wood oil obtained from the fungus infected plant, ***Aquilaria agallocha,*** have been examined. Its perfumery value has been known to people in the orient since ancient times. The two main constituents are agarol (X) and hydroxyagariphilone (XI), whose structure and stereochemistry have been elucidated by eventually converting them to selinane (XII). Agarol is identical with dihydrocostol. Hydroxyagariphilone is similar to hydroxydihydroeremophilone, but unlike the latter it contains a *trans*-selinane structure.



(X)



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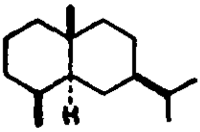

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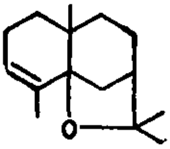


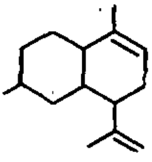
(XIII)

Another interesting constituent isolated from *agar*-wood oil is agarofuran (XIII). Its structure has been solved by a study of its degradation products and NMR spectra.

Vetiver oil

The examination of commercially important vetiver oil from different regions of India has led to the isolation of a series of new sesquiterpenoids, whose structures have been elucidated. Among these are the cadalenic hydrocarbons (XIV) and (XV) and khusinol (XVII), and the crystalline unusual selinenic secondary alcohol,laevojunenol (XVIII).

The absolute configuration of laevojunenol has been fully elucidated. It is the laevo antipode of the dextrorotatory alcohol junenol (XIX) previously isolated by Šorm from juniper berry oil. During this investigation the absolute configuration of junenol itself has also been established. Junenol (XIX) and dihydrojunenol (XX) have been synthesized from costunolide via the lactones (XXI) and (XXII). The synthesis involved controlled reduction of the lactones with lithium aluminium hydride to hydroxyaldehydes (XXIII) and (XXIV), followed by Huang-Minlon reduction.



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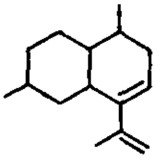

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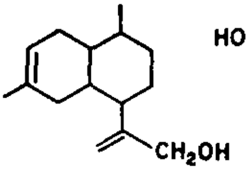

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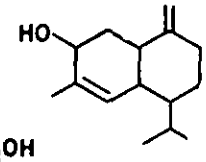

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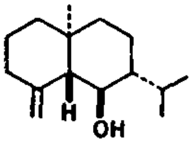

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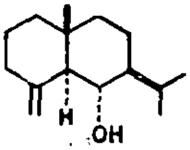

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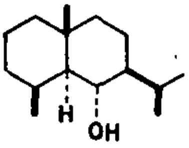

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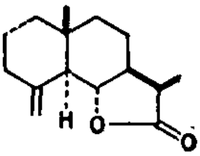

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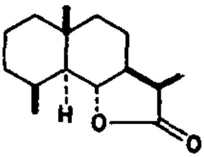

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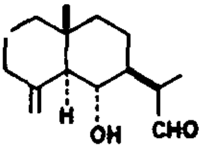

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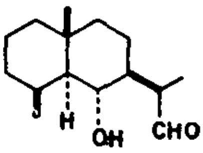
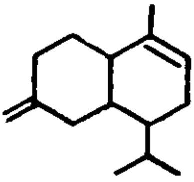


(XXIV)

Among other sesquiterpenoids whose structures have been elucidated are γ-cadinene (XXV) from Malabar lemongrass oil (*Cymbopogon nardus* Rendle), eupatene (XXVI) and eupatol (XXVII) from the oil of *Eupatorium odoratum*.



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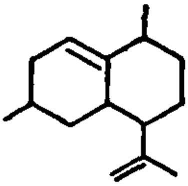

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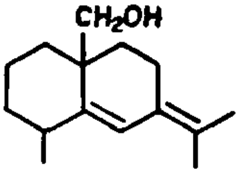


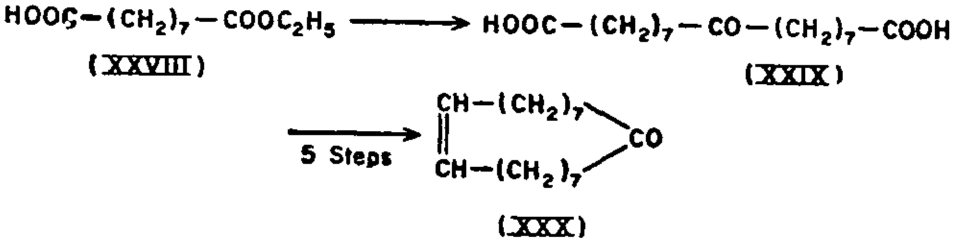
(XXVII)

SYNTHETIC AROMATIC CHEMICALS

Under this group considerable work has been carried out on the development of new practical routes for the synthesis of macrocyclic musk compounds, such as civetone, dihydrocivetone, exaltone, exaltolide and ambrettolide. Macrocyclic musks are widely used in high-grade perfumery preparations and are very expensive. Ketones of this group are of animal origin and the lactones are obtained from plant sources. Using easily available indigenous raw materials such as oleic acid, kamlolenic acid, erucic acid and aleuritic acid, it has been possible to establish practical syntheses for civetone, dihydrocivetone, exaltone, exaltolide, ambrettolide, isoambrettolide, dihydroambrettolide, and several other ketones and lactones. In some cases successful pilot-plant trials have been completed.

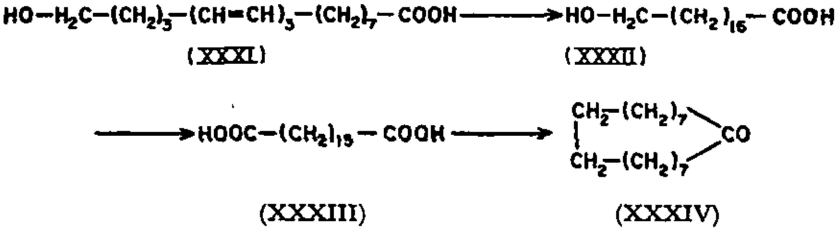
Civetone

For the synthesis of civetone, azelaic acid semi-ester (XXVIII), obtained in high yield from ethyl 9,10-dihydroxystearate, has been converted to civetone dicarboxylic acid (XXIX) by the ketene-dimerization reaction. The ethylene ketal of the diethyl ester of civetone dicarboxylic acid on cyclization and subsequent reactions yielded civetone (XXX):



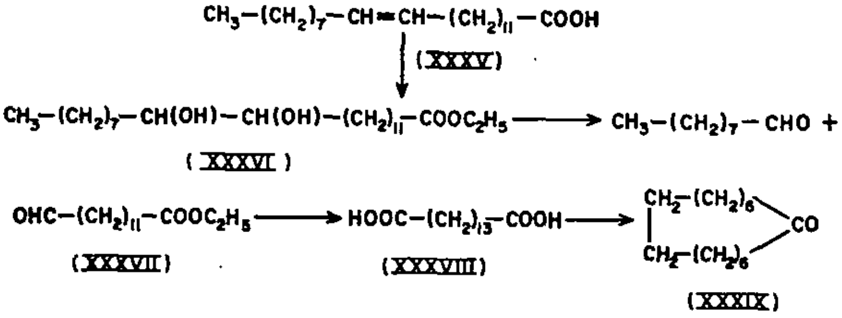
Dihydrocivetone

Dihydrocivetone (XXXIV) has been synthesized from kamlolenic acid (XXXI), which is the main constituent of *kamala* seed oil (*Mallotus philippinensis*). On hydrogenation it gave ω-hydroxystearic acid (XXXII), which was degraded to pentadecane-1,15-dicarboxylic acid (XXXIII) by the Barbier-Wieland degradation, followed by oxidation. The diethyl ester of this acid on cyclization and subsequent reduction gave dihydrocivetone (XXXIV).

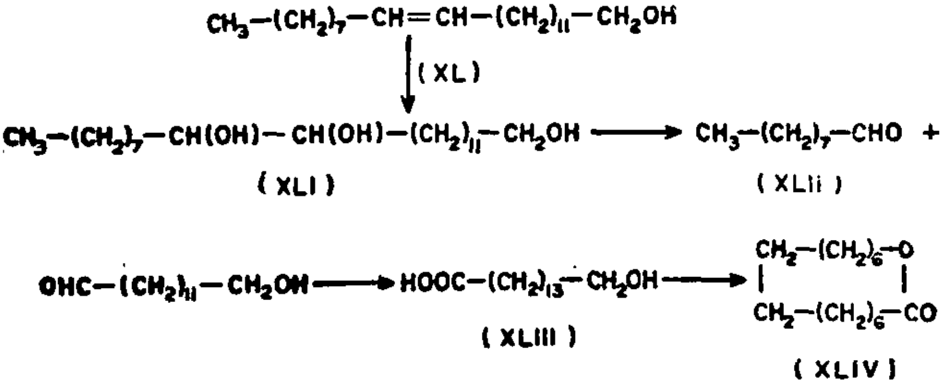


Exaltone and exaltolide

For the synthesis of exaltone and exaltolide, erucic acid (XXXV) obtained from mustard oil was used as the starting material. Ethyl 13,14-dihydroxybehenate (XXXVI), obtained by hydroxylation of ethyl erucate, was converted to ethyl ω-aldehydo-undecane-1-carboxylate (XXXVII) by treatment with sodium metaperiodate. This aldehydo-ester on reaction with malonic acid, ethyl malonate or ethyl bromoacetate and subsequent reactions was converted to tridecane-1,13-dicarboxylic acid (XXXVIII) in excellent yield. Its diethyl ester gave exaltone (XXXIX) on cyclization and reduction.

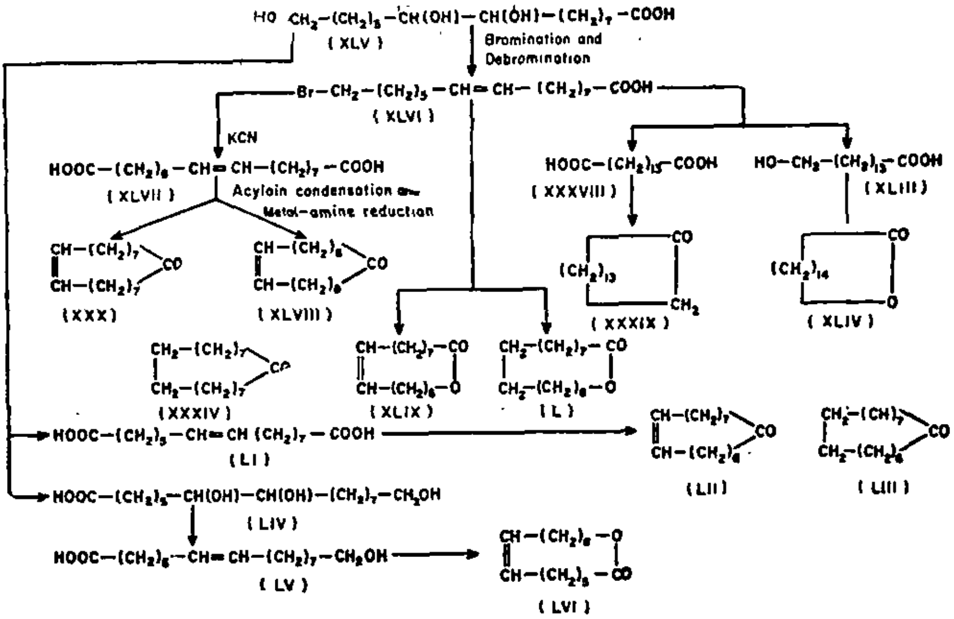


ω-Hydroxy-pentadecane-1-carboxylic acid (XLIII) was synthesized from erucyl alcohol (XL), following a similar procedure. It was lactonized to exaltolide (XLIV) in high yield.



