NATIONAL CHEMICAL LABORATORY POONA

Annual Report

1971-72

NATIONAL CHEMICAL LABORATORY
POONA

1971-1972

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH

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CONTENTS

INTRODUCTION

The National Committee on Science and Technology (NCST) is currently engaged in formulating a National Plan for Science and Technology. The NCL is taking a very active part in this effort especially in the field of R & D for chemical industries. The main thrust of these plans is to promote self reliance in Science and Technology so that the country can realise its planned goals of socio-economic progress mainly as a result of its own efforts. Fortunately a good science and technology base and a reasonably good industrial capability exist in the country. R & D institutions such as the NCL have a key role to play in these national efforts.

The R & D programme of NCL is presently under review by our research analysis group working in concert with our research staff and our Advisory Panels and the Executive Council (E. C.). A sub-committee of the E. C. headed by Shri L. Kumar is currently examining a perspective plan of NCL from the point of view of its relevance to country's socio-economic needs and goals as outlined in Five Year Plans and the national tasks identified by the NCST.

In keeping with the above internal appraisal and introspection, the NCL's objectives have been more clearly defined and its activities planned accordingly. This Annual Report thus begins with a statement on the objectives of the NCL and the areas of its R & D activities. The work of the NCL is then presented in chapter I, under 14 major scientific, technological and management areas. In each area of work the R & D projects under investigation are presented in the following order: sponsored, pilot plant, product / process oriented research and basic research projects. Apart from discussing the work done in these areas an attempt has been made also to draw pointed attention to the relevance of the work in progress with problems of industrial development and the country's socio-economic needs. The present annual report therefore is significantly different from the previous ones.

RESEARCH UTILIZATION

Chapter II deals with data regarding research utilizaton. The achievements of the NCL as regards establishment of commercial productions based on processes developed in the laboratory are described in table I (page 82).

The total social benefits accruing from the activities of the discipline-oriented laboratory, such as the NCL working outside the scope of an industrial enterprise are not easily quantifiable. However, one possible quantifiable yardstick for judging the performance of such laboratories may be their countribution to the Gross National Product through research utilization. The status report on research utilization (1971-72) presented in Table I (page 82) indicates that the near exponential growth of industrial productions based on NCL know-how has been maintained. This is evident from the following figures :—

Year	1968-69	1969-70	1970-71	1971-72
No. of processes in production/ value of total production, Rs. in lakhs	47/70	53/130	55/284	51/471

Table II represents 48 additional processes which are awaiting production. Good progress has been reported by the user industries on the commercialization of ten processes which are likely to be implemented in the next 1-2 years. With the establishment of processes having larger value impact such as, production of opium alkaloids, Vitamin C, chloromethanes, chlorobenzenes, *p*-nitrophenol, dimethylaniline, hexachloroethane, etc., it is estimated that the total value of annual production based on NCL know-how will soon be over Rs. 10 crores.

A few outstanding achievements by way of new productions established during the year are cited below.

During the year under review production on Catechol, tert-Butylcatechol, Ferrites (Hard) and Hot and Cold sachets has been newly established.

(1) M/s. Percynic Chemicals, Bombay, established commercial production of catechol and tert-butylcatechol, since January 1972. During the three months under report the firm produced nearly 2 Tonnes of catechol (Rs. 33.6 thousand) and 1.6 Tonnes of tert-butylcatechol (Rs. 77 thousand). NCL process for catechol consists of extraction of catechol rich cut of polyvalent phenol available with Neyveli Lignite Corporation. Catechol is an intermediate for the manufacture of tert-butylcatechol and also used in drugs. Tert-butylcatechol is used as a stabilizer in the synthetic rubber industry. The estimated requirement of tert-butylcatechol in the country is 20 TPA valued Rs. 6 lakhs.

(2) Production of hard ferrites has been established by M/s. Semiconductors Ltd., Poona, and during the first year, 2.6 lakhs numbers of ferrites valued at Rs. 2.8 lakhs have been produced. The primary applications of hard ferrites are in cycle dynamos, loudspeaker magnets, latching magnets, magnetic separators and in automobile magnets, wiper motors, alternators, tape recorder motors, etc. The estimated requirement of hard ferrites is of the order of 100 TPA by 1974.

(3) Hot and cold sachets containing a polysaccharide gel can serve as a thermal reservoir for heat and cold because of its large water content. This can be conveniently used as a substitute for hot water bag or ice bag. M/s. Thermo Chem Laboratories, Poona, have established production of this novel product with a capacity of 2 lakh sachets per year valued at Rs. 24 lakhs. The use and applications of these sachets were successfully demonstrated by the party in many exhibitions, trade meetings and Govt. hospitals.

(4) The semi commercial unit (300 TPA) for producing carbon tetrachloride and chloroform from methane installed at the site of M/s. Standard Alkali Ltd., Thana, was successfully operated this year with active assistance from NCL scientists. The firm is now planning for a commercial plant of 3000 TPA for which design and engineering assistance will be rendered by NCL. The fluidized bed technology developed for this project is sophisticated and would bear comparison with the most modern plants elsewhere in the world. This successful joint collaboration of industry and NCL will not only bring confidence in Indian technologists and industry, but may open new vistas for export of technologies.

In all 9 NCL processes have been released this year to 11 parties through NRDC. They are Benzoic acid from methyl benzoate, Substitute for Bostik sealant, Dimethylaniline, Hard ferrites, Monochloroacetic acid, DOP/DBP, *p*-Nitrophenol, Hot and Cold sachets and Terpineol.

Know-how available from NCL is presented in Table III (page 102) which gives information on the field of utilization, capacity envisaged, imported component, fixed and working capital, scale of development and provisional terms of release for 77 processes. Out of these, 16 are already implemented on commercial scale, whereas licences have been given for other 10 products/processes.

Information given in Tables I-III will be useful in motivating prospective entrepreneurs to start new enterprises based on NCL technology.

Information on special facilities available in the NCL, services rendered to industries and research institutes, industrial liaison activities, seminars, lecturers, etc., is included in Appendices.

SPONSORED PROJECTS

A review of sponsored projects so far undertaken in the NCL has been presented in tabular form (page 123). During 1971-72 work on twelve sponsored schemes was completed and 10 more new schemes were undertaken for investigation. The total number of sponsored schemes completed so far (upto March 1972) is now 76 and total receipts on account of these projects amounted to Rs. 58 lakhs. The total value of commercial productions based on sponsored R & D work amounted to Rs. 369.65 lakhs.

PRODUCT ORIENTED RESEARCH

Of the 118 product oriented projects, 10 relate to solid state materials, 15 relate to inorganic chemicals and products, 24 relate to naturally occouring organic compounds/products, 31 relate to industrial organic chemicals, 16 relate to polymers, resins and elastomers, 17 relate to chemical engineering and process development work and 5 relate to instrument development.

BASIC RESEARCH

Basic research in progress is also described under the relevant areas of R & D activity. Results of this work have been published in 112 research papers. In all 20 scientists (including 3 research fellows) received post graduate degrees.

COST / BENEFIT ANALYSIS

The cost / benefit analysis of the work of the laboratory is presented on page 112, whereas figures for inputs represent actual expenditure, the benefits (output) accruing from NCL's work include statements of receipts on account of premia, royalties, sponsored projects, analytical / testing charges, FCP sales, consultancy, etc., (direct benefits) alongwith indirect benefits such as generation of R & D capability (research degrees awarded), inquiries answered, research publications, advice to industry and Government departments, etc.

As against total inputs (for 1971-72) of Rs. 94.35 lakhs (Rs. 70.41 lakhs recurring; Rs. 12.42 lakhs capital; Rs. 2.85 lakhs pilot plant; Rs. 8.67 lakhs contributions by sponsors); Rs. 1.42 lakhs were received by way of royalties and premia, Rs. 8.67 lakhs as sponsorship fees, Rs. 2.67 lakhs as sale of FCP chemicals and other products, Rs. 2.69 lakhs as analytical fees, miscellaneous receipts, etc. The total value of industrial products for the year under review was Rs. 471 lakhs of which 40% may be claimed as contribution to Foreign Exchange savings.

OBJECTIVES AND ACTIVITIES

The National Chemical Laboratory was established in 1950 with the motto ' To advance knowledge and to apply chemical science for the good of the people.' The objective of the laboratory is as follows :

' The pursuit of scientific and industrial research in the fields of chemistry and chemical technology relevant to the present and future industrial, technological and socio-economic needs of the country.'

In accordance with the general objectives of the CSIR and the above specific objectives of NCL, the Laboratory shall be engaged in the following Research & Development (R & D) activity :-

i) Projects of national priority as evolved in consultation with The National Committee on Science & Technology (NCST);

ii) Projects sponsored by industries in the public/private sector in consonance with national priorities;

iii) Projects directed towards improvement, adaptation and development of existing and imported technology;

iv) New technology relevant to the country's economic and industrial needs in keeping with the national objectives of self-reliance;

v) Basic scientific research that is necessary for industrial research in progress and from the view point of future advances in technology in consonance with national priorities;

and vi) Technical advisory services like information, extension and consultancy and analysis and testing of special nature.

Currently the above activities are conducted in the following area :

1. Natural and synthetic organic chemicals of importance to industry and agriculture; such as dyestuffs, pigments, detergents, intermediates, petrochemical down-stream products, drugs, pharmaceuticals, hormones and pheromones,agrochemicals and plant nutrients, etc.

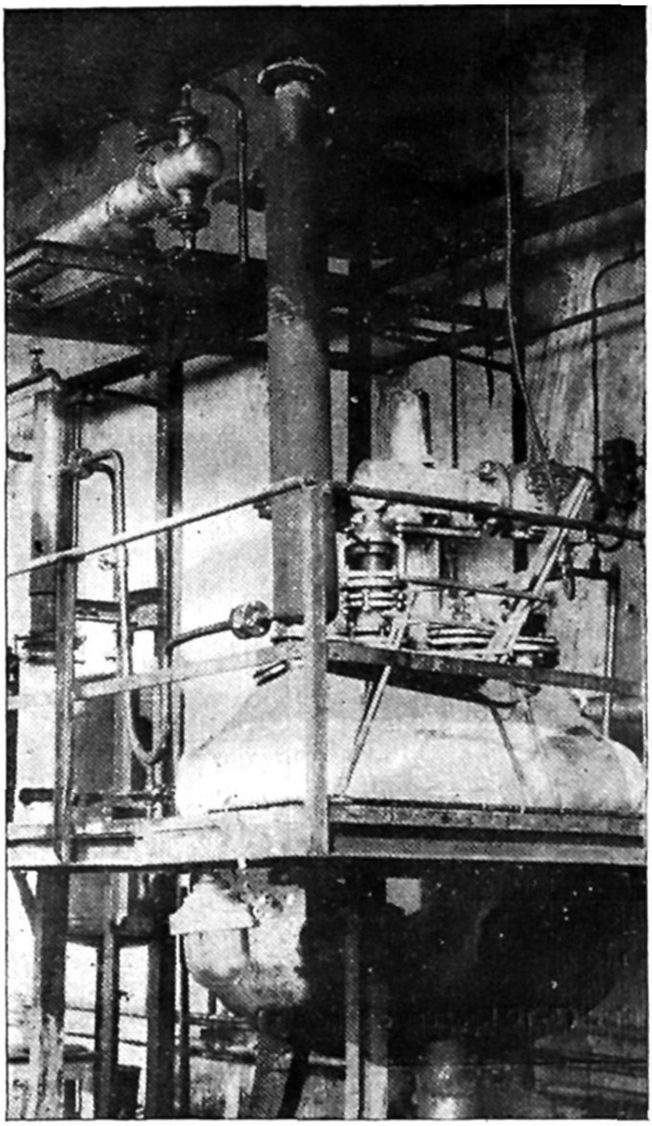
2. Industrial inorganic chemicals, industrial catalysis, solid state materials, utilization of natural inorganic resources, organometallic and co-ordination compounds, physico-chemical studies and analysis and instrumentation.