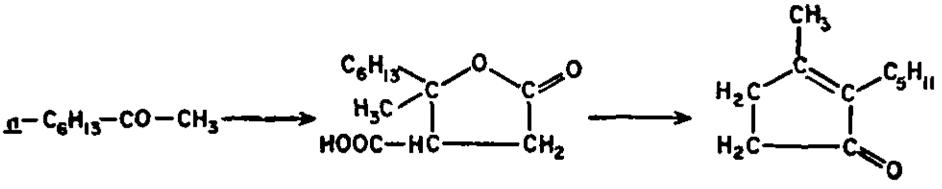
Macrocyclic compounds from aleuritic acid

Aleuritic acid (XLV) obtained from shellac is an attractive raw material for the synthesis of macrocyclic musk-like compounds, since it contains α,ω-functional groups and vicinal hydroxyl groups in the middle of the carbon chain, which provide a means for introducing the necessary double bond. Through a series of reactions depicted in the attached chart, aleuritic acid has been converted to dicarboxylic acids with 15, 16 and 17 carbon atoms and ω-hydroxy acids with 15 and 16 carbon atoms. Cyclization of these acids led to the syntheses of civetone (XXX), isocivetone (XLVIII), dihydrocivetone (XXXIV), cyclohexadecenone (LII), cyclohexadecanone (LIII), exaltone (XXXIX), exaltolide (XLIV), isoambrettolide (XLIX), dihydroambrettolide (L) and ambrettolide (LVI).



Dihydrojasmone

Jasmone, the active principle of the *jasmone concrete*, is an expensive chemical and is difficult to obtain in quantity either from natural sources or through synthetic routes. Its dihydro derivative can be prepared more easily and is also a suitable substitute for the parent compound. A method for the practical preparation of dihydrojasmone has been developed using octan-2-one obtained from castor oil as the starting material. Stobbe condensation of octan-2-one (LVII) with diethyl succinate, followed by cyclization, furnished paraconic acid (LVIII), which was pyrolyzed and cyclodehydrated by treatment with polyphosphoric acid to yield dihydrojasmone (LIX).



(LVII) (LVIII) (LIX)

ENZYMES

Fundamental studies on enzymes were undertaken to elucidate the mechanism of formation of citric acid in *A. niger*, fumaric acid in *Rhizopus nigricans*, reduction of sulphate to sulphur by *Desulfovibrio desulfuricans*, proteolysis by *Bacillus lichenformis*, and metabolism of tricarboxylic acids such as *trans*-aconitic in *Pseudomonas*. In addition the production and properties of enzymes of potential industrial importance, such as bacterial amylase and fungal acylase, were investigated.

Enzymes of Aspergillus niger

All the key enzymes of the glycolytic pathway were found to be present in *A. niger*. Aldolase from the mould was obtained in an electrophoretically homogeneous form with a specific activity comparable to that of the enzymes from yeast or muscle. It was found to be a metalloprotein, which was inhibited by EDTA and reactivated by Zn^{++}, Fe^{++}, Co^{++} and Mn^{++}. The aldolase of several other fungi was also found to require metals for their activity. The *A. niger* phosphohexoseisomerase obtained in a state of high purity, on the other hand, required no cofactors or metals. A TPN-specific glucose-6-phosphate dehydrogenase of high specific activity was also obtained from *A. niger*. It was free from other interfering enzymes and was suitable for the estimation of TPN and glucose-6-phosphate.

Enzymes of Rhizopus nigricans

The presence of all the enzymes of the glycolytic pathway and the tricarboxylic acid cycle was demonstrated in *Rhizopus nigricans*. Fumarase, although present in the mould, had very feeble activity.

Enzymes of Desulfovibrio desulfuricans

Hydrogenase of high purity was obtained from the sulphate-reducing bacteria, *Desulfovibrio desulfuricans*. The purified enzyme was free from cytochrome impurities and was found to require iron for the reduction of methylene blue or benzyl viologen. It was also observed that bacterial cytochrome C_3 was required for the reduction of flavins, but not for methylene blue or benzyl viologen. Sulfurylase from this bacterium was purified and its properties studied. The organism was also found to contain a new enzyme, which catalyzes the liberation of inorganic phosphate from ATP in the presence of sulphite. A soluble enzyme system from *D. desulfuricans* was obtained which catalyzes the conversion of pyruvate to acetyl phosphate, CO_2 and ethanol, and which requires inorganic phosphate for activity. Two different pyrophosphatases, an insoluble enzyme which requires cobalt and a soluble one which requires magnesium, are also present in this bacterium.

Proteolytic enzymes of Bacillus lichenformis

The proteolytic system of *B. lichenformis* was investigated; the proteinase and peptidase fractions were separated and their properties studied.

Aconitate isomerase

A new enzyme was isolated from a soil *Pseudomonas* which catalyzes the conversion of *trans*-aconitate to *cis*-aconitate.

Bacterial diastase

The requirements of diastase for desizing in the textile industry are at present met by imports. An attractive process for the production of bacterial diastase by submerged fermentation using cheap indigenous raw materials was developed; after pilot-plant studies and very favourable reports on the product from several cotton mills, it has been leased to a firm for commercial exploitation.

Fungal acylase

An enzyme which hydrolyses *N*–acyl–L–amino acids was isolated from *Aspergillus oryzae*. The acylase was inhibited by metal chelating agents and reactivated by Zn^{++} or Fe^{++}. The resolution of DL–amino acids was effected by this enzyme, which specifically hydrolyzes the L–isomer of *N*–acyl amino acids. A distinctive property of the enzyme was its high activity with acyl derivatives of tryptophan, which are relatively resistant to hydrolysis by acylases from other sources.

PROTEINS

The work on proteins comprised (a) determination of amino acid composition of several important proteins and peptides, such as phosphopeptones from casein, shark–fin ray elastoidin, some tryptophane-containing peptides obtained by partial hydrolysis of proteins, and silk fibroins, (b) development of transfusion gelatin as a plasma expander, and (c) studies on the physiological role of proteins.

Phosphopeptone from casein

Seven different phosphorus–containing peptides were isolated by paper electrophoresis from bovine casein after digestion with pepsin and trypsin. Significant differences were observed in the composition of the peptides isolated from α–casein and β–casein. The amino acid composition of these peptides was determined.

Shark–fin ray elastoidin

The amino acid composition of shark–fin ray elastoidin was determined by the method of Stein and Moore. This elastoidin was found to contain much higher tyrosine than collagens from other sources. Two peptides were isolated in pure form from the peptic hydrolysates of shark elastoidin and their amino acid composition was determined.

Silk fibroins

The amino acid composition of the fibroins and sericins of mulberry silk and of the three Indian non-mulberry silks, *Tussa, Muga* and *Eri*, was determined. Mulberry fibroin contained more glycine and less alanine than the fibroins of non-mulberry varieties. The sericins differed from the fibroins in having a higher serine, lysine and aspartic acid content and a lower content of glycine and alanine. Ultracentrifugal studies were performed on mulberry silk fibroin in lithium thiocyanate solution. At higher thiocyanate concentation the protein appears to break down to units of M. W. 40,000.

This work was undertaken at the instance of the Central Silk Board which is sponsoring a physico-chemical study on the solution properties and structure of Indian silk.

Tryptophane peptides

Several tryptophane peptides were synthesized and compared with an equivalent amount of free tryptophane in colorimetric and microbiological assays. The results indicated that neither of the two methods can be used with accuracy for the assay of tryptophane in protein hydrolysates which contain part of the amino acid bound as peptide.

Transfusion gelatin

Since blood or plasma is difficult to obtain and preserve on a large scale, the production of plasma substitutes for the treatment of shock has been extensively studied in many countries. A new enzymic process for the preparation of transfusion gelatin was developed on a laboratory scale and the material was found to give satisfactory results in animal tests, which were carried out in collaboration with the Armed Forces Medical College, Poona. Pilot-plant work on the production of transfusion gelatin was undertaken to determine the reproducibility of the preparation in large-scale experiments and to obtain sufficient material for clinical trials, which are in progress to evaluate the efficacy of the product. Pilot-plant investigations on the production of high-grade and edible gelatin from hides have also been carried out.

The physiological effect of low–protein diets (ICMR enquiry)

Physiological functions, such as haemopoesis, basal metabolic rate, reproduction and nitrogen–balance, were studied in several generations of albino rats maintained on low–protein rice diets. Impairment of some of these physiological functions was observed even in the first generation, and it was accentuated in later generations.

MICROBIOLOGY

The principal activities in this field were (a) maintenance of the National Collection of Industrial Micro–organisms, (b) development of a bench–scale fermentation process for citric acid, (c) establishment of semi–continuous techniques for the microbiological production of sulphur, (d) the initiation of a new fermentaion project, the microbiological transformation of terpenes, (e) culture of plant cells and tissues, (f) fundamental studies on mould metabolism and on the mechanism of spore germination, and (g) synthesis and screening of some bacterial compounds.

National Collection of Industrial Micro–organisms

The culture collection was transferred from the Indian Institute of Science, Bangalore, to the NCL in 1951. This collection has increased considerably in size and at present contains more than 1100 bacteria, yeasts, fungi and other non-pathogenic organisms. Cultures from the NCIM are available free of charge to scientific institutions and others. About 2000 cultures have been sent to laboratories in India and abroad during the period under review. A "Directory of Collections of Micro-organisms and List of Species Maintained in India" was published by the CSIR in 1953. A revised Directory for India was compiled and published in 1960 by the Permanent Committee of the British Commonwealth Collections of Micro-organisms in London.

Citric acid

The optimum conditions of citric acid fermentation by surface cultures of *A. niger* on commercial cane sugar have been established after a study of different factors that influence the fermentation. The requirements for trace metals such as Zn and Fe have been defined. In bench-scale pilot runs conducted with polythene trays containing 35 to 50 l. of medium, corresponding to depths of 2 to 3", citric acid yields amounted to 60 per cent of the total sugar. These experiments have been repeated by a firm on a larger scale and the results confirmed.

Attempts to isolate high acid-yielding strains of *Aspergillus niger* which can tolerate higher concentrations of heavy metal ions have not been successful. Cheaper sources of sugar, such as jaggery and molasses, before or after deionization by ion-exchange resins, were poor substrates for citric acid fermentation with the strain used.

Microbiological production of sulphur

A semi-continuous process for the microbiological production of sulphur from gypsum, using sewage sludge as the reducing agent, was developed. The sludge and gypsum are fermented anaerobically at 30° with semi-continuous replacement of the fermenting medium (20 days replacement time) in an air-tight system. The gases in the closed system are recirculated at the rate of 10–12 l./day per l. of sludge. The hydrogen sulphide evolved is swept out and trapped as a metal sulphide. Sterilization of materials and apparatus and adjustment of *p*H are not necessary. Consistent yields of sulphides (1.5–1. 7 g as H_2S/ml raw sludge) were obtained during a period of three months. The replacement time for the medium could be shortened to get a greater amount of sulphide, but the percentage yield became smaller (1.1–1.3 per cent for a replacement time of 10 days). These results are comparable to those reported by Butlin *et al.* from Chemical Research Laboratory, Teddington, England, in pilot plant experiments.

Terpene fermentation

It is known that some essential oil bearing plants, such as *Aquilaria agallocha (agar* wood), yield sweet smelling oleoresin only when heavily infected with micro-organisms, the healthy plant yielding only hydrocarbons. In this context investigations were initiated to study the action of micro-organisms on terpenoid hydrocarbons. A preliminary screening of the strains of fungi infecting *agar* wood and several others available at the NCIM resulted in the selection of a strain of *Aspergillus niger* which oxidized terpenoid hydrocarbons rapidly. *d*–α–Pinene was converted by this mould to *d*–*cis*–verbenol, *d*–verbenone and *d*–*trans*–sobrerol; α–santalene yielded *tere*santalic acid and *tere*santalol. Camphene, longifolene and β–santalene gave a dicarboxylic acid which could be isolated only as the anhydride, $C_{11}H_{16}O_3$. Δ^3–Carene, carane and humulene were comparatively resistant to the action of this mould.