3. Fermentation processes, industrial microorganisms (NCIM) and enzymes, plant and animal tissue culture.

4. Industrial polymers and elastomers, adhesives and coatings, man made fibres and auxiliaries.

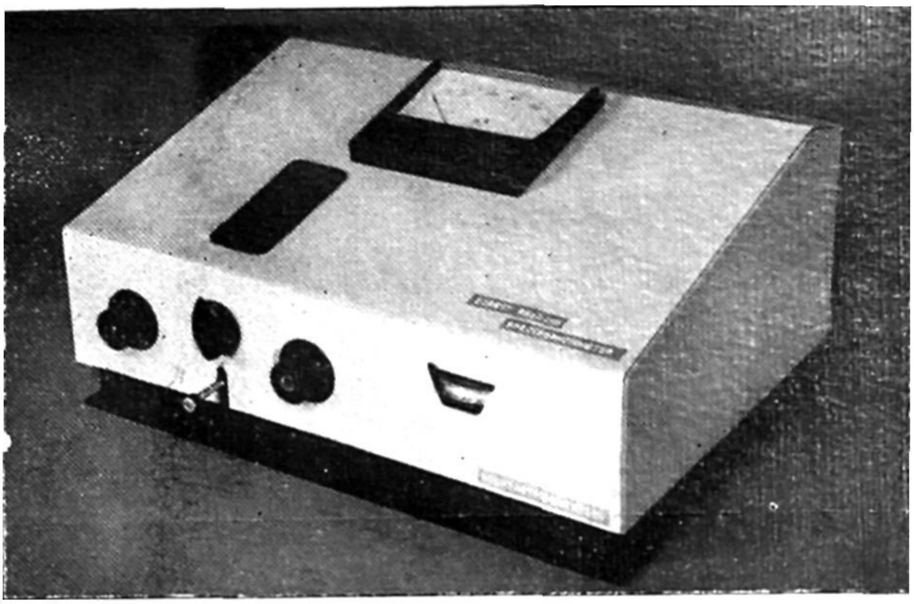
5. Process development related to petrochemicals and bulk organic chemicals, scale up of processes and studies in chemical engineering, Design of industrial plants based on process know-how developed in the NCL

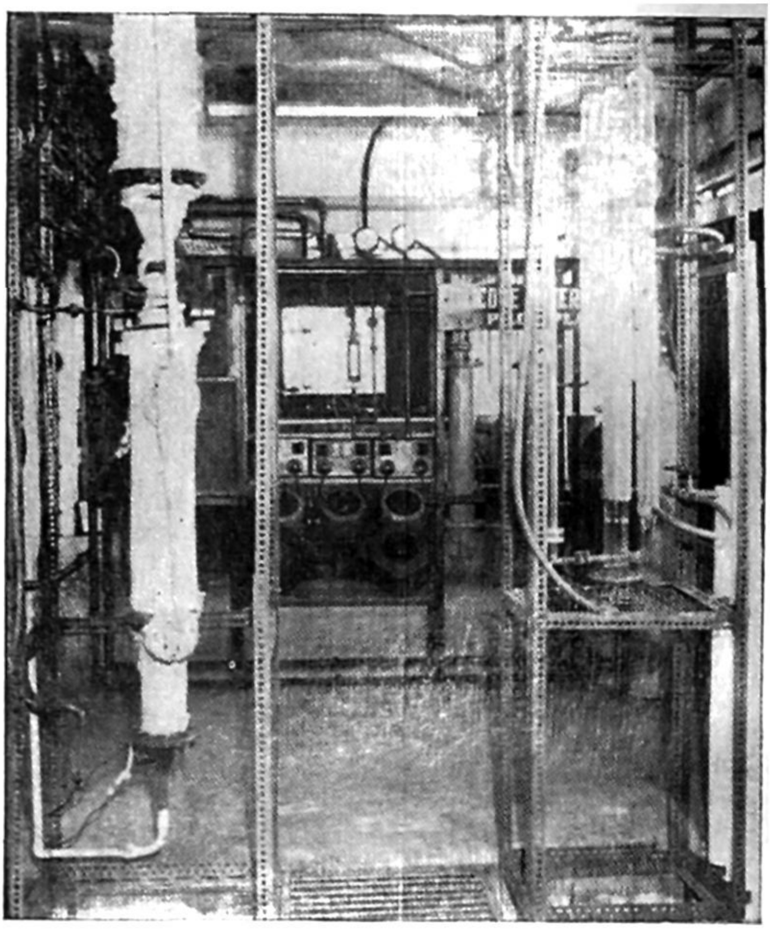
6 Studies in research analysis and management.



tert-Butylcatechol plant–
M/s Percynic Chemicals,
Bombay.

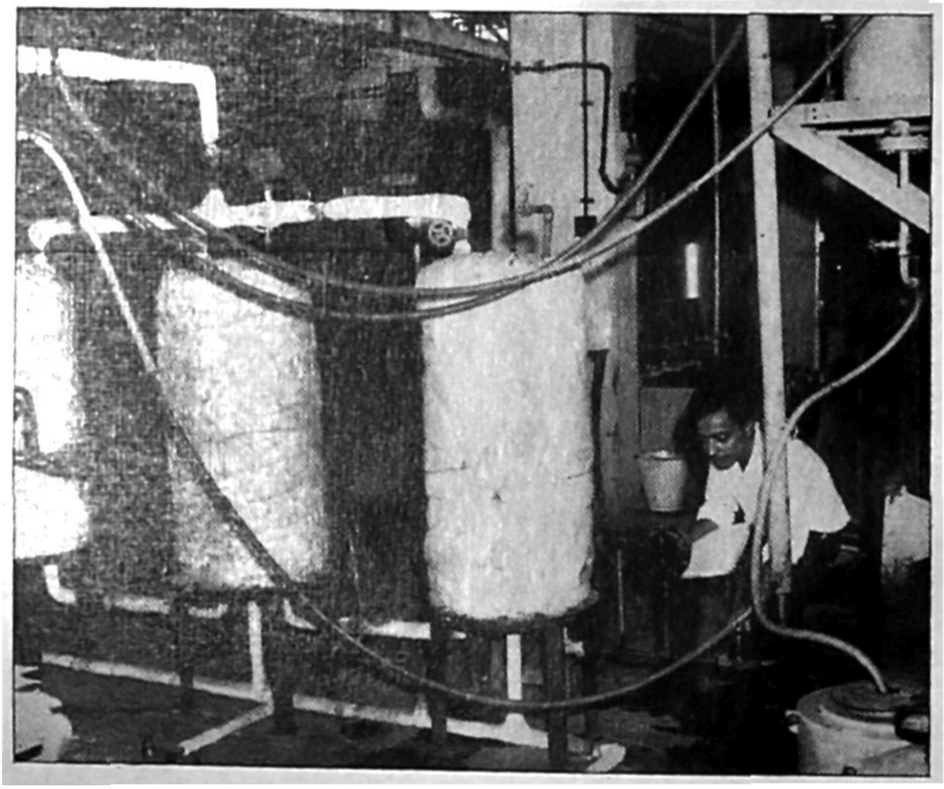
Prototype Direct reading spectrophotometer / colorimeter





Silicone intermediate pilot plant (2kg /hr.) at NCL

Ethylenediamine pilot plant (5-10 Kg. / day continuous) at NCL



RESEARCH AND DEVELOPMENT PROJECTS

1. SOLID STATE MATERIALS

1.1 *Polycrystalline silicon :* (ATT-9/69)

High purity silicon used in several electronic devices is normally prepared by (i) thermal decomposition of SiH_4 (silane) and (ii) hydrogen reduction of trichloro or triiodosilane. Both the methods have some advantages and disadvantages.

In the bench scale unit, installed last year, several runs were taken and process conditions optimized for the preparation of epitaxy grade silicon tetrachloride and silane.

While considering the economic feasibility of the silane route, the question of availability of lithium aluminium hydride required for hydrogenation was studied. It was finally concluded that preparation of silicon by silane route solely depends on availability and cost of $LiAlH_4$. Since imported cost of $LiAlH_4$ is high and prices of imported silicon are comparatively low, further work on the project has been suspended. However in case $LiAlH_4$ is made indigenously and is available at the level of c. i. f. price silane route for the preparation of silicon will still be economically feasible.

1.2 *Tin oxide potentiometer :* (ATT-61/71)

The tin oxide potentiometer is a new concept leading to the development of a product having extraordinary technical qualities and large industrial potential. The demand for the tin oxide potentiometer will largely depend on how far it replaces carbon potentiometers and wire wound potentiometers in a vast number of applications.

A detailed study was undertaken of electrical, optical and film thickness properties of the electrically conducting tin oxide films which were deposited on glass substrates. During the study of electrical properties of the tin oxide layers deposited by spraying techniques it was found that, the relationship between resistance and the length of the deposited film, within a prescribed limit is linear. This aspect of study is being extended to longer lengths of deposited films.

A significant breakthrough was achieved in the hitherto difficult problem of maintaining a smooth transition from a metallic end contact to a semiconducting tin oxide film. The technique involved the superdeposition of a small area of highly degenerate tin oxide in between the metallic contact and the track. This resulted in a smooth contact region which gave improved resistance/length characteristics.

Current voltage characteristics for a large number of specimens have been plotted and the results and the plots are being evaluated for suitability as resistive tracks.

1.3 *Flexible magnets* : (ATT-170/71)

The project was undertaken as an extension of the hard ferrite work now being implemented by a few commercial firms. These parties showed interest in the development of these special type of magnets mainly used in refrigerator-door gaskets, toys and educational kits.

Based on chlorinated polythene, a process for the preparation of flexible magnets of the following specifications was standardized :

Ferrites : binder(polymer)ratio	87:13 by weight
Flux density (Br)	1600 ± 50 gauss
Coercivity (He)	1400 ± 50 Oersted
BH (max)	0.45 Mega G. O.
Density	3.4 g. per c. c.

Physical properties such as flexibility and hardness, etc. of the product compared well with the imported samples. The process is now being offered to industry.

1.4 *Gamma ferric oxide* : (ATT-171/71)

The manufacture of recording tapes has started recently in the country based on imported magnetic iron oxide compositions. It is gathered that the the present estimated requirement is about 6.5 TPA valued at about Rs. 52,000 and expected requirement in 1975-76 is about 30 TPA valued at about Rs. 2.5 lakhs.

A process has been developed to prepare the required grade of gamma ferric oxide by precipitating at first alpha hydrated ferric oxide from ferrous sulphate solution by bubbling air in the presence of scrap iron. This is then reduced to ferrosoferric oxide in a reducing atmosphere. This ferrosoferric oxide by is further oxidized to gamma ferric oxide by heating in air. The needle shaped crystals so produced in the first step retain their shape practically unaltered through all these stages.

The preparation of yellow hydrated ferric oxide has now been scaled up from the laboratory stage (50 g./day) to a bench scale producing 1 kg./day. The second stage has also been scaled up from 10 g./batch to 300 g./batch in a locally fabricated rotary furnace. The last stage can be easily completed on the same scale in the rotary furnace itself or in open shallow pans kept in an electric oven.

The magnetic properties of the gamma ferric oxide produced on the bench scale are found to compare favourably with those of an imported sample.

1.5 *Synthetic gem stones* : (ATT-172/71)

Trial runs for the growth of sapphire crystals in a modified Verneuil furnace was reported earlier. Further modifications have been introduced, in this unit to improve flame conditions, heat rate and automatic lowering. On the basis of the data collected in the above experiment, a new equipment suitable for commercial production of synthetic gem boules has been designed and is under fabrication.