Plant cells

The growth of plant cells by submerged fermentation was investigated in order to study the metabolism of these cells and the formation of useful products characteristic of plant materials. Several plant tissues were grown on semi–solid media, and callus tissue from an indigenous variety of beans, which was characterized by very rapid growth in shake flasks and in a fermentor under submerged conditions, was selected for further study. The nutritional requirements for other plant tissues, such as opium, their growth in submerged culture, and the formation of characteristic plant products, are being investigated.

Mould metabolism

The germination of *A. niger* spores was markedly accelerated in the presence of 0.5 per cent CO_2. Tracer studies with $C^{14}O_2$ revealed that under optimum conditions of citric acid fermentation by *A. niger* in surface culture as much as one–sixth of the total carbon in citric acid may be derived from atmospheric $C^{14}O_2$. It was also found that participation of atmospheric $C^{14}O_2$ in citrate synthesis varies with the age and chemical activity of the mould.

Soluble and insoluble metaphosphates were found to be present in the mycelium and spores of *A. niger*. The variation of the metaphosphates during growth under surface and submerged conditions was studied. The major portion of the labile phosphate of the spores consisted of metaphosphate.

BIOSYNTHETIC PREPARATION OF LABELLED COMPOUNDS

The objective of this project was to devise simple methods of obtaining labelled compounds for use at the NCL. An improved method was developed for the preparation of uniformly labelled glucose and fructose by photosynthesis with tobacco leaves. The radioactive starch was hydrolyzed by salivary amylase and then with dilute mineral acids. Glucose and fructose were separated by an ion–exchange resin catalyzed acetonation, by which fructose was preferentially converted to the acetone–soluble di–isopropylidene derivative.

A new gas circulation apparatus was devised with which labelled formate was prepared in good yields by the reduction of $C^{14}O_2$ with hydrogen in the presence of *E. coli* and $CH_3C^{14}OOH$ from labelled CO_2 by Grignard synthesis.

POLYMERIZATION STUDIES

A systematic investigation in chain transfer by different solvents in the polymerization of monomers, such as styrene, methyl acrylate, methyl methacrylate and vinyl acetate, has been carried out. A kinetic study to determine the role of di-radicals in certain vinyl polymerization reactions has led to useful results. A study of vinyl polymerization in aqueous solution initiated by hydrazine under high vacuum conditions has provided evidence in favour of an initiation reaction involving hydrazyl radicals formed by one-electron oxidation of hydrazine by metallic ions. Studies in temperature dependence of monomer reactivity in copolymerization reactions were carried out and a modified method for deriving reactivity constants was evolved.

SOLUTION BEHAVIOUR OF POLYMERS

Extensive studies in the solution behaviour of certain polymers and natural rubber have been made and certain important conclusions reached. Experimental evidence has been obtained to show that dissolution of rubber in a solvent is a diffusion process in which both components interdiffuse into one another. It has been shown that the Huggins' constant k′ has a unique value at zero shear which is independent of molecular weight for a given polymer, and it is a fundamental property associated with the inherent character of the polymer chains and their interaction with the solvent at a particular temperature. It has also been found that for any polymer the value of k′ is 0.5 at the precipitation point which is independent of temperature and nature of solvent-nonsolvent. It was further concluded that the anomalous increase of a viscosity at high dilutions could be attributed to the extension and disentangling of polymer chains. The viscosity-concentration plots have been found to be influenced by shaking of the rubber solutions prior to viscosity determination. Further experiments with different polymers have suggested that adsorption effects, which were put forward by some workers to explain such anomalies, are insignificant in hydrocarbon polymers, but become important only in polymers with polar groups.

RUBBER

It has been established that chlorination of natural rubber is a four-stage process involving substitution with concomitant cyclization, substitution, addition and finally substitution. It was found that in the hydrochlorination of natural rubber complete cyclization precedes hydrochlorination, and the procedure for employing the hydrochlorination reaction for determining unsaturation in rubber and rubber derivatives is faulty. It has been shown that natural rubber in chlorinated solvents undergoes chemical changes and in the presence of daylight chlorine adds on to the rubber molecules. The changes in chemical structure following cyclization of natural rubber latex have been studied by infrared absorption spectroscopy and a new method for determining unsaturation in cyclized rubber was developed. Conditions for the grafting of methyl methacrylate on rubber molecules by a redox system based on hydrazine were investigated. A definite improvement in the mechanical properties of the vulcazinate prepared from the graft rubber was noted.

A process for the preparation of liquid rubber, which can be used for casting printing rollers and for hard rubber linings, has been leased to industry.

A rubber research group was established in 1959 under the joint auspices of the Council of Scientific and Industrial Research and the Indian Rubber Manufacturers' Research Association. This group has been engaged in problems of immediate interest to the rubber industry, such as the processing of Indian China clays and the preparation of blowing agents and adhesives for various applications.

ION-EXCHANGE

Cation-exchange resins based on phenol, cashewnut shell liquid (CNSL), bhilawan shell liquid, naphthalene and polystyrene have been prepared and their physico-chemical properties investigated. The physico-chemical characteristics of a recently developed anion-exchange resin based on melamine are being studied. A process for the manufacture of cation-exchange resin from CNSL was carried out on a pilot-plant scale, and this resin is now being commercially produced. A detailed study of the use of CNSL cation-exchange resin for water softening was conducted, which has led to its industrial application for water softening. A process for polystyrene based cation-exchange resin is being released to industry.

Diffusion studies with ion-exchange resins included the effect of ionic size and charge in the Donnan diffusion of chloride ions, sorption and desorption of phenol with two cation-exchange resins, and the use of tracer technique for the self-diffusion of rubidium into Amberlite IRC–50. The use of cation-exchange resins as catalysts in the reaction between acetone and iodine and in the inversion of cane sugar has been investigated.

A detailed laboratory examination followed by a bench-scale study in the de-ionization of cane juice, thereby increasing the yield of sugar, was undertaken. The data so obtained formed the basis for a semi-industrial scale investigation which is now in progress at the National Institute of Sugar Technology, Kanpur, under the auspices of the National Research Development Corporation. Electrodialysis technique using membranes prepared in the laboratory has been used in demineralization and desalting applications on a laboratory scale.

COATINGS, ADHESIVES, ETC.

The utilization of fatty acids from certain inedible vegetable oils in the preparation of modified alkyd compositions was investigated. The mechanism of wrinkle formation from such compositions was also studied.

Studies have been also carried out on the preparation of modified urea formaldehyde resins soluble in organic solvents, phenol-formaldehyde and furfural-aniline resins for the preparation of paper-base honeycomb structures, preparation of integrated mica sheets from scrap mica, and utilization of indigenous asbestos in the manufacture of battery containers. Lacquers for food containers based on cashewnut shell liquid have been found to be satisfactory in laboratory tests and are now being submitted to prolonged storage tests. Processes for a can-sealing composition, pressure-sensitive adhesive tapes, and modified urea resins for use in coating textile bobbins have been released to industry. An auto-hardening oil for binding sand cores has been found to be a very satisfactory substitute for imported materials, and details for its production have been made freely available to industry. A process has been developed for making rigid filters for tube wells.

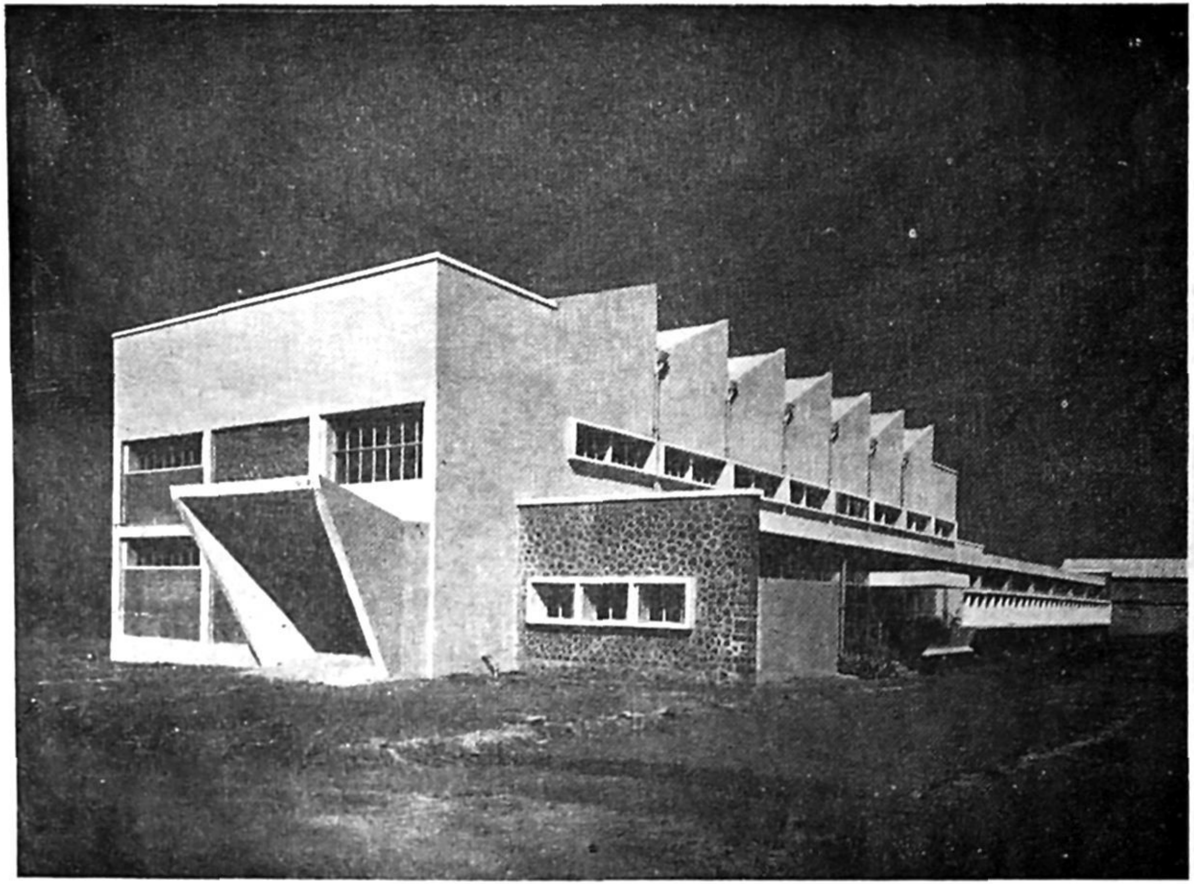
CHEMICAL ENGINEERING

The functions of the Chemical Engineering Division are (a) to supply chemical engineering data and designs for processes developed in other Divisions, (b) to develop processes initiated in the Division from the laboratory to the pilot-plant stage, and (c) to undertake both experimental and theoretical investigations in basic chemical and process engineering. Since one of the main objects of the NCL is to assist the Indian chemical industry by demonstrating the feasibility of known or new reactions and processes for chemical products, a major activity of the Chemical Engineering Division, within the compass of its process development programme, is to design and operate pilot plants. Also, in view of the recent trend in chemical engineering to eliminate the pilot plant as a necessary step in the design of large scale units and to undertake these designs exclusively from basic chemical engineering data, theoretical and experimental studies in certain fields of chemical engineering science have been undertaken as a necessary part of its programme.