1.6 *Radiosonde thermistors* : (ATT-175/71)

Radiosonde thermistors are used for measuring the temperatures of upper atmosphere with the help of baloons by using radiosonde techniques. These devices thus are extremely useful in both civil and defence aviation and in meteorological forecasting. The project was undertaken at the request of Deputy Director General of Observatories (Instruments), Indian Meteorological Department, New Delhi. The demand is estimated to about 30,000 pieces/year valued at Rs. 4.5 lakhs. At present these are totally imported.

Different metallic oxides in definite proportions were mixed with a binder and extruded into rods followed by sintering. Thermistors so produced were evaluated by the Meteorological Dept. and found to be satisfactory. The process is being offered to industry.

1.7 *Photoconducting materisls* : (AB-5/62)

The project aims at investigation of new processes and techniques for production of photoconducting materials such as layer, thin films and crystals for use in devices.

A method to obtain thin layers of photoconducting CdSe has been successfully worked out wherein chemical deposition techniques have been adopted for the first time.

These thin layers which show great promise as photodetectors, photoconductive cells, hetrojunction devices, solar energy converters and image conversion devices, can be prepared by a simple process based on complex formation between a cadmium salt and ammonia, and the decomposition of the complex in the presence of selenourea giving rise to the thin layer of CdSe.

Work is in progress on various properties of these layers including current-voltage characteristic, space charge limited current behaviour, pulsed field conductivity, thermally stimulated currents, electrical conductivity at low temperatures, sandwitch layers (electrical properties of), photothermoelectric effect and spectral response of photoconductivity.

1.8 *Ferroelectric materials* : (AB-2/65)

Ferroelectric ceramics are commercially used in phonograph pickups, transducers for high power generation, wave filters and memory and display devices. The present investigation is primarily aimed at developing suitable ferroelectric ceramics for use as phonograph pickups.

A few compositions of modified PZT (lead zirconate titanite) have been developed which has high dielectric constant ($\sim$ 800), spontaneous polarization ($>20\mu$ coulomb/cm^2) and radial coupling coefficient. A method has been developed to cut the discs into small strips and to polarize the strips in a suitable manner for fabrication of phonograph pickups. Efforts are being made to standardize the sintering process so that product of reproducible properties may be obtained.

1.9 *Thick film materials* : (AB-75/69)

Thick films are widely used for integrated hybrid circuits in the form of conductors, resistors, capacitors, etc. In view of their increasing applications for integrated circuits in modern microelectronics, there is likely to be a good demand for these devices in the country.

On enquiry from a private firm work on the development of silver conductive paste formulations was undertaken and samples prepared. According to the evaluation reports from M/s. Bharat Electronics, Bangalore, further work to modify the compositions has been undertaken.

A special silver conductive paste was formulated for M/s. Tempo Semiconductors, Bombay, for various types of thermistors marketed by them. Their thermistors were coated at NCL and soldering was done after the necessary firing schedule. The firm expressed satisfaction about the samples. Some more quantity of the paste and solder composition have been sent to the firm for large scale testing at their end.

1.10 *High permeability ferrites* : (AB-90/71)

Pot core ferrites which are also known as high permeability ferrites are believed to be imported. Estimated present requirements are of the order of 30 TPA valued at Rs. 30 lakhs. These are used in radio and television sets, telecommunication instruments, extra high tension transformers, low loss filters etc.

These are essentially Zn-Mn (ous) soft ferrites with high purity of the order of 5000, low loss, and low disaccommodation.

Very pure and active oxides prepared specially for the purpose were made to react with one another in an inert atmosphere to give the manganous-zinc ferrite. The powder was pressed into toroids employing a special binder. The sintered toroids showed a permeability approaching 3000.

1.11 *Studies on non-crystalline solids :* (B-2.7/70)

Vanadium oxide glasses containing thallium were prepared and their electrical and optical properties were reported earlier. Some compositions without thallium oxide and having V_2O_5 — P_2O_5 systems were found to show ferroelectric properties.

Semiconducting glasses based on P_2O_5 — V_2O_5 with dopents including Tl have properties of negative temperature coefficient (NTC) and high electrical resistivity.

Recently it has been established that these glasses have a pronounced piezo-electric behaviour. The elucidation of the mechanism of this behaviour is also of considerable theoretical interest.

The temperature dependence of the d. c. conductivity was studied from 200° to 450° K. Comparing the conductivities of various samples, the effect of the introduction of thallium in the binary system is being studied.

A bridge was fabricated to study the a. c. conductivity of these glasses in the frequency range 50 to 15,000 cps.

Apparatus has been set up for the measurement of the piezo-electric properties.

2. PHYSICO-CHEMICAL STUDIES

2.1 *Thin films*

2.1.1 *Structure of thin films :* (B-4.3/58)

A detailed study on structure, crystal growth process, phase transition, etc. of the vacuum deposited thin films formed on various substrates at different temperatures has been made by electron diffraction technique. Systems studied were mainly Ga-Se, Ga-Te, Ga-Sb, Al-Sb, Co-Sb, Mn-Sb, Ga-P, Sb-P and also a few alloys of silver with cadmium, indium and tin. It was observed that the substrate temperature exercised a considerable influence not only on epitaxy but also on the crystallinity and phase composition. Once an epitaxial layer was formed further growth was conditioned not only by the temperature of the substrate but also by the substrate deposit pair, lattice fitting, rate of deposition, etc.

Deposits of GaSb and GaP formed on different faces of rock salt were polycrystalline in nature upto about 3000°. At about 400° epitaxial growth was observed and the deposits developed 2-d $\{100\}$ and 2-d $\{211\}$ orientations the latter being rotated by 30°, 60° and 90°. At higher temperatures 2-d $\{100\}$ orientation was predominant. On the cleavage face of mica the deposits developed 2-d $\{111\}$ orientation at a temperature more than 400°.

Deposits of Ga_2Te_3 were polycrystalline up to 300° even on single crystal substrates. Deposits formed on $\{100\}$ face of NaCl at 350° developed 2-d $\{211\}$ orientation orientated by 30° and 60° etc. On (110) face these showed 2-d $\{110\}$ orientation. At 450° on (111) face of NaCl deposits grew epitaxially but instead of having normal ZnS type cubic structure these developed a new phase, tetragonal with $a_0 = 5.58$ Å, $c_0 = 4.52$ Å and with 2-d $\{110\}$ orientation rotated by 90°. Ga_2Se_3 did not show epitaxial growth up to 400° even on single crystal substrate. On the other hand, on mica the deposits showed 2-d $\{111\}$ orientation with twinning on $\{111\}$ plane. Deposits of NiAl (Raney alloy) grew epitaxially on rock salt single crystals developing 2-d $\{100\}$ orientation in a (100) face. These deposits had CsCl type of structure with $a_0 = 2.88$ Å. On (110) NaCl there developed 2-d $\{111\}$ orientation. Another new phase (cubic) was noticed in case of the deposits formed at 450° with $a_0 = 4.82$ Å.

In case of CoSb having NiAs type structure showed a new phase (cubic) with $a_0 = 4.42$ Å when the deposits were formed at about 350°. At still higher temperature another cubic phase with $a_0 = 5.28$ Å was noticed. Similar studies were made on MnSb and various orientations or different temperatures were observed.

Silver alloys, with Sn, Cd and In grew epitaxially at appropriate substrate temperatures developing different 2-d orientations.

2.1.2 *Physics of thin films* : (B-4 4/58)

Samples of a thin film thermistor prepared last year had high resistance. Attempts were made to lower down the initial resistance and several samples with intial resistance in the range of kilo ohms instead of megohms were prepared and sent to Institute of Tropical Metereology, Poona and National Aeronautical Laboratory, Bangalore, for evaluation.

The electrical resistance of new specimens was measured in the temperature range of − 95° to + 120°. Most of the films were having TCR value between − 1.5 to 3.0%° per C° and the initial resistance in the order of kilo ohms. Necessary stabilizing treatments were given to these samples. Some of these were delivered to two concerned parties for their assessment regarding the suitability of these specimens for their use.

Electrical properties viz. Hall effect, electrical conductivity, magnetoresistance, thermoelectric effect, etc. were measured for Bi, Sb and Te films in the temperature range 78° to 500°K. From these values carrier concentration, mobility, energy band gap in the impurity as well as intrinsic conductivity range, TCR, thermoelectric power, mean free path, etc. were calculated. In case of Te films Hall coefficient R_H was found to increase with thickness as well as field strength. Most of the samples were ' p ' type. R_H was found to increase initially with temperature and after reaching maxima again decreased. Energy band gap was found to increase with decrease in film thickness. Electrical properties of several metals and their oxides are also being studied.

The dielectric and optical properties of evaporated films of niobium oxide were studied and it was found that the dielectric constant of the film was dependent on film thickness. The values obtained were from 12 to 130 for thickness range of 200Å to 3000Å . Temperature coefficient of capacitance (TCC) and effect of frequency (1-150 KHz) breakdown voltage, etc. were also measured. Pronounced ageing effect was noticed for these films.

Optical constants i. e. ' n ' (refractive index) and ' k ' (extinction coefficient) of these films were measured and studied by transmission method. ' n ' was found to vary from 1.6 to 2.09 and ' k ' from 0.22 to 0.12 in the wave length ranging between 7000 to 4000Å for a film (500Å). Optical properties of several semiconducting oxide and chalcognide films were also investigated. ' n ' and ' k ' values were evaluated. Variation of these constants with wave length and film thickness was noticed. In some cases values of ' n ' and ' k ' increased by two-folds with change of wave length as well as thickness.

2.2 *Radiation chemistry* : (B-3/67)

2.2.1 *Temperature effects in the radiation chemistry of water* : A study of radiolysis of aerated and deaerated ferrous sulphate systems showed that the small temperature coefficient of the aerated ferrous system was due to competition between the reactions

$$\overset{+}{H}_2 + O_2 \longrightarrow HO_2 + \overset{+}{H} \text{ and}$$

$$\overset{+}{H}_2 + \overset{2+}{Fe} \longrightarrow H_2 + \overset{3+}{Fe}$$

When the air is replaced with oxygen the second of these reactions is suppressed, the well known G of 15.6 rises to 16 and becomes temperature independent.

It follows that all the primary yields of the radiolysis of water are independent of temperature. A theoretical analysis of this work, particularly of the role of dielectric constant of water differentiating between the above reactions has been published.

2.2.2 *Photochemical hydroxylation of benzene :* Studies with specially prepared pure H_2O_2 showed that only three (not one as earilier suspected) out of ten H_2O_2 molecules decomposing photochemically give rise to hydroxyls (as measured by the phenol and hydroxymocondialdehyde). The remaining atoms decomposed perhaps by a bimolecular collision mechanism. The result is vital to all studies of photolysis of H_2O_2, where it has been hitherto assumed that all the H_2O_2 molecules decompose unimolecularly via a free radical mechanism. The quantum yield of decomposition of H_2O_2 is ~ 1 and that ol OH radicals is ~ 0.3.

2.2.3 *Thermal hydroxylation of aromatic hydrocarbons :* The hydroxyal radical reacts with diphenyl five times with benzene. The product is a mixture of hydroxy diphenyl and phenol. The mole fraction of alcohol giving the maximum yield of phenol is proportional to the ratio of the rate constants for the reactions

$$\text{OH} + \text{Aromatic} \xrightarrow{+O_2} \text{Phenol and}$$

$$\text{OH} + \text{Alcohol} \xrightarrow{+O_2} H_2O + \text{acetaldehyde}$$

Based on these observations, a method for evaluating the rates of reaction of OH radicals with the organic compounds, can be developed.