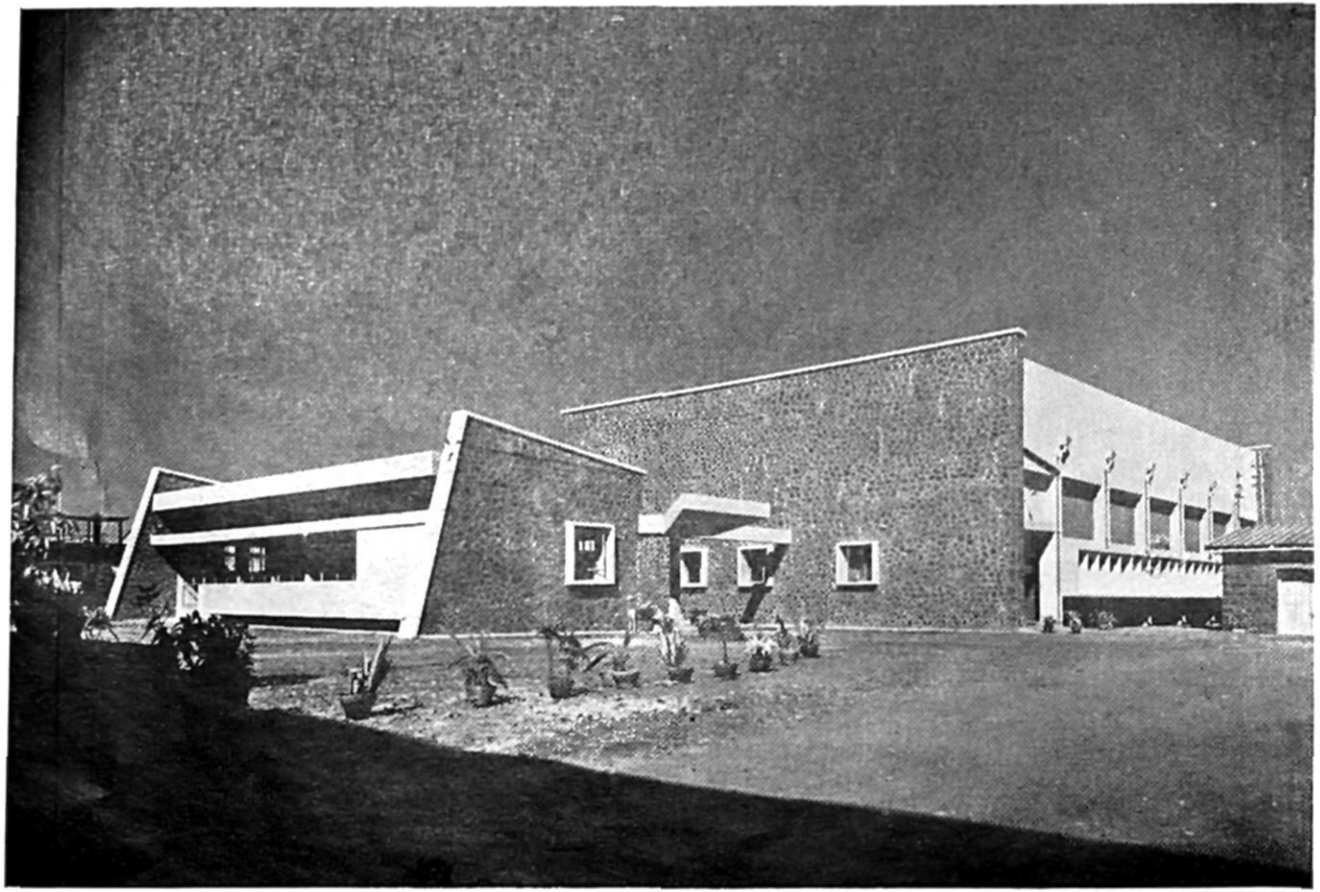
Pilot plant development

A few examples may be cited of processes which have heen released to industry, and for which in most cases complete chemical engineering designs were supplied, including material and energy balances, equipment and material flow sheets, process equipment designs, piping schedules, instrumentation flow sheets, plant lay-outs, detailed cost estimates, and operating instructions.

Extensive pilot-plant work has been carried out for optimising the process conditions in the various steps leading to the synthesis of vitamin C from D-sorbitol. A departure from the established industrial practice at one stage provided considerable scope for chemical engineering studies. The designs for the entire plant are being prepared by Hindustan Antibiotics in collaboration with the Chemical Engineering Division of the NCL.



PILOT PLANT BUILDING, DIVISION OF CHEMICAL ENGINEERING



WORKSHOP

The production of ethylene from ethanol, and ethylene dichloride from ethylene and chlorine, was thoroughly investigated on a pilot-plant scale and commercial designs offered on the basis of the results. Preliminary laboratory investigations had provided the necessary process data on the use of Indian bauxites as catalysts for the dehydration of ethanol to ethylene. Using these results, a pilot-plant for ethylene was designed and set up. For this purpose the design of the various auxiliary units comprising the pilot plant was carried out by conventional methods, while the design of the main unit, the reactor, in the absence of a rate equation, had to be carried out empirically. As a first step, this procedure was considered justifiable since it was anticipated that the data obtained on the pilot plant will be used as the basis for the rational design of a commercial unit. The pilot plant reactor employed a fluidized bed of catalyst, and extensive data were collected on the characteristics of this reactor. Based on these data, a rate equation was developed which was subsequently used for the design of the commercial reactor. The production of ethylene dichloride from ethylene and chlorine was accomplished in a stirred reactor of special design in which the throughput was high at substantially complete chlorine conversion; the exothermic heat of reaction was removed by means of a coil. The effects of mass transfer (agitation), gaseous feed rate and liquid hold-up were extensively investigated, and the commercial reactor designed on the basis of these results.

Batch pilot scale experiments were carried out for each of the steps involved in the production of bacterial diastase and complete designs for the commercial units were supplied.

Nicotine sulphate is an insecticide used in agriculture for combating pests and as a fumigant in hot-houses and green-houses. The usual processes for the production of nicotine sulphate from tobacco and tobacco wastes are based on steam distillation or solvent extraction of nicotine previously released as the base by alkali treatment. The process developed at the NCL is of the latter type and consists of the following operations: drying and grinding of tobacco; mixing of tobacco with alkali to release the nicotine in extractable form; extracting nicotine with water; transferring nicotine from the aqueous extract to kerosene; and fixing the nicotine in the kerosene extract as the sulphate by treatment with sulphuric acid. The necessary chemical engineering data for the solid-liquid and liquid-liquid extraction steps were gathered to facilitate the design of the commercial units. The dryer, grinder, extractors and other equipment were designed on the basis of batch pilot-plant experiments.

Several pilot-plant schemes will be released to industry shortly. Among these may be mentioned: carbon tetrachloride from sewage gas methane; PVC from ethylene dichloride; ethylene chlorohydrin from ethylene and chlorine; ethyl acetoacetate from acetic acid, ethyl alcohol and sodium hydroxide; hexachloroethane from ethylene and chlorine; extraction of costus root oil; and synthesis of vitamin B_6. Of these schemes the chlorohydrin project has been sponsored by a firm, and the extraction of costus root oil is expected to be undertaken on a production basis by the National Research Development Corporation of India at the NCL.

Viscose rayon yarn and staple fibre are finding increasing use in the Indian textile mills and handloom industry, but viscose production is dependent on imported pulp. Investigations on the production of rayon-grade pulp from bamboo, bagasse and indigenous hardwoods were therefore undertaken. A pilot plant to produce about 75 lb. of bleached pulp was designed and the equipment is being assembled. A standard viscose making and spinning pilot plant has been procured for the evaluation of the pulps.

Basic chemical engineering research

Although the major assignment of the Division has been the development of processes and the design and operation of pilot plant, basic investigations within the limitations of time and staff have been carried out in thermodynamics, chemical reaction kinetics, transfer phenomena and transport properties. A series of papers have appeared in well-known chemical engineering journals and a few are in the process of communication.

Generalized procedure for estimating heats of vaporization

Starting from Clapeyron's vapour pressure equation, separate generalized correlations have been developed for the gas and liquid phase contributions appearing in the equation. Using Watson's generalized expression for the evaluation of liquid expansion factor, the following equation was developed using conventional symbols :

$$\frac{L}{T_c} = \frac{4.58 \log p_r}{\int_1^{T_r} (1/zT_r^2)\, dT_r} = \frac{0{\cdot}0244\,(p_r - 1)}{\int_1^{T_r} (\omega/T_r)\, dT_r}$$

A rigorous graphicals solution of this equation was worked out to enable the estimation of the heat of vaporization of a substance from a knowledge of its critical temperature, critical pressure and vapour pressure data. A graph was constructed for L/T_c vs. T_r with p_r as parameter, the ranges covered being 0.5 to 1 for T_r and 0.001 to 0.99 for p_r. The correlation was tested for a variety of substances up to the critical point and found to be very accurate, particularly for organic compounds and elementary (or simple) molecules for which the average error in the use of this correlation is less than 2 per cent.

Estimation of gaseous densities

Reduced density charts have been prepared based on the theorem of corresponding states and on the assumption that the critical ratio RT_c/P_cV_c is constant for a variety of substances. These charts, covering a range of 0.1–30 for p_r and 1–15 for T_r, were developed in two portions, one for high p_r range and the other for low p_r range. A critical density nomogram was also prepared for determining the actual density after estimating the reduced density from these charts. The two charts, together with the nomogram, are very useful in process design where densities of fluids must be estimated at several conditions of temperature and pressure.

Effect of pulsations on the performance of a liquid-liquid extraction column

The performance of a pulsed extraction column has been studied using the system benzene–acetic acid–water in a glass column packed with Raschig rings. HTU values were observed to decrease up to 50 per cent with pulsation. The existence of an optimun amplitude was observed for each frequency, its value varying exponentially with frequency, and a correlation was developed relating pulse frequency and amplitude to HTU at fixed flow rates. Literature data on two other systems were found to conform well with this relationship which is as follows:

$$f = q\,(r - \mathrm{HTU}) + s \log a$$

where q, r and s are constants of a system. The limits of validity were established as :

$$\mathrm{HTU}\ (\text{unpulsed}) > \mathrm{HTU} \geq \mathrm{HTU}\ (\text{min.})$$

.or

$$a\ (\text{optimum}) \geq a > a\ (\text{unpulsed})$$

where a is the amplitude and f the frequency of the pulse.

The effect of varying the disperse phase rate at a low continuous phase rate was also studied and it was found that pulsation resulted in an almost uniform extraction efficiency over a wide range of the dispersed phase rate. Flooding and pressure drop investigations were also completed and the data are being analysed.

Kinetics of the chlorination of ilmenite in a fluidized bed

A method for the chlorination of the iron oxides in ilmenite has been worked out using carbon monoxide and chlorine in a batch fluidization reactor, and optimum conditions for the preferential chlorination were determined. Mass transfer was shown to influence the reaction at gaseous fluid velocities below about 25 ft./min. This indicates that the mass transfer step is resistance to diffusion across the film surrounding the solid particles and not mass transfer between the dense and disperse phases of fluidization which has been suggested by some investigators as an important mass transfer step in fluidized bed reactions.

A rate equation was developed (for the removal of total iron oxides) using batch reactor data by treating this solid-gas, non-catalytic, heterogeneous reaction in a manner analogous to that for a gaseous reaction catalysed by solid surface, but making allowance for the decreasing solids weight. The four rate constants appearing in the equation were individually correlated with temperature according to an Arrhenius type equation. The correlations developed are for the removal of iron oxides only so that it should be possible to completely isolate the titanium content of ilmenite from its ferric impurities.