2.2.4 *Radiation polymerization of trioxane :* A detailed study on the trioxane irradiation and subsequent thermal polymerization has been made. By a proper choice of parameters like irradiation dose, temperature and time of polymerization, it was possible to obtain polymers with a conversion of about 60% and molecular weight comparable to commercial polyoxymethylene (Du Point's Delrin). The thermal stability of our samples were however inferior and attempts are being made to modify conditions to obtain the desired sutability.

2.3 *Transport properties*

2.3.1 *Isotope effect for impurity of diffusion of cobalt in copper :* (B-2.1/67)

In the earlier report on the diffusion of ^{57}Co and ^{60}Co in copper, it was reported that the value of $\left[1-.\frac{D_{60}}{D_{57}}\right]$ where D_{57} and D_{60} are the diffusion coefficients of the two isotopes ^{57}Co and ^{60}Co respectively in copper experimentally determined equal to 0.01903 appeared to be consistent with diffusion by the vacancy mechanism.

The many body interactions involved in solid state diffusion make it necessary to relate the isotope effect E to the correlation factor for diffusion as follows :

$$D = f_i \triangle K = \left(1 - \frac{D_{60}}{D_{57}} \right) \Big/ 1 - \left(\frac{m_{57}}{m_{60}} \right)^{\frac{1}{2}}$$

where m_{57} and m_{60} are the masses of the two isotopes ^{57}Co and ^{60}Co. $\triangle K$ is the correction factor which accounts for the many body interactions and can be interpreted as the fraction of the translational kinetic energy associated with the motion in the jump direction that is possessed by the jumping atom at the saddle point. One way of determining the value of $\triangle K$ is by a comparison of measured isotope effect in self-diffusion and the correlation factor f_i calculated theoretically from a knowledge of the crystal structure of the host lattice and assuming the mechanism of diffusion. The value of $\triangle K$ for self-diffusion in copper by vacancy mechanism was found to be 0.87. Combining this value of $\triangle K = 0.87$ and the measured value of $E = 0.763$ for diffusion of ^{57}Co in copper, the value of correlation factor was $f_i = 0.87$. This is in very good agreement with the theoretically calculated value of $f_i = 0.88$ based on the electrostatic model confirming further that the diffusion of cobalt in copper takes place by the vacancy mechanism.

The correlation factor f_i for the impurity diffusion in f.c.c. lattice may be approximated by the expression :

$$f_i = \frac{w_1 + (7/2)\, w_3}{w_1 + w_i + (7/2)\, w_3}$$

where w_i is the rate of exchange of vacancy neighbouring a solute with any four of the solvent atoms that are also neighbours of the solute, w_i the rate of exchange of the vacancy with an impurity and w_3 is the rate of exchange of a vacancy neighbouring a solute atom with any one of the seven solvent atoms adjacent to the vacancy but bot neighbours of the solute. If it is assumed that the solvent jump rates w_1 and w_3 to the vacancy are nearly equal, which is a reasonable assumption, we obtain that $w_1 \simeq 0.6\, w_o$ where w_o is the rate of exchange of the vacancy with a solvent atom in self-diffusion. Hence we obtain $w_i > w_3 > w_o$ which signifies that the solvent jump rates are faster than the impurity jump rates. This conclusion is consistent with the observation that the diffusion coefficient of cobalt in copper is smaller than the self-diffusion coefficient of copper.

2.4 *Application of Mössbauer spectroscopy :* (B-3.1 & 3.3/67)

2.4.1 *Nature of precipitated tin oxide in internally oxidized tin-silver alloy :*

Mossbauer spectroscopic investigation of 1 atom percent tin-silver alloy

...2

partially oxidized at 800° showed that the internally precipitated oxide is a mixture of SnO and SnO_2. The presence of SnO at 800°, has been attributed to an oxygen pressure in the alloy matrix lower than the dissociation pressure of SnO_2 and 800°. The composition of the internally precipitated oxide has been found to change to SnO_2 on prolonged heating in oxygen.

2.5 *Thermodynamic studies :* (B-3.6/70)

2.5.1 *Thermodynamic excess properties of binary liquid mixtures :* The Gillespe still for measurment of vapour-liquid equilibria has been modified to make measurements both at isobaric and isothermal conditions and over a wider range of temperature and pressure.

Preliminary experiments have been conducted to explore the possibility of the measurements of second virial coefficients of gases from the measurements of absolute vapour densities by thermogravimetric spring balance, as a function of decreasing vapour pressure.

Isothermal vapour-liquid equalibrium data at 40° for the binary system : n-butylamine-n-butylalcohol over the entire composition of n-butylamine have been determined. The activity coefficient of the components are being computed for the eventual estimation of the excess free energies.

The excess volumes of mixing of the system, n-butylamine-n-butylalcohol, have been determined by the density method of 5° intervals in the range 25° to 45°. The volume changes of mixing have been found to be negative and the effect increases with the increase of temperature.

The heats of mixing for the system : $n\text{-}BuNH_2\text{-}n\text{-}BuOH$ have been determined at twenty different mole ratios evenly distributed throughout the entire concentration range. The experimental values of the heats of mixing H_m at 25° can be expressed by the expression :

$$\triangle H_m = X_1 X_2 [B+C (X_1-X_2) + D (X_1 - X_2)^2]$$

where x_1 and x_3 are the mole fractions of n-BuOH and $n\text{-}BuNH_2$ respectively. The values of the constants in cal/mole are $B=2428.0$, $C= -92.46$ and $D= +88.14$.

2.5.2 *Thermodynamic properties of complex ions of the transition metals :* The analysis of the changes in the thermodynamic functions $\triangle G$, $\triangle H$ and $\triangle S$ produced as a result of the interaction of Ni (II) ion with cysteine in aqueous solution showed that the enthalpy change H associated with the formation of Ni(II)-S bond is nearly half of that expected of the Ni(II)-N bond. The thermodynamic parameters and the visible ultraviolet absorption spectrum of the bis-cysteino-Ni(II) chelate indicated that it has a square planar symmetry.

2.6 *Crystallographic studies :* (B-2.5/59)

The preliminary structure of *m*-nitrobenzoic acid was determined by direct methods. The asymmetric unit in this structure consists of two crystallographically independent molecules which form dimers. The refinement of the structure is in progress.

Three dimensional X-ray data was collected for determining the structure of *o*-dimethyl-amino-benzoic acid. The structure analysis is in progress. The preliminary results are : Monoclinic crystals; space group $P\frac{21}{n}$ with $a = 7.70$, $b = 15.77$, $C = 7.57$ Å, $\beta = 100°$.

With a view to determine the electron distribution in the entire molecule in order to study the bonding effects and test the quantum chemical theories of bond formation, a low temperature apparatus was set up to enable the structure determination near the liquid nitrogen temperature (i. e. about $-180°$). Preliminary experiments have been performed and further work is in progress.

2.7 *Spectrochemical studies :* (B-5.1/63)

In continuation of the work on the infrared spectra, molecular structure correlation of amino benzoic acids, the spectra of *N*-methyl and *N*-dimethyl derivatives of *ortho, meta* and *para* amino benzoic acids have been investigated. Except the *N*-dimethyl anthranilic acid all the compounds were found to be neutral molecules in the solid states. The *N*-dimethyl anthranilic acid was found to be Zwitterionic in the solid state as has been reported recently. The neutral structure for the *N*-methyl and *N*-dimethyl *meta* amino benzoic acd is in sharp contrast to the Zwitterionic form of the *meta* amino benzoic acid.

Infrared spectra of *N*-methyl, *N*-dimethyl and *N*-acetyl derivatives of *o*-, *m*- and *p*-amino benzoic acids have been analyzed to obtain information on their structures with special referecne to their dipolar or neutral characters. These substituted acids were found to have essentially neutral structures in the solid state.

2.8 *Dielectric properties of organic compounds :* (B-2.6/65)

The dipole moments of n-long chain alcohols evaluated in pure liquid state showed a decrease with increase in the temperature. This effect has been attributed to molecular association and hindered molecular rotation in the liquid state. The association factor ' S ' and the Kirkwood correlation factor ' g ' for a series of n-long chain alcohols have been calculated and discussed in the light of molecular structure.

3. INORGANIC CHEMICALS AND PRODUCTS

3.1 *Utilization of minerals*

3.1.1 *Conversion of bauxite into anhydrous aluminium chloride :* (SP-70/71)

India has large resources of bauxite ore which has not been hitherto utilised for the manufacture of anhydrous aluminium chloride. Although domestic consumption of aluminium chloride is not very high (150 T/month) there is a sizable export potential for this commodity.

The project aims at studies on chlorination of bauxite ore normally containing not less than 55% Al_2O_3 to get maximum yields of aluminium chloride with maximum chlorine efficiency.

In case of an externally heated reactor, chlorine efficiency of the order of 90% and almost 100% chlorination efficiency with respect to total oxides content (Fe_2,O_3 Al_2O_3, TiO_2) has been obtained.

Series of chlorination experiments have been carried out using excess carbon in briquettes and mixtures of oxygen and chlorine, air and chlorine, enriched air and chlorine etc. Attempts are being made to maximise chlorination efficiency after which scale up experiments on 2 kg./hr. of $AlCl_3$ bench unit will be undertaken.

3.1.2 *Hydrated calcium silicate from Wollastonite :* (SP-71/71)

Wollastonite, a naturally occurring calcium metasillicate contains about 40-50% silica and about 48-49% lime with impurities such as oxides of iron, aluminium, titanium and manganese. The mineral as such is used in ceramics, rock wool manufacture, tile making and in the glass industries.

Work on the preparation of hydrated calcium silicate from Wollastonite was taken up on 400 g. and later on, 1 kg. scale. Various conditions such as acid concentration, particle size of the mineral, order of addition of the reactants, dilution etc., were studied.

The project is now concluded and final report sent to the sponsor.

3.1.3 *Studies on the nature of reduction of ilmenites :* (B-6.6/71)

The phases, ilmenite ($FeO. TiO_2$) and pseudobroxite ($Fe_2O_3 . TiO_2$) which are detected in the naturally occurring ilmenites, have been synthesized and studies on their carbothermic reduction and chlorination have been carried out.

Attempts to synthesize other phases such as $Fe_2O_3.3TiO_2$ and $2FeO.TiO_2$ reported in literature have not so far been successful.

3.2 *Utilization of industrial inorganic bye products*

3.2.1 *Removal of silica from black liquor :* (SP-60/70)

At the instance of a paper pulp unit, work on the removal of silca from ' black liquor ' was undertaken.

Procedures for removing about 97-98% of silica present in black and green alkaline liquors (dilute and concentrated black liquors containing 7-10 g. or upto 24 g. of SiO_2 per litre and green liquor sample containing 13-15 g. SiO_2 were developed. Factors, such as , solid contents, temperature, reaction time etc., for efficient silica removal from differnt black and green liquor samples were studied in detail. The alkali losses were found to be within 1% in all the experiments carried out with green liquor. Experiments with dilute black liquor samples on 5 and 10 litre scale were also carried out.

The calcined lime sludge sample obtained by causticization of the treated green liquor was found to be poor (54% active lime) and as such could not be used again. A simple method was developed to obtain good quality quick lime from the sludge. The sample of quicklime obtained by this method contained 71.3% active lime which was comparable with the active lime present in a freshly activited lime sample supplied by the firm (71-72%). The slakeability of the calcined lime samples obtained by causticization of the treated green liquor was examined with a lime sample (for comparison) obtained by burning lime stone directly. The value obtained was the same (95%) in both the lime samples.

The project work was completed satisfactorily and the experimental results were sent to the sponsor. It is reported that the party is taking further steps to implement the technology developed in the sponsored scheme.