The results of this investigation demonstrate the use of batch rate data in the design of continuous reactors and *vice versa.* Assuming *a* to represent the moles of Cl_2/min. in the feed, *b* the moles/min. of CO_2 and *x* the conversion of iron oxides, W/F (where W is the weight of ilmenite and F the total gaseous feed rate) is given by :

$$\frac{W}{F} = B^2 x - \frac{E}{a+b} \ln (a - \overline{a+b}\, x) - \frac{F'}{a+b} \ln (b - \overline{a+b}\, x) + \frac{E}{a+b} \ln a + \frac{F'}{a+b} \ln b$$

Where B, E, F′ are integration contants related to the four rate constants of this reaction.

Performance of a pulsed heat exchanger

The object of employing any type of agitation in a heat transfer equipment is to reduce the film thickness of the fluid or, in an extreme case, to break the film, thus decreasing the thermal resistance across it. One of the methods of imparting agitation is the use of pulsations or vibrations. Since the early patent on the subject by Andreas in 1942, surprisingly little has appeared in the literature on the performance of pulsed heat exchangers. The fragmentary data available are contradictory. An investigation was therefore undertaken to determine the effect of pulsation on heat transfer. Employing three specially designed tube-and-shell heat exchangers to cover a wide range of Reynolds numbers, overall heat transfer coefficients were obtained at frequencies of 40, 81 and 160 cycles per minute, the pulses being damped to various degrees to provide variation in amplitude at each frequency. The results clearly showed that pulsations enhance the heat transfer coefficient in the turbulent region. For the type of pulsation resulting from the use of a reciprocating pump, the broad observations were: (a) unless the pulse frequency exceeds a certain value, the effect is not perceptible; (b) the improvement in U due to pulsation increases as the Reynolds number is increased, the effect being almost negligible beyond a certain Reynolds number; and (c) the extent of pulse dampening (i.e. amplitude) at any frequency has no effect on heat transfer rate.

Thermal dehydrochlorination of ethylene dichloride

The complete kinetics of the thermal dehydrochlorination of ethylene dichloride to vinyl chloride and acetylene has been investigated, considering also their pyrolysis to carbon and hydrogen. The experiments were carried out in a specially designed reactor which ensured isothermal conditions in the reaction zone. The data were analysed on the basis of overall reaction rates estimated in terms of the rate of disappearance of ethylene dichloride. Two plots were developed, one for the high conversion range (low space velocity) and the other for the low conversion range (high space velocity). The correlations were based on the relationship between reciprocal space velocity and log $(1/1-x)$ in the high conversion range, and between $1/\log(1/1-x)$ and space velocity in the low conversion range. This method of plotting is useful since in each case the curve can be extrapolated to zero on the space velocity co-ordinate.

Having obtained rate equations for the two ranges of overall conversion, conversion to vinyl chloride was estimated in terms of selectivity which was defined as the ratio of conversion to vinyl chloride to overall conversion. It was found that the selectivity at each temperature was practically independent of space velocity. Thus an overall equation was developed for the rate of disappearance of ethylene dichloride in terms of the usual Arrhenius constants, and a straightline correlation of selectivity and temperature enabled the incorporation of selectivity also in this equation. Using these equations, the problem of designing a coil type reactor for the thermal decomposition of ethylene dichloride to vinyl chloride can be rigorously approached by any of the suggested methods in the literature.

Thermal decomposition of calcium carbonate

The process of thermal decomposition of calcium carbonate has been treated as one governed by simultaneous heat and mass transfer under quasi steady state conditions. Rigorous expressions, based on heat transfer rate, were derived using the properties of the system for the prediction of decomposition times for definite geometrical shapes like spheres and cylinders. The usual shell model was assumed as the basis for the decomposition scheme and the following expressions developed:

Spheres : $\theta_D = \alpha\,(0.33 + 0.17\,K_H)$

Cylinders : $\theta_D = \alpha\,(0.50 + 0.25\,K_H)$

Where θ_D = decomposition time

K_H = modified Nusselt Number

Based on an analysis of simultaneous heat and mass transfer, an equation was obtained which bears close resemblance to the theoretical expression for the evaporation of small spheres into quiet air. This equation was rearranged to give:

$$\frac{h}{k_g} = \frac{C_p}{E'}\;\frac{(\mu/\rho\,D_m)}{(C_p\,\mu/k_c)}$$

Where E' = porosity factor, all the other symbols
being conventional,

which is a modified form of the usual correlation of mass transfer based on the Chilton-Colburn analogy.

The mathematical treatment was extended to a two phase solid system ($MgCO_3-CaCO_3$) characterized by finite but different equilibrium decomposition temperatures. The mathematical analysis involved the solution of simultaneous differential equations; this was done by the modified Rünge-Kutta method with the help of an IBM 650 computer. The equations developed for the single phase system ($CaCO_3$) were found to represent the experimental data reported from the Chemical Engineering School at M. I. T. remarkably well; for the two phase system experimental data are not available for confirming the proposed equations.

The reactor unit concept in process design

One of the important variables required to be computed in the design of a catalytic reactor is the weight of catalyst necessary for a given duty. The use of the several available generalized procedures requires a knowledge of the rate equation for the reaction under consideration, and often such a rate equation may not be readily available. Further, economic considerations may point to the use of a space velocity at which mass transfer effects cannot be neglected. Indeed this may quite often be the case as is apparent from the fact that, for a given catalyst weight, increasing the space velocity (for decreasing the mass transfer resistance) results in a corresponding increase in pressure drop leading to a rise in operating cost, which may more than compensate the attendant decrease in reactor cost due to a decrease in reactor diameter. It may thus be seen that an approach based on a simultaneous but independent evaluation of the kinetic and mass transfer effects (without requiring a rate equation) would be of considerable use in reactor design.

Thus, based on the original concept of the height of a reactor unit, first introduced by Lampichler and later elaborated by Hurt, a complete theoretical analysis was made of the various steps in a catalytic reaction of any order, and a method was worked out for the simultaneous evaluation of the resistance to mass transfer from the fluid bulk to the catalyst, the so-called chemical resistance, and the resistance to mass transfer from the catalyst surface to the fluid bulk. These various resistances were expressed in terms of height units, and the overall equation expressed as

$$\mathrm{HRU} = \mathrm{HTU}_1 + \mathrm{HCU} + \mathrm{HTU}_2$$

where the middle term may be identified as the height of reactor unit and the other two terms as the heights of transfer unit for the two directions of transfer. In estimating the mass transfer resistance for the products, the concept of effective partial pressures (due to inadequate residence time) was introduced and a contact efficiency was defined. The proposed method has been illustrated by evaluating the three resistances in an SO_2 converter.

Correlation of liquid phase properties

The failure of the strict application of statistical mechanics to yield a complete theory of liquids has resulted in a number af attempts to *construct simplified models* which would permit the representation of the more important features of the liquid state based on satisfactory mathematical analyses. Using one such model, Eyring and coworkers developed "a free volume" theory of the liquid state which has been satisfactorily applied to the theory of transport phenomena in liquids. Based on this concept of Eyring, a correlation was developed interrelating some of the important liquid phase properties like surface tension, viscosity, molecular volume, and energy of vaporization, within a temperature range of 0^0 to 50^0. The basis of the correlation was the dual expression for the free volume, one of them being related to the velocity of sound in liquids. The equation was derived as:

$$\frac{U_L \eta V_m^{2/3}}{MT^{1/2}} = 9{\cdot}27 \text{ exp.} \frac{1.091 N^{1/3} \sigma V_m^{2/3}}{RT}$$

$$\text{and } \triangle E_{vis} = \frac{\triangle E_{vap}}{n} \cdot \frac{0{\cdot}55 N^{1/3} \sigma V_m^{2/3}}{RT}$$

where ΔE_{vis} is the activation energy for viscous flow, all other symbols being conventional. The correlation was tested with data for about a dozen representative liquids and was found to bear out the validity of the derivation procedure.

Catalytic reduction of d-glucose to d-sorbitol in aqueous medium

Liquid phase reduction of d-glucose to d-sorbitol with suspended Raney nickel catalyst has been studied under conditions where mass transfer effects were effectively shielded. An equation relating the initial reaction rate to the important process variables was derived:

$$-R_0 = 0{\cdot}508 \times 10^4 \text{ exp.} - \frac{2600}{T} C_{OG} \cdot C_C \cdot P_{H_2}^{1/2}$$

where R_0 = initial reaction rate;
C_{OG} = initial glucose concentration;
C_C = catalyst concentration;
P_{H_2} = effective H_2 partial pressure.

The catalytic reduction was presumed to be effected by a process of slow dissociative chemisorption of hydrogen atoms on the catalyst surface.

In addition the effect of the following variables was studied: initial glucose concentration, RPM of stirrer, and use of mixtures of ethanol-water as solvent for glucose. It was found that increase in glucose concentration (20 to 60 per cent) had negligible effect on the initial reaction rate provided the catalyst mass used was based on the glucose content. It was also observed that above a stirrer RPM of 600 the mass transfer effects were negligible and that increasing the concentration of ethanol in the solvent mixture increased the initial reaction rate.

APPENDIX I

Executive Council as on 31-3-1960

Shri P. A. Narielwala, Director, Tata Industries (Pvt.) Ltd., Bombay. (*Chairman*)

Shri Charat Ram, Delhi Cloth and General Mills Co. Ltd., Delhi.

Dr. K. A. Hamied, Cipla, Bombay.

Dr. B. C. Guha, University College of Science and Technology, Calcutta.

Dr. B. P. Godrej, Godrej Soaps (Pvt.) Ltd., Bombay.

Dr. D. K. Banerjee, Indian Institute of Science, Bangalore.

Dr. M. R. Mandlekar, Director of Industries, Government of Maharashtra, Bombay.

Dr. G. P. Kane, Development Wing, Ministry of Commerce and Industry, New Delhi.

Major-General Partap Narain (Retd.), Utkal Machinery (Pvt.) Ltd., Rourkela.

Dr. Jagdish Shankar, Atomic Energy Establishment, Bombay.

Dr. D. Banerjee, Vice-President, Indian Rubber Manufacturers' Research Association, Calcutta.

Shri Arvind N. Mafatlal, Bombay.

Director-General, Scientific and Industrial Research.

Financial Adviser to CSIR.

Director, NCL.

APPENDIX II

Staff of the National Chemical Laboratory in Post on 31st March 1960

Director

K. Venkataraman, D.Sc.

Deputy Director

R. C. Shah, Ph.D.

Assistant Directors

S. C. Bhattacharyya, Ph.D.
A. B. Biswas, Ph.D.
J. Gupta, D.Sc.
V. Jagannathan, Ph.D.
S. L. Kapur, Ph.D.
K. G. Mathur, D.Sc.
M. U. Pai, Ph.D.
Sukh Dev, D.Sc.

Senior Scientific Officers (Grade I)

P. K. Bhattacharyya, Ph.D.
L. K. Doraiswamy, Ph.D.
S. D. Mahulikar, B. E.
H. B. Mathur, Ph.D.
B. C. Subba Rao, Ph.D.
Uma Shankar, Ph.D.

Senior Scientific Officers (Grade II)

S. C. Banerjee, M.Sc.
Bhola Nath, Ph.D.
J. L. Bose, D. Phil.
M. K. Gharpurey, Ph.D.
A. Goswami, Ph.D.
M. Goswami, Ph.D.
B. S. Joshi, Ph.D.
N. Krishnaswamy, Ph.D.
N. K. Maitra, M.Sc.
S. S. Malhotra, Ph.D.
G. Narasimhan, Ph.D.
C. R. Narayanan, D. Phil.
M. R. Raghavendra Rao, Ph.D
J. C. Sadana, Ph.D.
D. N. Sen, Ph.D.
A. P. B. Sinha, Ph.D.
K. P. Sinha, Ph.D.
C. Sivaraman, Ph.D.
G. M. Vyas, Ph.D.