3.2.2 *Chlorination of bauxite residue :* (SP-69/71)

Substantial quantity of bauxite residue (15-20 T/day) is available as bye product from the ferric alum plant of the sponsor. The residue shows, on analysis, TiO_2 about 20%, Al_2O_3 about 25%, Fe_2O_3 about 6% and SiO_2 about 16%. Preliminary work showed that chlorination of the residue to give both titanium tetrachloride and aluminium chloride is economically feasible if the percentages of silica and calcium oxide in the raw material is kept within limits.

Under the present sponsored scheme, study of chlorination of bauxite residue at high tempeatures has been undertaken first on a small scale and afterwards on a larger scale (0.5 to 1 kg. $TiCl_4$/hr.) to obtain process parameters and engineering designs.

Work on laboratory scale chlorination in a 3" i.d. tube has been successfully completed, and parameters such as temperature of chlorination, chlorine velocity, feed rate of oxygen and heat inputs were studied.

Based on the above data larger assembly was fabricated and operated· Chlorine and oxygen feed rates were controlled and $FeCl_3$ and $AlCl_3$ traps maintained at desired temperaturs. In order to increase the $TiCl_4$ condensation efficiency a shell and tube condenser was installed. With these arrangements the assembly has been working smoothly and satisfactorily. At the end of these trials a process packet including flow diagram, complete process description equipment and assembly sketches and material and energy balances, will be supplied to the sponsor.

3.2.3 *Recovery of chlorine from flue gases of vapour phase oxidation of* $TiCl_4$

In the chloride process for the production of rutile TiO_2 from ilmenite ore, it is necessary that the chlorine obtained during vapour phase oxidation of $TiCl_4$ be recovered for its use as a chlorinating agent.

The flue gases consisting of a mixture of N_2, P_2,CO,CO_2 moisture and chlorine have to be stripped with a suitable solvent which absorbs chlorine.

Initial experiments were carried out by absorbing pure chlorine gas in carbon tetrachloride. Experiments invertical column (50"x2.5" dia.) using synthetic mixture of gases (as obtained in vapour phase oxidation) showed promsing results when CCl_4 was recirculated till it was almost saturated with respect to chlorine.

These experiments will be scaled up and will be tried in the actual vapour phase oxidation set up. In addition other solvents such as sulphur monochloride and trichloroethylene will also be tried to determine the suitability and commercial feasibility of the methods of recovery.

3.2.4 *Recovery of HCl from iron chloride liquors obtained from ilmenite beneficiation*

The economic consideration regarding enrichment of ilmenite by carbothermic reduction and acid leaching indicates that the beneficiation process will be viable if hydrochloric acid is recovered from the bye product, ferrous chloride liquor.

Preliminary experiments were carried out with pure ferrous chloride, and conditions of reduction of $FeCl_2$ with hydrogen gas at 600° were optimized.

Crude ferrous chloride liquor obtained from beneficiation containing small amounts of free HCl, iron, calcium and titanium was purified by removing calcium and titanium ions as sulphates and by converting free HCl to ferrous chloride.

Reduction experiments were carried out in a horizonal furnace. It was observed that large quantities of hydrogen are required for effective reduction and the excess hydrogen has to be recovered and recycled.

Further work is in progress.

3.3 *Inorganic pigments*

3.3.1 *Cadmium pigments :* (AB-81/70)

Cadmium sulfoselenide pigments are used in plastic, ceramic and printing ink industries because of their fastness properties and heat and alkali resistant properties. Pigments worth about Rs. 39 lakhs are at present imported annually.

Three well known shades, namely, yellow, orange and red in these pigments were prepared, their tinting strengths were determined by ASTM method and were found to be comparable with the correspoding imported samples.

The tentative cost estimates showed that the processes developed for the preparation of the three varieties of pigments could be economically implemented if the main raw materials viz. cadmium and selenium are imported.

Samples of all the three shades have been sent to industry for evaluation and their reports are awaited.

3.3.2 *Pearl pigments :* (AB-91/71)

Pearl pigments are used to impart lustre, brilliance, depth and reflectance to plastic, leather, artificial pearls, moulded polyester for buttons, novelties, nail polish, paints and printing inks, etc. The demand is around 100 TPA valued at Rs. 120 lakhs.

Laboratory scale (50 g.) experiments for the preparation of bismuth oxychloride were carried out and the procedure was standardized. Basic lead carbonate was also prepared by carbonating the solution of lead acetate. 0.01 mole to 0.5 molar concentrations of lead acetate were used and the pH condition for getting the desired product was determined. A few autoclave experiments were also carried out. The particle size of the product obtained by carbonation was irregular but fine whereas the particle shape of the autoclave product was hexagonal but coarse. A few samples of lead arsenate and phosphate were also prepared.

Development of pearl pigments based on above chemicals will be considered if industry show interest in sponsoring further work.

3.4 *Industrial inorganic chemicals*

3.4.1 *Sodium hydrosulphite* (ATT-189/72)

The current method for making sodium hydrosulphite uses zinc dust which is mainly imported. Development of sodium formate route for making sodium hydrosulphite would avoid the use of imported zinc dust (worth about Rs. 50 lakhs/ year). This method may give finer product and is likely to be cheaper.

The chemistry of the process is given by the following equation :

$$HCOONa + NaOH + 2SO_2 \longrightarrow Na_2S_2O_4 + CO_2 + H_2O$$

Preliminary laboratory experiments(300 g./batch)to prepare sodium hydrosulphite by this route has shown encouraging results.

The product is of high purity and stability and the conversion of sulphur dioxide is satisfactory. Scaling up of the process is under study.

3.4.2 *Vapour phase oxidation of $SiCl_4$*

Finely divided silica known as ' white carbon ' has important application as a reinforcement filler in compounding silicones and rubbers.

Preliminary experiments showed encouraging results when silicon tetrachloride was burned in a flame propogated with L.P. gas and oxygen. Here the reaction involved hydrolysis, as sown below.

$$SiCl_4 + 2H_2O \longrightarrow SiO_2 + 4HCl$$

A larger reaction assembly has been installed wherein burning is carried out in a silica tube with multiple baffle arrangement fitted in a brick chamber. Gas absorption towers have been provided to absorb the effluent reaction product (HCl gas). Feed rates of silicon tetrachloride, fuel gas, oxygen and compressed air are being determined and burner design is being modified to obtain optimum burning. The product obtained from the reaction assembly had 1% moisture and surface area of about 100 m^2/g measured by the BET method. The bulk density was 0.06 g./cc.

Evaluation of the product for compounding rubber is being underaken.

3.5 *Organo-metallic compounds*

3.5.1 *Stabilizers for PVC :* (ATT-161/70)

Organo-tin compounds containing sulphur are extensively used for the stabilization of PVC. These stabilizers impart high heat stability and give transparent PVC products. These compounds are used in rigid PVC mouldings and will be in increasing demand in future. A process was developed for making dibutyl tin maleate (solid). Octyl tin compounds are non-toxic and are widely used in PVC bottles etc. The work of making dioctyl tin mercaptides and liquid dibutyl tin maleate is in progress.

3.5.2 *Tin and titanium organics :* (AB-99/72)

Some expertise has been developed in the synthesis of organo tin and titanium compounds during the last few years. Work on the preparation of organo-tin compounds required as PVC stabilizers has been undertaken.

A new process for the manufacture of dibutyl tin from butyl iodide and tin metal was developed. The economics of the process much depended on the recovery of iodine on the basis of butyl iodide consumed. Using cheaper butyl chloride in place of butyl iodide the preparation was studied in detail. However another procedure using butyl chloride, magnesium and stannic chloride was later standardized on 500 g./batch of dibutyl tin oxide.

Using similar procedure dioctyl tin oxide was successfully prepared. These organo-tin oxides were then converted into PVC stabilizers by reacting them with suitable organic acids and mercaptides.

Tributyl tin oxide finds application as an ideal wood preservative. A laboratory method (500 g./batch) for making this material from butyl chloride, magnesium and stannic chloride has also been standardized. A sample (10 kg.) was supplied for defence requirements. Another sample was sent to Plywood Research Institute, Bangalore, for making adhesives with fungicidal properties.

Several new benzyl tin organics were prepared. A colourless titanoxy stannate was prepared from dibutyl tin oxide and octyl titanate and its PVC stabilizing property is under study.

3.5.3 *Coordination compounds :* (B-6/63)

In continuation of the previous work on the reactivity of chelated ligands, bis-(oximinopropyl-salicylaldoximato)-copper (II), bis-(oximinopropyl-5-nitrosalicyladoximato)-copper (II), bis-(oximinopropyl-5-

bromosalicylaldoximato)-copper (II), bis-(oximionopropyl-β-resorcylaldoximato)-copper (II), bis-(oximinopropyl-5-nitro-β-resorcylaldoximato)-copper(II) and bis-(oximinopropyl-2-hydroxy-1-naphthaldoximato)-copper (II) have been isolated for the first time, (from the respective parent chelates) using propionic anhydride. Some butrylated and valerylated derivatives also have been successfully isolated.

.New beryllium (II) chelates with various substituted acetoacetanilides, for instance, 4-chloroacetoacetanilide, 2,5- and 2,4-dichloroacetoacetanilides, 4-bromoacetoacetanilide, 2-methoxy-, 4-methoxy- and 2, 5-dimethoxy-acetoacetanilides and 2,5-dimethoxy-4-bromoacetoacetanilide were isolated and characterized from their elemental analysis, IR and NMR spectra. These chelates on bromination and nitration yielded the corresponding bromo- and nitro-substituted derivatives.

O-Vanillin complexes with Cu(II), Co(II), Ni(II), Be(II) and Al(III) were found to undergo nitration and bromination reactions. The products obtained were identified as bis-(5-nitro-*o*-vanillinato)-M(II) or bis-(5-bromo-*o*-vanillinato)-M (II), where M = Cu(II), Co(II) and Ni(II). However, nitration of beryllium(II) and aluminium (III) chelates resulted in a metal free product namely, 5-nitro-*o*-vanllin.

Work on cyclopentadienyl metal complexes has been continued since *similar compounds have been reported to be of commercial impotance in* the fields of polymerization, nitrogen fixation, antiknock fuel additives, etc.

In the course of studies on cyclopentadienyl titanium complexes, some new bimetallic complexes containing titanium bonded to cyclopentadienyl groups were synthesized. With zinc, magnesium and tin as other metal, these complexes had a sulphur-containing ligand, toluene-3,4-dithiol or thiosalicylic acid. These complexes having the compositions $(\pi\text{-}C_5H_5)_2$ TiLM Cl_2.X, $\{(\pi\text{-}C_2H_5)_2\ Ti\}_2L_3M.X$, etc. (where L = sulphur containing ligand, M = divalent metal and X = coordinated solvent molecule), had green colour and were characterized by their IR and NMR spectral studies. Synthesis of similar complexes with trivalent aluminium as the other metal was not successful.

3. 5. 4 *Surface adsorption of long chain paraffin metal sulphates :* (B-6.7/71)

A thorough study of the surface adsorptions of long paraffin chain metal sulphates of the type $C_{10}SO_4M$ (M : Ca, Mg, Na, Pb, Mn, Cu,Co) made through surface tension measurements of their aqueous solutions (in the conc. range of 1×10^{-4} to 2×10^{-1} g. mols/litre at 50°) showed that the critical micelle concentrations for all the metal decyl sulphate solutes were near about 10^{-3} g. mols/litre. Using the modified Gibb's equation, the concentrations of the solute molecules at the solution-air interface were evaluated.

The area per molecule of the solute at the surface or the amount of the substance adsorbed per unit area of the surface as in accordance with the variation of the ionic radii of the respective metal ions.

In order to further confirm the nature, behaviour, shape and size of the above solutes in the bulk as well as in the surface of the solvent, light scattering measurements have been conducted. Rayleigh scattering (RO) and differencial refractive index increments dy/dc led to the micellar molecular weights and size and charge of the micelles.

4. STUDIES IN PHYSICAL ORGANIC CHEMISTRY

4.1 *Conformational studies :* (B-8.45/69)

When a secondary or primary hydroxyl group is esterified, the proton or protons on the carbon bearing the hydroxyl group undergo considerable deshielding in their NMR spectra. This can be interpreted as either due to the inductive effect of the carbonyl which has a resonating structure or due to the field effect of the carbonyl, or due to the operation of both these effects. Studies on suitable model compounds having γ-and δ-lactones fused to rigid systems have now shown that this deshielding effect is almost entirely due to the field effect of the carbonyl and that the contribution of the inductive effect, if any, is very small.

4.2 *Steroidal alkyl ethers :* (B-8.55/70)

The mass spectral fragmentation of saturated and unsaturated steroidal alkyl ethers and ethers of 4,4-dimethyl steirods are studied and compared with the fragmentation of steriodal alcohols, trimethyl silyl ethers and ethylene ketals. It is found that unlike the trimethyl silyl ethers, the small fragment containing the alkoxy group is neither the base peak nor a very strong peak in the spectrum. A singificant peak occurs at (M-ROH $)^+$, however, sometimes even constituting the base peak of the spectrum. The fragmentations of the alkyl ethers are largely dependent on their environments. The generalization sometimes made that methyl ethers are better leaving groups than the corresponding alcohols is not always valid. It has also been found that in the ring A or B substituted or unsaturated steroids, the side chain is found to cleave successively from the isopropyl group towards ring D, as in corresponding hydrocarbon chains.

4.3 *Mass spectrometry :* (B-5.7 & 5.8/65)

4.3.1 *The electronic states of molecular ions and their influence on rearrangemetns :* The hydrogen and hydroxyl rearrangements occuring in *meta* and *para* substituted benzaldoximes reveal significant differences. The hydrogen transfer is significant in *para* isomer whereas hydroxyl transfer is more significant in the *meta* isomer. The results have been rationalized on the basis of diffrent electronic states of molecular ions and the ability of electron and charge site to trigger hyrogen and hydroxyl transfer.

The effect of introducing substituents in the aromatic ring on the intensity of molecular ions has been studied in some *para* substituted ketals. The results have been sent for publications.

4.3 2. *Photochemical, pyrolytic and electron impact processes : Similarities and differences:* The retro Diels Alder reaction in epimeric 3,5,6-triphenyl 4-nitro-Δ'-cyclohexenes has been studied under electron impact and pyrolytic conditions. Under electron impact, it is a preferred process. Thermally, the compounds undergo aromatization to give 1,2,4-triphenylbenzenes in high yields. The influence of stereochemistry on fragmentation modes operating in these compounds has been rationalized. In the mass spectra of the corresponding 4-amino compunds the charge distribution in the molecular ion appears to be different as is shown by the charge distribution in the retro Diels Alder products. This work is being extended to compounds containing phenyl and heterocyclic groups. The chemical ionization behaviour of these systems are being examined.

4.3.3 *Mass spectral analysis of oligosaccharide derivatives :* The mass spectra of the per methyl ethers of some fructose containing oligosaccharides and their alditol methyl ethers have been examined with a view to find out the usefulness of mass spectrometry to locate fructose units in these sugars. The results have been presented at the 8th International Symposium on the Chemistry of Natural Products at New Delhi.

4.3.4 *Mass spectral analysis of pesticidal residues*

For the repository of the pesticide stadards, a standard sample of malathion was prepared. Comparison of the sample with a US standard showed NCL sample purer. Samples were sent to a few laboratories in India for assessing purity.

Preliminary work connected with the analysis of malathion residues on wheat using mass spectral techniques has been nearly completed. Attempts have been made to identify the metabolites.

4.4 *Studies on conjugated system :* (B-5.2/62)

The earlier work on phthalic esters and *o*-dinitro benzenes has been extended to 4- and 5-substituted 2-nitrobenzoic esters. In the latter series chemical shifts for $C_{(3)}$-proton vary with substituents in the predicated manner. The $C_{(6)}$-proton shifts behave generally in the same way as the shifts for the $C_{(3)}$-protons of 4-substituted phtahlic esters. The behaviour of the fluoro derivative is however anomalous. The $C_{(3)}$-proton shifts in 4-substituted 2-nitrobenzoic esters are also totally anomalous. The reasons for this will be sought in future work. The implications of the results so far obtained in these studies for resolution of optically active compounds will also be studied.

4.5 *Structural and stereochemical studies :* (B-8.23/68)

4.5.1 *Ascorbic acid and its derivatives :* The work on ascorbic acid derivatives has been continued and structural isomerism in these studied. Definite evidence for the existence of some of these in bicyclic form has been obtained. 2- C-Benzylascorbic acid seems to undergo an interesting transformation on acetylation. The nature of the transformation is not yet clear; but it is very likely that it has an important bearing on their stability of the bicyclic form of the material. In order to bring out the significance of the present results for the redox beaviour of ascorbic acid and dehydro-ascorbic acid, additional work is proposed to be carried out.

4.5.2 *Analysis of NMR spectra of multiple spin systems :* In the course of earlier studies several instances have been encountered where chemical shifts observed are difficult to rationalize. It is found that extended studies of other systems will be required to make clear the origin of these shifts. In this connection preliminary work has been done on a number of systems.

4.5.3 *Proton chemical shifts in condensed thiophenes :* The spectra of condensed thiophenes studied earlier have been re-examined. More chemical shifts have been determined with the aid of double resonance experiments. The ring current and steric effects for the various protons have been estimated quantitatively using relevant information from literature. The question of sulphur d-orbital participation in bonding is being reconsidered in the light of the results obtained.

4.6 *Studies of chemical reactivity with NMR :* (B-5.10/70)

4.6.1 *Condensation of 2,4-dinitrochlorobenzene with o-hydroxyaryl amides :* Some work reported about ten years ago from this laboratory indicated that the base catalyzed condensation of 2,4-dinitrochlorobenzene with *o*-hydroxyl-arylamides probably involves intramolecular nucleophilic catalysis and that the reaction may proceed by direct attack on the amide nitrogen. There has been recent report indicating that this conclusion may be incorrect. The properties of the materials involved have now been examined with the help of nuclear magnetic resonance and valuable results obtained. The work will be continued and it is hoped that a clarification of the mechanism of the reaction will be available.

4.7 *Electronic spectra and electronic structure* : (B-5.9/68)

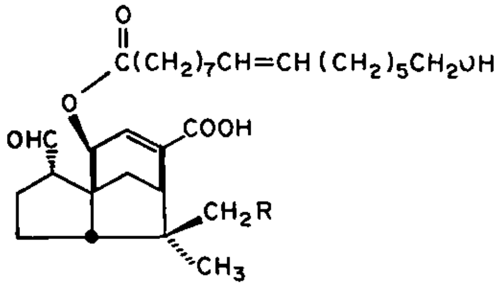
The electronic structures of ketene and acyl cation have been investigated by the molecular orbital method in the CNDO/2 approximation. Minimization of energy with respect to the various geometrical parameters has been done. The results obtained will be used along with those to be obtained for some larger related systems for a comparision of properties of derivatives of carboxylic acids.

5. NATURALLY OCCURING ORGANIC COMPOUNDS / PRDOUCTS

5.1 *Constitution of lac* : (SP-24/61)

The scheme envisages investigations on the chemical constitution of lac resin.

Soft resin was isolated as a more soluble fraction of lac. On systematic column chromatography it gave four pure components. The structures of two of them were established and their syntheses achieved earlier. The remaining two compounds are now shown to be the esters (I) and (II) derived from jalaric acid and laccijalaric acid respectively.



(I) R=H

(II) R=OH

The ' odoriferous material ' from Palas seed lac has been isolated (8% yield) and chromatographed to furnish two pure compounds. Structural work on these is in progress.

5.2 *Wood resinoids*

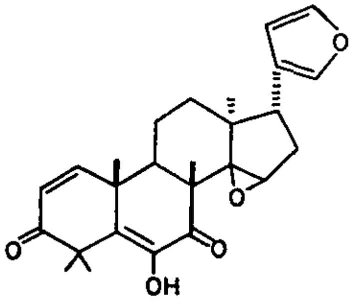
5.2.1 *Soft organic flux* : (ATT-85/68)

Good quality soft organic flux used for soldering electrical joints of sophisticated electronic equipment used in radar and aircraft industry is not made in India and is mostly imported. The flux should be non-corrosive and should not leave any residue. Attempts to produce a soft flux entirely from indigenous resinous raw materials have been made and a sample which showed promise has been sent to industry for evaluation.

5.2.2 *Cedrela toona :* (B-7.2/67)

Besides the known major limonoid cedrelone (I), three new minor limonoids-isocedrelone(II), compound-X and compound-Y have been isolated from the petroleum ether solubles of the acetone extract of *Cedrela toona* Roxb. This is the first reported isolation of isocedrelone as a natural product.

Based on spectral data and chemical correlation with the known compounds, meldenin and Δ^1-meldenindiol, structures (III) and (IV) have been assigned to compound-X and compound-Y respectively. Chemical investigation of *Cedrela toona* is now complete.



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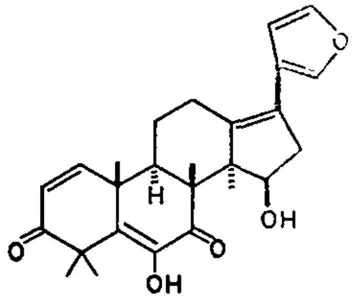

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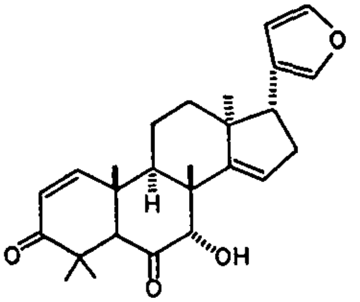

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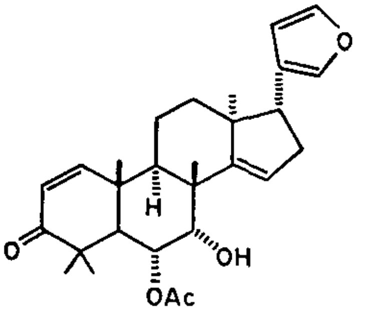


IV

5.3 *Wood phenolics :* (SP-28/66)

The 5-year project on wood phenolics was terminated in September 1971 and the main results are outlined below.

From *Artocarpus*, *Morus*, *Garcinia* and other genera 48 new compounds have been isolated; and the structures of 23 have been fully determined. Several belong to new types. In addition, at least 35 compounds were isolated which were known earlier to occur in other plant species or as synthetic products.

Artocarpin from *A. heterophyllus* exhibited significant antitubercular activity in preliminary tests; although the compound proved to be of no practical value by further tests, it indicated that wood extractives may be a source of products with useful biological properties.

The Moraceae is a large family of over 50 genera and nearly 1400 species, including such important genera as *Artocarpus*, *Morus* and *Ficus*. A detailed study of the wood phenolics of 6 *Artocarpus* and 5 *Morus* species has led to the isolation of several flavonoids with certain common features. The present state of our knowledge of the chemistry of the Moraceae has been reviewed with particular reference to wood phenolics. The chemotaxonomy of the Moraceae has been discussed in relation to flavonoids and stilbenes; the phenolic constituents can be useful in the taxonomy of the Moraceae if the investigation is extended.

Tissue culture studies were undertaken with the object of using this technique for following biosynthetic pathways of phenolic constituents of forest trees. Optimal conditions for the continuous culture of teak, jack, mulberry and poplar tissues have been established.