Junior Scientific Officers

I. J. Babbar, Ph.D.
D. S. Bendale, M.Sc.
H. L. Bhatnagar, Ph.D.
K. K. Chakravarty, D. Phil.
N. L. Dutta, D. Phil.
N. D. Ghatge, Ph.D.
R. V. Gopala Rao, Ph.D.
T. R. Ingle, Ph.D.
G. R. Kelkar, Ph.D.
M. S. Kothari, M.S.
M. N. Krishnamurthy, B.Sc. (Chem. Tech.)
Miss S. B. Kulkarni, Ph.D.
M. V. Kunte, M.Sc.
Lakhbir Singh, M.Sc.
Mansa Ram, Ph.D.
A. U. Momin
P. M. Nair, Ph.D.
L. M. Pant, Ph.D.
G. V. Potnis, M.Sc.
S. K. Pradhan, Ph.D.
B. V. Ramachandran, Ph.D.
P. N. Rangachari, Ph.D.
A. S. Rao, Ph.D.
P. R. Subbaraman, Ph.D.
R. V. Subramanian, Ph.D.
S. S. Subramanian, M.Sc.
J. P. Varma, Ph.D.
V. Yatirajam, Dr. rer. Nat.

Officers (Technical)

R. V. Kulkarni, Dip. M.E. & E.E.
Workshop Superintendent
V. S. N. Sarma, B.Sc.
Stores Officer
M. K. Joshi, M.B.B.S.,
Resident Medical Officer

Pool Officers

V. V. Ghaisas, Ph.D. (Tech.)
S. N. Kulkarni, Ph.D.
M. S. Narasinga Rao, Ph.D.

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B. S. Baliga, M.Sc.
N. A. Bhat, B.Sc.
D. C. Bigg, B.Sc. (Tech.)
P. H. Brahme, B.Sc. (Tech.)
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A. K. Chatterjee, M.Sc.
V. Damodaran, M.A.
A. V. Deo, M.Sc.
A. D. Deshpande, B.Sc. (Tech.)
R. S. Dhavalikar, M.Sc.
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B. B. Ghatge, Ph.D.
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C. I. Jose, M.Sc.
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S. K. Kundra, M. Sc. (Hons.)
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C. G. Lele, M.Sc.
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A. Raja Rao, M. Tech.
D. R. V. Ramanan, B.Sc.
R. Rama Rao, Ph.D.
B. Ranganathan, Ph.D.
C. D. Sabane, Ph.D.
N. R. Sanjana, Ph.D.
S. C. Sethi, M.Sc.
P. G. Sharma, M.Sc.
D. K. Shastri, M.Sc. (Tech.)
M. C. Srinivasan, Ph.D.
A. V. Subbaratnam, M.Sc. (Pharm.)
M. K. Unni, Ph.D. (Tech.)
J. R. Vakil, Ph.D.
H. G. Vartak, M. Sc.
B. G. Zambre, B.Sc.

Junior Scientific Assistants

S. M. Abhyankar, B.Sc. (Tech.)
A. S. Bawdekar, M.Sc.
H. B. Bhat, M.Sc.
G. L. Chetty, M.Sc.
S. Chidambaram, B.Sc.
P. R. Chinchole, M.Sc.
N. P. Damodaran, M.Sc.
B. D. Dasare, M.Sc.
A. K. Dhavale, M.Sc.
R. G. Gokhale, M.Sc.
P. Govindankutty, B.Sc.
S. K. Gupta, M.Sc.
V. K. Hinge, M.Sc.
Mrs. P. Jose, M.Sc.
T. C. Joseph, B.Sc. (Hons.)
N. M. Joshi, Dip. Radio Eng.

R. S. Joshi, M.Sc.
U. N. Kantak, B.Sc.
V. H. Kapadia, M.Sc.
K. B. Kaushal
V. Krishnan, B.Sc.
S. Krishnappa, B.Sc.
G. H. Kulkarni, M.Sc.
J. Lobo, B. Sc. (Hons.)
M. L. Maheshwari, M.Sc.
A. F. Mascarenhas, B.Sc.
S. R. Modak, M.Sc.
V. R. G. Moorti, M.Sc.
A. V. Morey, M.Sc.
V. N. Mulay, B.Sc.
M. N. S. Murthy, M.Sc.
Miss D. M. Nair, B.Sc.
D. D. Nanavati, M.Sc.
C. S. Narayanan, M.Sc.
V. G. Neurgaonkar, M.Sc.
S. K. Pandit, M.Sc.
N. J. Parthasarthy, B.E. (Chem.)
S. G. Patnekar, M.Sc.
L. D. Pethe, M.Sc.
V. B. Phadke, M.Sc.
S. Ramanathan, B.Sc. (Tech.)
S. K. Ramaswami, B.Sc.
A. V. Rao, B.Sc. (Tech.)
C. J. Sanchorawala, B.Sc. (Tech.)
T. S. Santhanakrishnan, M.Sc.
P. S. Sarfare, M.Sc.
V. P. Saxena, M.Sc.
A. Shanmugasundaram B. E. (Chem.)
P S. Shetty, M.Sc.
R. N. Shukla, B.Sc.
R. Srinivasan, M.Sc.
S. R. Srinivasan, M.Sc. (Tech.)
T. K. K. Srinivasan, M.Sc.
Miss K. L. Surve, M.Sc.
N. P. Suryanarayanan, M.Sc.
K. R. Varma, M.Sc.
N. V. Viswanathan, B.Sc.
M. S. Wadia, M.Sc.

Assistants (Technical)

K. D. Kaushal, Dip. M.E.,
Principal Foreman
M. K. Kelkar, B.Sc., Dip. Lib.,
Librarian
Niranjan Singh,
Chief Glass Blower Supervisor
D. C. Trama, B.Sc.
Head Laboratory Supervisor
D. V. Singh,
Laboratory Supervisor
V. S. Iyer,
Stores Supervisor
Tirath Ram, B.A.
Stores Supervisor

Senior Research Fellows

P. S. Aggarwal, Ph.D.
Miss S. Kanthamani, M.Sc.
C. C. Menon, Ph.D.
U. K. Pandit, Ph.D.
Mrs. S. Srikanta, Ph.D.
L. Viswanathan, Ph.D

Junior Research Fellows

R. B. Badachhape, M.Sc.
S. B. Badachhape, M.Sc.
K. I. Bhatia, M.Sc.
S. B. Bhatia, M.Sc.
A. B. Deshpande, M.Sc..
A. C. Ghosh, M.Sc.
D. D. Godse, M.Sc.
A. S. Gulati, M.Sc.
R. K. Gupta, M.Sc.
S. A. Kagal, B.Sc.
K. K. Laroia, M.Sc.
B. A. Nagasampagi, M.Sc.
M. S. R. Nair, M.Sc.
S. K. Paknikar, M.Sc.
R. C. Pandey, M.Sc.
R. Pratap, B.E. (Chem.)
J. R. Prehlad, M.Sc.
A. V. Rama Rao, B.Sc. (Tech.)
Mrs. S. K. Ramaswami, M.Sc.
M. V. Ramiah, M.Sc.
V. C. Rane, B.Sc. (Tech.)
A. M. Shaligram, M.Sc.
V. S. Sharma, M.Sc.
M. K. Sinha, M.Sc.
R. Srinivasan, B.Sc. (Tech.)
S. S. Tavale, M.Sc.
S. A. Telang, B.Sc.
U. N. Upadhyaya, M.Sc.
K. K. Vijay, M.Sc.

APPENDIX III

PUBLICATIONS

1950

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APPENDIX IV

PATENTS

1. *Ind. Pat.* 41338
   A plastic material from shellac and resinols like bhilawan shell liquid or cashew shell liquid. SIDDIQUI, S., SARIN, K. K. and VARMA, J. P.
2. *Ind. Pat.* 41339 [*U. S. P.* 2,615,855]
   Moulding powders from shellac and resinols like bhilawan shell liquid or cashew shell liquid. SIDDIQUI, S., SARIN, K. K. and VARMA, J. P.
3. *Ind. Pat.* 41340 [*U. S. P.* 2,707,686]
   Improvements in or relating to the production of moulding powders from shellac and bhilawan resins. SIDDIQUI, S., SARIN, K. K. and VARMA, J. P.
4. *Ind. Pat.* 41878
   Manufacture of heat-insulating light-weight padding material for roofing boards or the like from agricultural waste. SIDDIQUI, S., SARIN K. K., VARMA, J. P. and DAYAL, P.
5. *Ind. Pat.* 43292
   Improvements in or relating to the manufacture of emulsifying compositions. CHAKRAVARTI, K. K. and SIDDIQUI, S.
6. *B. P.* 650342
   Improvements in or relating to the conversion of bhilawan shell liquid to a non-vesicating drying product for the manufacture of lacquer varnish, stoving enamels, water-proofing or like materials. SIDDIQUI S. and DHAR, D. C.
7. *B. P.* 653037
   Improvements in or relating to the conversion of bhilawan shell liquid to a non-vesicating drying product for the manufacture of air-drying varnish compositions, enamels or the like. SIDDIQUI, S.
8. *Ind. Pat.* 42885
   Preparation of modified oils from vegetable oils for use in paints and varnishes. AGGARWAL, J. S., BUDHIRAJA, N. C. and SHARMA, P. G.
9. *B. P.* 657695
   Improvements in or relating to compositions particularly suited for use in the manufacture of patent leather. SIDDIQUI, S. and DHAR, D. C.
10. *Ind. Pat.* 43164
    Improvements in or relating to the manufacture of coating compositions. AGGARWAL, J. S. and SETHI, S. C.

11. *U. S. P.* 2,591,623
Process for the production of plastic material from shellac and resinols like bhilawan shell liquid or cashew shell liquid, suitable for making moulding powders, coating compositions, adhesives or the like. SIDDIQUI, S., SARIN, K. K. and VARMA, J. P.

12. *Ind. Pat.* 43544
Preparation of modified glycerol phthalate alkyds. KAPOOR, S. L. and SARIN, K. K.

13. *Ind. Pat.* 43979
Improvements in and relating to the production of wrinkle finishes. KAPUR, S. L. and SARIN, K. K.

14. *Ind. Pat.* 43827
Preparation of cation exchange resins. SHAH, H. A. and BAFNA, S. L.

15. *Ind. Pat.* 44359
Preparation of ion exchange resins and the uses thereof. SHAH, H. A. and BAFNA, S. L.

16. *B. P.* 684328
Improvements in or relating to manufacture of inks for stamping, printing, stencilling or like purposes. SIDDIQUI, S., AGGARWAL, J. S. and MATHUR, S. K.

17. *Ind. Pat.* 44737 [*B. P.* 734576]
Air-drying wrinkle finish coating compositions. AGGARWAL, J. S. and SHARMA, P. G.

18. *Ind. Pat.* 47179
Improvements in or relating to sugar cane wax. KULKARNI, A. B.

19. *Ind. Pat.* 49022
A latex composition particularly suited for can-sealing purposes. BALLABH, J. and RAGHUNATH, D.

20. *Ind. Pat.* 45116
The preparation of new bactericidal and surface active quaternary ammonium compounds. DAMODARAN, M. and SIVARAMAN, C.

21. *Ind. Pat.* 46994
Extraction of nicotine and an extraction tower for the purpose. GEDEON, J.

22. *Ind. Pat.* 45666 [*Turk. Pat.* 6542].
Manufacture of nicotine sulphate from tobacco or tobacco wastes. GEDEON, J. and GOSWAMI, M.

23. *Ind. Pat.* 49033
Treatment of hides for production of gelatin or glue. KINCL, F. A. and POTNIS, G. V.

24. *Ind. Pat.* 47382
A process for the modification of cashew nut shell liquid. AGGARWAL, J. S. and MATHUR, H. H.