From the teak tissues, two isomeric dihydroxy-methoxymethyl-anthraquinones have been isolated. The structure of one has been determined, and it has not been found in teak heartwood so far. The structure (2, 5-dihydroxy-1-methoxy-3-methylanthraquinone) has been confirmed by the synthesis of its dimethyl ether. Labelled acetate has been incorporated, and further experiments with labelled juglone and shikimic and mevalonic acids should provide evidence for its biosynthesis.

A new triterpene has been isolated from *Populus nigra* tissues, and its structure partially elucidated.

5.4 *Perfumery compounds and essential oils*

5.4.1 *Development of perfumery products based on longifolene and isolongifolene :* (SP-53/70)

The sponsored scheme attempts to find suitable outlets for the commercial utilization of Δ^3-carene, longifolene and isolongifolene available from Indian turpentine oil.

Based on earlier work, a number of compounds prepared from longifolene, isolongifolene and Δ^3-carene have found market acceptability as perfumery materials. The sponsor has already established commercial production of them.

It was observed that during the preparation of these derivatives a part of longifolene isomerises to isolongifolene.

Oxidation of isolongifolene furnishes two products, a ketone and a lactone. Experimental conditions for these products have been optimized on 1 kg. batch scale, and the details supplied to the sponsor.

5.4.2 *Development of perfumery products based on Δ^3 -carene :* (SP-54/70)

Gardinone, a perfumery material obtained from Δ^3 -carene is a mixture of five constituents. All these compounds have been isolated in pure form by chromatography and structure assigned to them on the basis of their spectral (IR, PMR and Mass) data.

A technical method for the preparation of an epoxide of Δ^3-carene (500 g. scale) which gives good yield has been worked out and the material sent to the sponsor for its evaluation in perfumery.

Δ^3-carene was hydrogenated to carane on a large scale. The cyclopropane ring of carane was cleaved and the resulting olefins hydroxylated and fractionated to furnish three diols. Dehydration of the diols yielded a mixture of menthones. These were converted to menthols by reduction.

Conditions have been standardized to get the maximum yield of the oxidation product of Δ^3-carene wherein one major and several minor products were obtained.

5.4.3 *Solvent extraction of sandalwood oil :* (SP-74/71)

The conventional method for obtaining sandalwood oil from sandalwood, is by steam distillation. As petroleum solvents are now available in the country, work on the project has been undertaken to find out the commercial feasibility of solvent extraction process.

The objective of the project is to develop an economic process for extraction of the sandalwood oil (by solvents) acceptable to consumers.

Following preliminary investigations, several solvents have been tried for the extraction of the oil from sandalwood powder. Some of the results are encouraging. Purification of solvent extracted sandalwood oil (crude) and evaluation of its properties are in progress.

5.4.4 *Utilzation of β-himachalene :* (AB-17/67)

β-himachalene is a major constituent of the essential oil of *Cedrus deodara* Loud. Attempts are in progress to find uses for the same.

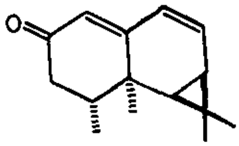
The preparation of β-himachalene oxide has been standardized and it has been tested for juvenile hormone activity against red cotton bugs; the results are encouraging.

The oxygenated portion of the essential oil of *Cedrus deodara* Loud. has also been tested for insect hormonal activity, with encouraging results.

The oxidation mixture from dihydrohimachalene has been seperated and the major product shown to be *ar*-himachalene.

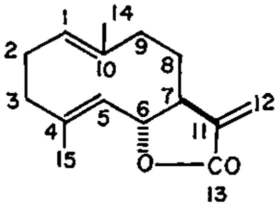
5.4.5 *Chemical examination of jatamansi oil, transformation products and synthesis of terpenoids :* (B-7.4/60)

The structure of nardostachone isolated from *N*-jatamansi oil has been revised as 1,8,9,10-tetradehydroaristolane-2-one.



5.4.6 *Transformation products of constunolide and dehydrocostus lactone :* (B-8.15/66 and B-8.19/65)

Costunolide (I) and related products when subjected to selenium dioxide oxidation gave the corresponding iso-compounds (II) in which the C_1-C_{10} double bond of (I) has migrated to the C_9-C_{10} position. The minor products of the reaction included primary hydroxylactones and in the case of dihydrocostunolide (III) the hydroxylactone (IV) is isolated and characterized.



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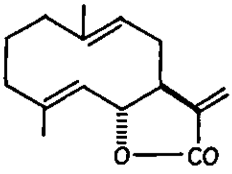

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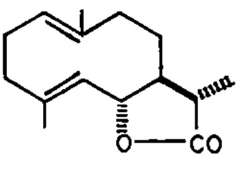

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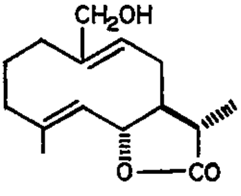
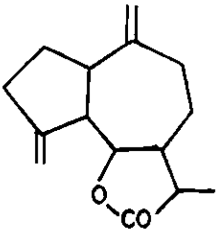


IV

Dihydrodehydrocostus lactone (V) on partial hydrogenation lead to a mixture of two dihydro derivatives, of which one compound, viz. (VI) in which the cyloheptane exodouble bond of (V) has migrated to the tetrasubstituted position, has been isolated and characterized.



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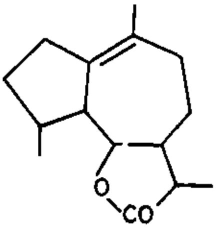

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5.4.7 *Compounds related to selinane, elemane and p-menthane :* (B-8 16/67)

Compounds related to selinane and clemane are widely distributed in nature and some of them such as santonin are of medicinal value. The epoxidation of *cis*-pulegol and 2-benzylidene has been studied. *p*-Menth-8(10)en-3, 9-olide has been synthesized.

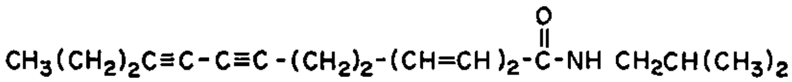
5.5 *Insect and pest control products*

5.5.1 *Insect hormones and phermones :* (AB-74/69)

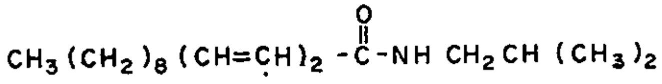
Under this project several indigenous plant products will be screened for juvenile hormone activity. Chemistry of the plant materials is being studied and compounds are synthesized from their isolates which may exhibit the desired activity.

Spilanthes oleraceae

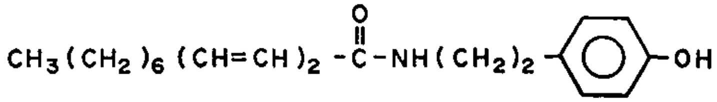
Three compounds isolated from the petroleum ether and chloroform extracts of *Spilanthes oleraceae* have been characterized by physical and chemical methods : two are isobutyl amides (I) and (II) and the third (major) is a tyramide (III).

I 

$CH_3(CH_2)_2C\equiv C-C\equiv C-(CH_2)_2-(CH=CH)_2-\overset{\overset{O}{\|}}{C}-NH\ CH_2CH(CH_3)_2$

II 

$CH_3(CH_2)_8(CH=CH)_2-\overset{\overset{O}{\|}}{C}-NH\ CH_2CH\ (CH_3)_2$

III 

$CH_3(CH_2)_6(CH=CH)_2-\overset{\overset{O}{\|}}{C}-NH(CH_2)_2-$

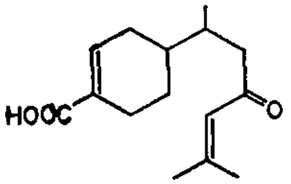
Mass spectrum and GLC of the saturated fatty ester of hydrolysed (III) show the presence of small quantities of other homologues.

Vitex negundo

Work has been initiated on the constituents of *Vitex negundo*. Acetone extract of the powdered wood deposited a yellow powder. This mixture of polyhydroxy compounds could not be separated by chromatography over cellulose powder or silica gel. Chromatography of the acetylated material, however, yielded two crystalline compounds.

Cedrus deodara Loud

The acetone extract of *Cedrus deodara* Loud. has been found to exhibit juvenile hormone activity against *Dysdercus koenigii*. In efforts to pinpoint the active compounds, it has been found that the activity resides almost entirely in the acidic terpene fraction of the total wood extract, in which three monocyclic (I-III) and three (IV-VI) bicyclic sesquiterpene acids have been identified.



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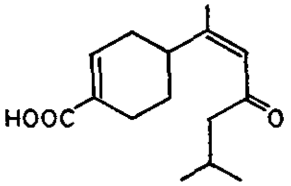

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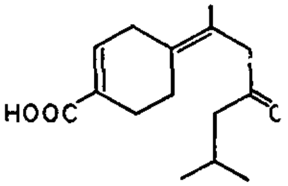

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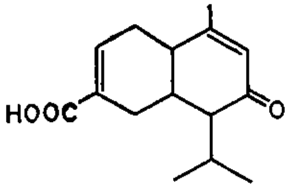

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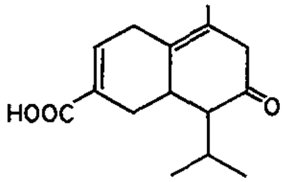

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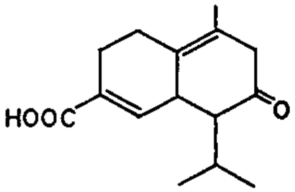


VI

5.6 *Drugs from medicinal plants*

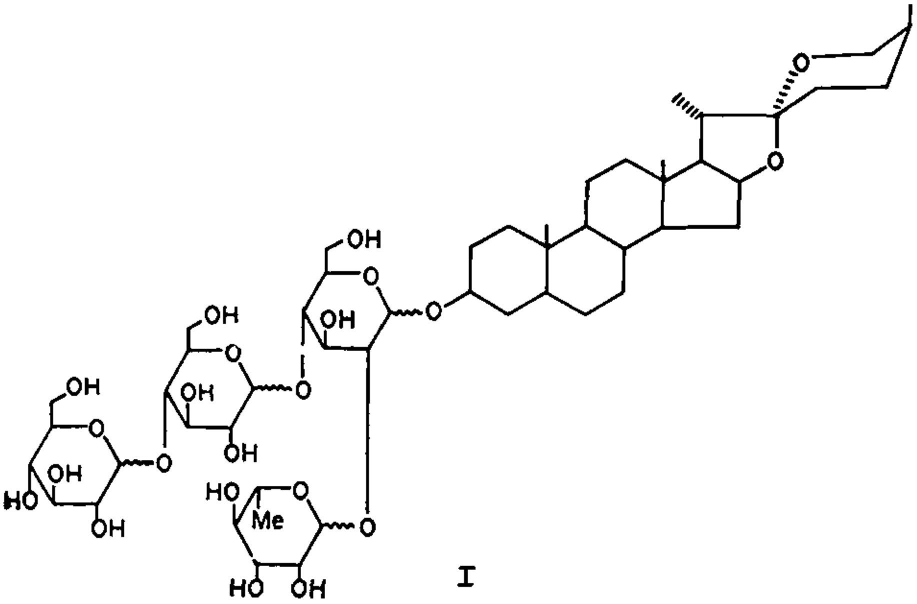
5.6.1 *Composite drug research scheme on Indian medicinal plants:* (SP-23/65)

At present work on chemistry of pharmacologically active extracts of *Boerhavia diffusa* Linn (Punarnava), *Asparagus racemosus* Willd (Shatavari) and *Commiphora mukul* (Gugul) is being continued.

Boerhavia diffusa Linn : Ethyl acetate extract had earlier shown anti-inflammatory activity. Initial broad cut chromatographic separation of this extract into four fractions and their pharmacological testing demonstrated that the activity was spread in all the fractions. Systematic chromatographic separation of the extract to isolate the individual components is in progress.

Attempts to isolate the active principle (diuretic) from the petroleum ether extract is under way.