25. *Ind. Pat.* 47439
A process for the manufacture of a mixed nitrogen-phosphorus fertiliser. GADRE, G. T. and GUPTA, J. N.

26. *Ind. Pat.* 48529
A process for the thermal degradation of the seed oil of nim (*Melia azadirachta*, *Melia indica*). MITRA, C. R.

27. *Ind. Pat.* 46713
Utilization of the seed oil of nim for the production of oleic acid, stearic acid, palmitic acid and/or stearin. MITRA, C. R.

28. *Ind. Pat.* 46947
Isolation and separation of bitter principles from the trunk and/or root bark of nim. BHATTACHARJI, S. and MITRA, C. R.

29. *Ind. Pat.* 48530
Improvements in or relating to the utilization of the seed oil of nim. MITRA, C. R.

30. *U. S. P.* 2,666,717
Patent leather and coating composition for use in making the same. SIDDIQUI, S. and DHAR, C. C.

31. *Ind. Pat.* 47766
Cation exchangers. BAFNA, S. L. and SHAH, H. A.

32. *Ind. Pat.* 47817
A process for the manufacture of sodium tripolyphosphate. GADRE G. T., GHARPUREY, M. K. and GUPTA, J.

33. *Ind. Pat.* 48275
Waterproofing of boards used for sound-proofing purposes. RAO, N. V. C. and PANDE, J. B.

34. *Ind. Pat.* 47802
Purification and refining of non-edible, non-drying and so-called medicinal oils, such as Karanja, Polang, and Nageswar. MITRA, C. R.

35. *Ind. Pat.* 47815
Liquid preparations, such as paints or the like coating compositions. MCBAIN, J. W. and PANDE, J. B.

36. *Ind. Pat.* 47816
Duplicating inks. MCBAIN, J. W. and PANDE, J. B.

37. *Ind. Pat.* 48640
Preparation of starch from starch-bearing plant materials. DAMODARAN, M. and BABBAR, I.

38. *Ind. Pat.* 50758
A process for the preparation of modified alkyl resins. AGGARWAL, J. S; and SHARMA, P. G.

39. *U. S. P.* 2,685,593
Manufacture of useful compounds and products from the seed oil of *Mallotus philippinensis* Muell. Arg. (Kamala). AGGARWAL, J. S., SHARMA, V. N. and GUPTA, S. C.

40. *Ind. Pat.* 50483
    Soft iodo-bromide positive emulsions. CHATTERJEE, D. and GUJRAL, J. S.
41. *Ind. Pat.* 49364
    The manufacture of adhesive tapes. RAO, N. V. C.
42. *Ind. Pat.* 52014
    Preparation of halogenated rubber. RAMAKRISHNAN, C. R. and RAGHUNATH, D.
43. *Ind. Pat.* 49852
    Manufacture of azo dyestuffs from cashew shell liquid derivatives. PANSARE, V. S. and KULKARNI, A. B.
44. *Ind. Pat.* 52027
    Composition for preparing cylindrical rolls from washed safety base cinematographic films. MATHUR, K. G. and LELE, A. M.
45. *Ind. Pat.* 50926
    Manufacture of nicotinic acid. SHAH, G. D. and SHAH, R. C.
46. *Ind. Pat.* 52028
    A cement composition for jointing safety base cinematographic films. MATHUR, K. G. and LELE, A. M.
47. *Ind. Pat.* 52112
    Improvements in or relating to cast synthetic resins. SHAH, H. A.
48. *Ind. Pat.* 53636
    Improvements in or relating to coating compositions. SARIN, K. K. and KAPUR, S. L.
49. *Ind. Pat.* 52801
    A process of preparing a modified gelatin suitable for transfusion. DAMODARAN, M. and SINGH, G.
50. *Ind. Pat.* 53414
    Treatment of molasses to render it suitable for use in phenolic–type moulding compositions. GHOSH, A.
51. *Ind. Pat.* 54395
    A process for the manufacture of active manganese dioxide suitable for dry batteries. AGARWALA, U. C., SANJANA, N. R. and GUPTA, J.
52. *Ind. Pat.* 54713
    A process for obtaining alkali fluoride from alkaline earth fluoride and/or fluorine bearing minerals like fluorspar. SINGH, M. M. and GUPTA, J.
53. *Ind. Pat.* 54828
    Process for the manufacture of tri–ammonium phosphate. SESHADRI, K. and GUPTA, J.
54. *Ind. Pat.* 54829
    Process for the manufacture of an ammonium phosphate fertilizer from tri–ammonium phosphate. SESHADRI, K. and GUPTA, J.
55. *Ind. Pat.* 55172
    A process for the metallization of waste cinematographic film. MATHUR, K. G. and LELE, A. M.

56. *Ind. Pat.* 55171
Treatment of cashew nut shell liquid for use in electrical insulating varnishes. Thakar, K. R. and Gupta, J.

57. *Ind. Pat.* 55546
Improvements in or relating to ion-exchange materials. Ramakrishnan, C. S. and Krishnaswamy, N.

58. *Ind. Pat.* 54959
An electrical device for double action on/off control. Sastry, S. L.

59. *Ind. Pat.* 54867
A process for the manufacture of nicotine sulphate from tobacco and tobacco wastes. Bijawat, H. C., Razdan, R. and Potnis, G. V.

60. *Ind. Pat.* 56724
A water dispersible DDT paste. Rao, K. V. N., Bhide, S. P., Kulkarni, (Miss) S. B. and Biswas, A. B.

61. *Ind. Pat.* 56725
A new process for the purification of selenium. Sen, D. N. and Gupta, J.

62. *Ind. Pat.* 55757
A process for the preparation of long-chain unsaturated ketones and 1-ω-ketodicarboxylic acids from the said ketones. Menon, B., Nayak, U. G., Razdan, R. K., Chakravarti, K. K. and Bhattacharyya, S. C.

63. *Ind. Pat.* 56391
A process for the preparation of civetone dicarboxylic acid. Nayak, U. G., Chakravarti, K. K. and Bhattacharyya, S. C.

64. *Ind. Pat.* 56726
Preparation of water dispersible DDT as an oil bound paste. Kulkarni, (Miss) S. B., Biswas, A. B., Rao, K. V. N. and Bhide, S. P.

65. *Ind. Pat.* 57541
A process for the preparation of dihydrojasmone. Razdan, R. and Bhattacharyya, S. C.

66. *Ind. Pat.* 58203
Improvement in or relating to the preparation of ethyl ester of musk acid, 2-methyl tridecane - 1:13 - dicarboxylic acid. Chakravarti, K. K. and Bhattacharyya, S. C.

67. *Ind. Pat.* 58529 [*B. P.* 8772/57]
An improved process for the preparation of β-ionone. Krishna, H. J. V. and Joshi, B. N.

68. *Ind. Pat.* 59457
A new process for preparation of fine dusts or wettable powders of insecticides such as DDT. Kulkarni, (Miss) S. B., Kolhatkar, P. S., Biswas, A. B., Rao, K. V. N. and Kuber, M. V.

69. *Ind. Pat.* 59608
Porous rigid filters. Kapur, S. L. and Pandya, R. N.

70. *Ind. Pat.* 57888
Improvements in or relating to the production of hydroxy alkoxy, or aryloxy substituted aryl alkyl ketones. Bose, J. L. and Shah, R. C.

71. *Ind. Pat.* 58868
A process for the preparation of azelaic acid semi-ester suitable for making civetone dicarboxylic acid. NAYAK, U. G., CHAKRAVARTI, K. K. and BHATTACHARYYA, S. C.

72. *Ind. Pat.* 59927
A process for the preparation of pentadecane-1:15-dicarboxylic acid or its ester suitable for the preparation of dihydrocivetone. NAYAK, U. G., CHAKRAVARTI, K. K. and BHATTACHARYYA, S. C.

73. *Ind. Pat.* 60555
Production of liquid rubber. UMA SHANKAR.

74. *Ind. Pat.* 59419
A process for the preparation of tridecane-1:13-dicarboxylic acid or its ester, suitable for the preparation of exaltone (cyclopentadecanone). GHATGEY, B. B., NAYAK, U. G., CHAKRAVARTI, K. K. and BHATTACHARYYA, S. C.

75. *Ind. Pat.* 59497
Production of porous polymer suitable for preparing cation-exchange resins. GOVINDAN, K. P., PANDYA, R. N. and KRISHNASWAMY, N.

76. *Ind. Pat.* 59606
Preparation of cation-exchange resin from porous cashew nut shell liquid polymer. KRISHNASWAMY, N., PANDYA, R. N. and GOVINDAN, K. P.

77. *Ind. Pat.* 59853
Improvements in or relating to the preparation of costus root oil and the isolation of lactonic constituents therefrom. KELKAR, G. R. and BHATTACHARYYA, S. C.

78. *Ind. Pat.* 59295
A process for the production of alkali salts like alkali-nimbidinates having medicinal properties from bitter constituents of nim. MITRA, C. R.

79. *Ind. Pat.* 60826
Improvements in or relating to the production of hydroxy, alkoxy or aryloxy substituted deoxybenzoins and particularly of deoxyanisoin. BOSE, J. L. and SHAH, R. C.

80. *Ind. Pat.* 62302
A process for the preparation of 1-ω-ketodicarboxylic acid (or ester) suitable for the preparation of civetone and dihydrocivetone. CHAKRAVARTI, K. K. and BHATTACHARYYA, S. C.

81. *Ind. Pat.* 61585
A process for the manufacture of an ammonium phosphate-sulphate fertilizer. GUPTA, J., SESHADRI, K., LOBO, J. and RAO, M. N.

82. *Ind. Pat.* 63083
A new method for the preparation of 4-hydroxycoumarins. SHAH, V. R., BOSE, J. L. and SHAH, R. C.

83. *Ind. Pat.* 62890
A new process for the production of 4-hydroxycoumarin and its derivatives. SHAH, V. R., BOSE, J. L. and SHAH, R. C.

84. *Ind. Pat.* 64366
Developments in or relating to the production of oils and fats having materials for detecting their adulteration and preservation against spoilage. Joshi, B. S.

85. *Ind. Pat.* 66194
Improvements in or relating to can–sealing composition. Raghunath, D. and Kapur, S. L.

86. *Ind. Pat.* 64959
A process for the preparation of dihydrojasmone. Amin, J. H. and Bhattacharyya, S. C.

87. *Ind. Pat.* 64958
Improvements in or relating to polishing compositions. Shah, S. M., Hinge, V. K., Mhaskar, V. V. and Shah, R. C.

88. *Ind. Pat.* 66096
A process for the production of bacterial diastase. Babbar, I. J., Bekhi, R. M. and Srinivasan, M. C.

89. *Ind. Pat.* 65777
A new process for the production of 4–hydroxycarbostyrils. Shah, V. R., Bose, J. L. and Shah, R. C.

90. *Ind. Pat.* 65543
A process for the preparation of α,ω–dicarboxylic acids and ω–hydroxy acids suitable for conversion to macrocyclic compounds. Mathur, H. H. and Bhattacharyya, S. C.

91. *Ind. Pat.* 65977
Rubber base adhesive. Uma Shankar.

92. *Ind. Pat.* 45583
Manufacture of high quality gelatin from indigenous sources of hides and skins. Joshi, H. K., Uppal, M. M. and Potnis, G. V.

93. *Ind. Pat.* 46945
Manufacture of saponins (commercial grade) from Indian soapnuts. Gedeon, J.

94. *Ind. Pat.* 46946
Improvements in or relating to the manufacture of saponins (commercial grade). Gedeon, J.

95. *Ind. Pat.* 49851
Recovery of hydrochloric acid gas from unreacted chlorosulphonic acid in the manufacture of aromatic sulphonyl chlorides. Joshi, C. G., Kulkarni, A. B. and Shah, R. C.

APPENDIX V

PROCESSES RELEASED TO INDUSTRY DURING 1957–60

1. *Liquid Rubber* (*Ind. Pat.* 60555).
   Swastik Rubber Products Ltd., Kirkee, Poona.
2. *Nicotine sulphate from tobacco or tobacco wastes* (*Ind. Pat.* 45666 and 54867).
   Tobacco Bye-products Pvt. Ltd., Guntur, Andhra.
3. *Pressure-sensitive transparent adhesive tape* (*Ind. Pat.* 66803 and 67490).
   Anandilal Hemraj and Co., Bombay.
4. *Can-sealing composition* (*Ind. Pat.* 66194).
   Arya Chemical Works, Calcutta.
5. *Mixed nitrogen phosphorus fertilizer* (*Ind. Pat.* 47439).
   Agri-Union Services Pvt. Ltd., Bombay.
6. *Cation–exchange resin from commercial cashew-nut shell liquid* (*Ind. Pat.* 59497 and 59606).
   Tulsi Industries, Poona.
7. *Ammophos II from indigenous rock phosphate* (*Ind. Pat.* 61585).
   Agri–Union Services Pvt. Ltd., Bombay.
8. *Bacterial diastase for desizing* (*Ind. Pat.* 66096).
   Chemaux Pvt. Ltd., Bombay.
9. *Foundry core oil* (*Ind. Pat.* 65542).
   Released free of charge to foundries and others.
10. *Vitamin C* (non-patented).
    Hindustan Antibiotics Ltd., Pimpri.
11. *Factice from tobacco and safflower seed oils* (*non–patented*).
    Released free of charge to industries.
12. *Thermistors* (*non-patented*).
    Adept Laboratories, Poona.

APPENDIX VI

MEMBERS OF THE STAFF AND STUDENTS WHO QUALIFIED FOR THE DEGREE OF M. Sc. OR Ph. D. DURING THE PERIOD JANUARY 3, 1950 TO MARCH 31, 1960

S. No.	Name	Degree	University	Title of thesis	Year
1.	Chanan Singh	M. Sc.	Punjab	Studies on Biochanin C	1950
2.	C. Sivaraman	Ph. D.	Madras	Studies on liver fat metabolism	1950
3.	P. N. Rangachari	Ph. D.	Madras	Carbohydrate metabolism in *Aspergillus niger* with reference to citric acid formation	1953
4.	J. L. Bose	D. Phil.	Calcutta	Studies in the constituents of *chana* (*Cicer arientinum* Linn.)	1955
5.	K. K. Chakravarti	D. Phil.	Calcutta	Studies in essential oils	1955
6.	N. L. Dutta	D. Phil.	Calcutta	Chemical investigation of some Indian poisonous plants	1956
7.	B. B. Ghatgey	Ph. D.	Poona	Studies in essential oils	1956
8.	C. G. Joshi	Ph. D.	Poona	Lithium aluminium hydride as a reducing agent for anthoxanthins	1956
9.	V. V. Kane	M. Sc.	Poona	Studies in hydroxy carbonyl compounds	1956
10.	N. C. Sogani	Ph. D.	Agra	Organometallic compounds of analytical importance	1956
11.	Kartar Singh	Ph. D.	Punjab	Glycolytic enzymes in *Aspergillus niger*.	1956
12.	M. S. N. Rao	Ph. D.	Poona	A study of the interaction of metal ions with bovine serum albumin	1956

S. No.	Name	Degree	University	Title of thesis	Year
13.	K. P. Sinha	Ph. D.	Poona	On the theory and mechanism of solid-solid reactions	1956
14.	R. M. Joshi	M. Sc.	Poona	Studies in co-polymerisation	1956
15.	H. L. Bhatnagar	Ph. D.	Poona	Physico-chemical studies on polymer solutions	1957
16.	S. D. Ambegaokar	Ph. D.	Bombay	Physiological effects of low protein diet	1957
17.	P. R. Subbaraman	Ph. D.	Madras	Studies in the electrodeposition and estimation of germanium	1957
18.	D. D. Nanavati	M. Sc.	Bombay	Modification of nickel for the hydrogenation of oils and application of *N*-bromosuccinimide in the production of conjugation in fats and oils	1957
19.	P. S. Borkar	Ph. D.	Poona	Studies on α-amino acid acylases	1957
20.	Gurdas Singh	Ph. D.	Punjab	Modified gelatin as a plasma substitute (plasma volume restorer or expander)	1957
21.	B. D. Kulkarni	M. Sc.	Poona	A simple method for the synthesis of ^{14}C labelled sugars	1957
22.	K. C. Menon	Ph. D.	Madras	Physiological effects of dietary protein	1957
23.	B. V. Ramachandran	Ph. D.	Madras	Studies on casein phosphopeptone	1957
24.	P. S. Aggarwal	Ph. D.	Poona	A study on the structures of vapour phase deposits	1958
25.	A. K. Das Gupta	Ph. D.	Poona	Escape of impurities from solids	1958

S. No.	Name	Degree	University	Title of thesis	Year
26.	G. D. Palkar	M. Sc.	Poona	Diffusion in solid state	1958
27.	Y. N. Trehan	Ph. D.	Punjab	Crystal growth in solid-gas reactions	1958
28.	B. N. Joshi	M. Sc.	Bombay	Synthesis of vitamin A acetate	1958
29.	C. R. Mitra	Ph. D.	Poona	Investigation of certain indigenous medicinal oils	1958
30.	V. S. Pansare	M. Sc.	Bombay	Azo dyes from cashew-nut shell liquid derivatives	1958
31.	J. P. Varma	Ph. D.	Poona	Studies in the fatty oils of *Sterculia foetida* Linn. and *Momordica charantia* Linn.	1958
32.	N. B. Desai	Ph. D.	Bombay	Solubilized vat dyes and Raney nickel reduction	1958
33.	S. R. Modak	M. Sc.	Poona	Studies on the proteolytic system of *Aspergillus flavus-oryzae*	1958
34.	D. N. Seetharamarao	Ph. D.	Poona	Self-diffusion of ions in solutions	1959
35.	M. K. Unni	Ph. D.	Bombay	Application of chromatography to the purification of direct cotton dyes and vat dyes and the constitution of some vat dyes	1959
36.	Mansa Ram	Ph. D.	Punjab	Studies in tryptophan and its peptides	1959
37.	J. R. Vakil	Ph. D.	Bombay	Studies on the metabolism of *Aspergillus niger*	1959

APPENDIX VII

DEPUTATIONS ABROAD FOR THE PERIOD 3-1-50 to 31-3-60

S. No.	Name	Purpose	Place of deputation	Period
1.	J. Gupta	Visits to inorganic chemical laboratories (CSIR deputation)	U. K. laboratories	28-6-49 to 30-6-50
2.	M. U. Pai J. V. S. Ramanjaneyulu K. K. Sarin J. P. Varma	To attend the 75th Anniversary of the American Chemical Society and the International Chemical Conclave	New York	27-8-51 to 11-10-51
3.	M. Damodaran	Study of developments in biochemical techniques and processes (CSIR deputation)	Laboratories in U. K. and Sweden	15-4-51 to 3-11-51
4.	P. N. Rangachari	Research on fluorescent components in casein hydrolysates (Indo - German Co-operation Scheme)	University of Hamburg	8-10-52 to 16-11-54
5.	A. K. Das Gupta	Research in radiochemistry and use of isotopes in the determination of geological age of minerals (UNESCO Technical Assistance Programme)	McMaster University, Hamilton, Ontario	16-12-52 to 4-1-54

S. No.	Name	Purpose	Place of deputation	Period
6.	Lakhbir Singh	Training in instrumentation (Colombo Plan)	National Bureau of Standards, Washington, D. C.	8-9-53 to 21-3-54
7.	R. C. Shah	Study of developments in pure and applied organic chemistry (CSIR deputation)	U. K. laboratories	7-4-54 to 17-9-54
8.	S. L. Kapur	Study of developments in polymer chemistry and technology (CSIR deputation)	Laboratories in U. K., France, Holland and Italy	28-9-54 to 21-12-54
9.	A. Krishnan	Training in librarianship (U. S. Educational Foundation Fellowship)	Columbia University	17-6-55 to 16-7-56
10.	J. C. Sadana	Research on intermediary metabolism (Fellowship)	Johns Hopkins University, Baltimore, Maryland	3-8-55 to 20-9-56
11.	S. L. Bafna	Research on mechanism of heterogeneous catalysis by ion–exchange resins (Boesse Postdoctoral Fellowship)	Columbia University	5-9-55 to 4-6-56
12.	V. S. Govindarajan	Training in fermentation technology (Colombo Plan)	Laboratories in Canada	15-4-55 to 25-1-56
13.	N. Krishnaswamy	(a) Foreign students' summer project	M. I. T.	June to Sept. 1951
		(b) Research on ion–exchange membranes (Fellowship)	T. N. O. Central Laboratory and Plastics Research Institute, Delft, Netherlands	16-11-55 to 12-6-56

S. No.	Name	Purpose	Place of deputation	Period
14.	J. S. Aggarwal	Research on paints and allied subjects (Colombo Plan)	Paint Research Station, Teddington	22–2–57 to 6–6–57
15.	K. P. Sinha	Research on quantum mechanical aspects of solids (Colombo Plan)	University of Bristol	27–9–57 to 26–10–59
16.	C. Sivaraman	Research on physico–chemical processes in the purification of proteins (Colombo Plan)	University of Leeds	18–9–58 to 31–10–60
17.	J. L. Bose	Research in carbohydrate chemistry (Colombo Plan)	University of Birmingham	28–9–58 to 19–10–59
18.	N. L. Dutta	Research in organic chemistry	Duke University, Durham, N. C.	3–12–57 to 12–12–59
19.	A. V. Subbaratnam	Research on alkaloids	Montana State College	25–4–58 to 24–4–61
20.	K. Venkataraman	To give lectures and visit laboratories on an invitation from the Moscow Mendeleev Institute of Technology, the Leningrad Institute of Technology, and the Ministry of Higher Education of the USSR	USSR	17–9–58 to 19–10–58
21.	K. K. Chakravarti	Research on natural products	University of Wisconsin	26–4–59 to 17–5–60

S. No.	Name	Purpose	Place of deputation	Period
22.	L. K. Doraiswamy	Study of process kinetics and reactor design (Indo-French Technical Co-operation Scheme)	France	18–10–59 to 31–5–60
23.	G. Narsimhan	Study of applied kinetics and transport process (Indo-French Technical Co-operation Scheme)	France	18–10–59 to 31–5–60
24.	M. K. Gharpurey	Research in physical chemistry (Visiting Research Scientists Programme administered by the National Academy of Sciences, USA)	Johns Hopkins University, Baltimore, and Brown University, Providence	21–10–59 to 13–10–61
25.	A. Goswami	Research on crystal and surface structural studies by electron diffraction (Colombo Plan)	CSIRO Laboratories, Australia	22–9–59 to 3–9–60
26.	K. Venkataraman	(a) To lead a cultural delegation on an invitation from the German Academic Exchange Service (b) To give lectures at University College, Dublin (c) To visit the National Chemical Laboratory, Teddington	(a) Federal Republic of Germany and West Berlin (b) University College, Dublin (c) National Chemical Laboratory, Teddington	8–11–59 to 13–12–59

S. No.	Name	Purpose	Place of deputation	Period
27.	M. V. Kunte	Studies in chemical engineering with particular reference to applied kinetics, catalysis and fluidization (Colombo Plan)	University of Birmingham	1–10–59 to 1–10–61
28.	K. S. Rajan	Research in the chemistry of rare earths	Clark University, Massachusetts	20–8–59 to 19–8–61