Asparagus racemosus Willd (Shatavari) : Shatavarin-I, the major saponin, isolated from the alcohol extract has shown to have anti-oxytocic as well as galactagogue properties. It was established that in shatavarin-I, one rhamnose and three glucose units are linked to the aglycon-sarsasapogenin. The results of methanolysis of permethyl shatavarin-I copuled with the fact that shatavarin-I co-occurs with shatavarin-IV (see below) led to tentative assignment of structure (I) to shatavarin-I.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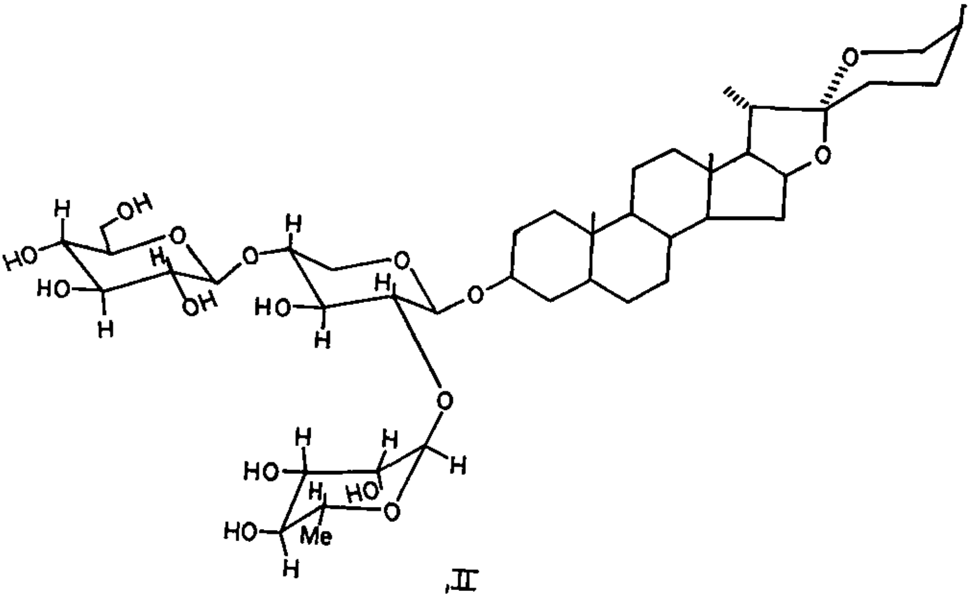
Structure (II) was assigned to shatavarin-IV based on spectra[1] analysis and degradative studies of shatavarin-IV and its derivatives.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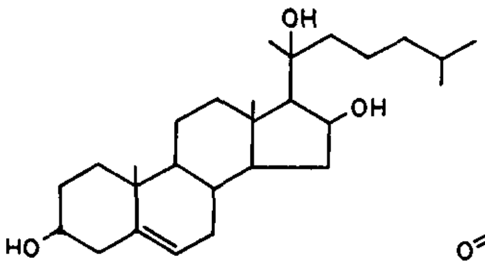
Commiphora mukul (Gugul) : Structures were assigned to three of the steroids, isolated from gum gugul earlier and the major pregnane type compound (Z-gugulsterone) was shown to exhibit a high degree of anti-cholesterol activity. Structures of two more minor steroids- gugulsterol-II (III) and gugulsterol-III(IV) - have now been established based on spectrochemical data and the structure of gugulsterol-II confirmed by partial synthesis from diosgenin.



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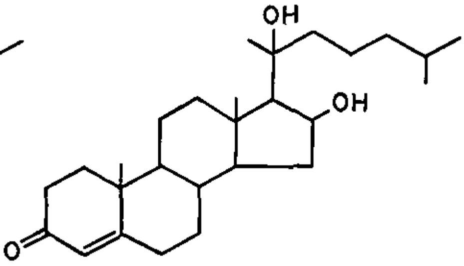

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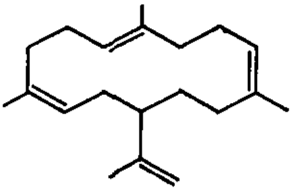
A diterpene hydrocarbon (V) and a diterpene alcohol (VI) have been isolated by extensive chromatography of the petroleum ether extract and their structures established on the basis of spectral data and degradative studies.



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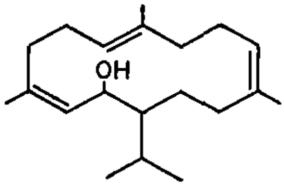

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5 6.2 *Extraction of morphine and other alkaloids from lanced poppy straw:* (SP-67/71)

Lanced poppy capsules still contain some alkaloids and hence if an economic method for the isolation of these alkaloids can be worked out, additional quantities of alkaloids will become available. It is estimated that nearly 4.725 tonnes of lanced poppy capsules are available per year.The scheme has been undertaken with a view to develop a technically feasible method for the extraction of alkaloids from lanced poppy capsules.

Recovery of morphine, as estimated in the extracts, from lanced poppy capsules using water containing lime or sodium bisulphite as the medium of extraction was found to be very low as compared to that obtained using *n*-butanol.

For actual recovery of alkaloids the extract has been passed through a column of cation exchange resin and the alkaloids adsorbed on the column have been released by ammoniacal methanol. A dark semi-solid has obtained on concentration of the above alkaloid, which has been worked up for recovery of morphine and other alkaloids. In addition to morphine (0.08%), narcotine (0.010%) and codeine (0.044%) have been obtained as crystalline bases. Another alkaloid (0.017%) so far not identified, has also been obtained. No thebaine could be isolated although its presence has been indicated by TLC.

Attempts to separate codeine from thebaine quantitatively using (i) gradient elution in a cation exchange resin column and (ii) TLC coupled with densitometry with a view to estimate codeine in capsules have not been successful.

5.6.3 *Colchicine :* (ATT-68/68)

Colchicine is valuable both in pharmacy and plant breeding and is in short supply in the world market. Indian demand is estimated at about 3-4 kg./year. There is a likelihood of an export market for this drug.

Seeds from the fruits of some new *Iphigenia* species (variety from Dhulia district) on extraction and usual work up gave 0.6-0.7% colchicine.

Effect of time of collection as well as drying conditions of the seeds of *Iphigenia stellata* fruits (twenty four samples) on the colchicine content have been studied.

Three different experiments on the extraction, isolation and purification of colchicine from seeds of *I. stellata* on one kg. batches were carried out, resulting in 40 g. of colchicine. A systematic procedure for isolation and purification of colchicine from *I. stellata* has thus been standardized on one kg. per batch scale.

The major components of the petroleum ether extracted material of the seeds of *I. stellata* have been identified.

5.7 *Polysaccharides and related products*

5.7.1 *Pulping of Kashmir soft woods :* (SP-30/67)

Suitability of Kashmir soft woods is being assessed for the manufacture of rayon grade and tyre cord grade dissolving pulp. The project has been sponsored by J and K Govt.

Except for the filterability value, pulps produced were found suitable for rayon staple and tyre cord. Work was specially undertaken for improving the filterability of the pulps during the period under report.

Suitable modifications were introduced in the processing of pulps and samples produced under optimum conditions were sent for testing to Forest Products Research Laboratory, Stockholm, Sweden. Counter testing of the samples was done under similar testing methods. Both the results are found in good agreement and the filterability of pulps produced in this project has now been proved to be comparable to similar commercial pulps.

5.7.2 *Production of Xylit :* (SP-81/72)

Xylit or Xylitol is a polyhydric alcohol currently in use as a harmless weetening agent beneficial to diabetics. It is the sweetest of all the alditols known so far.

Xylit is obtained by reducing xylose which occurs as the polymer xylan in hemicellulosic fractions of various agricultural wastes often contaminated with other pentosans and hexosans.

The project has been undertaken to survey some of the agricultural wastes rich in xylan and establish the feasibility of production of xylit from them on the basis of laboratory scale experiments.

Four agricultural wastes have been investigated so far and one of these has been found to be a good source of uncontaminated xylose. Work is in progress to determine the economic feasibility of production of xylit from this source.

5 7 3 *Dissolving pulps :* (PP-2/58)

Eucalyptus hybrid : It is reported by a commercial tyre cord unit that NCL-APS tyre cord pulp viscose (from Eucalyptus hybrid of South Indian origin) could be spun normally and super tyre cord may be made out of it. This work was extended to Eucalyptus hybrid available in U. P. and similar results were obtained. The know-how on U. P. Eucalyptus hybrid is being offered to industry.

In order to improve yield of pulp from Eucalyptus hybrid, prehydrolysis-neutral-sulfite semi-chemical pulping was adopted. It was found that the species respond well to above process. The increase in yield is about 7% in case of rayon grade pulp. The physical and chemical properties of these pulps are good and could be normally converted to rayon yarn of acceptable quality.

Silver fir (Abies pindrow) : Single stage kraft pulping and two stage craft pulping methods were adopted to silver fir, followed by cold alkali extraction in bleaching. The properties of single stage kraft pulp are suitable for rayon type pulp, while following two stage kraft process, pulp with 5% high α-cellulose content was obtained.

5.7.4 *Starch phosphate :* (ATT-54/64)

Starch phosphate has manifold applications in food, pharmaceutical and other industries. It is used in large quantities in the USA but not yet produced in India. A process has been developed for the production of tapioca starch phosphate which can be used with advantage in some food preparations. The product is a cream colour powder and is soluble in cold water. Its spectral and quantitative analysis indicated that the trace elements present in the preparation are within limits of impurities permitted by B. P. specifications. The product has been tested for its application in food industry at CFTRI, Mysore, and found to improve the texture of vegetable protein preparations, rehydration capacity of the food products and bodying characteristics of milk like protein beverages. The samples tested were also found to form useful component of weaning food formulations.

5.7.5 *Utilization of tamarind kernel powder (TKP) :* (ATT-137/69)

Attempts are in progress to modify TKP so that the same can be used with additional advantage in textile printing and sizing etc.

TKP phosphate and TKP borate have been made and evaluated at ATIRA, Ahmedabad. TKP phosphate has been reported to resemble hydrolysed corn starch with superior film extensibility characteristics than corn starch and hydrolysed corn starch. TKP borate gave more viscous paste with better shelf life and worked out more economical. Satisfactory performance has been reported for carboxy methyl TKP as thickener of vat and basic dyes in textile printing.

5.7.6 *Gum arabic substitute* : (ATT-107/70)

A process has been worked out to produce substitute for gum arabic from indigenous raw materials. A product, pale yellow in powder form, conforming to U. S. P. specification for acacia but with positive test for starch, has been produced and approved by P & T department and a pharmaceutical firm. Further work is in progress to produce substitute for gum arabic with negative test for starch.

5.7.7 *Modified cotton fabrics :* (ATT-158/70)

Surface acetylation of cellulose

In this joint programme by NCL and BTRA, Bombay, surface acetylation of cotton fabric is attempted to improve the properties of cellulose fabrics. Using ketene as the acetylating agent, several samples were treated using various types of acid catalysts. Phosphoric acid and perchloric acid in certain percentages do effective surface acetylation in ranges between 10-20%. However, such acetylated cloth appears to be similar to untreated material. It is likely that partially acetylated cellulose may have better mildew resistance.

...5

Internal lubrication of cotton fabrics

Many of the resin finishes carried out on cotton to produce the wash and wear effect tend to produce stiffness in the finished fabric resulting in loss in feel and handle of the fabric as well as reduced tear strength. One method of avoiding this unwanted side effect is to lubricate the treated fabric with lubricating agents like long chain compounds, silicone compounds, etc. Such superficial treatments produce effects that have no wash fastness and as such are not permanent.

In the present approach, the chemical combination of long chain residues with the hydroxyl groups of cellulose is attempted to produce covalently linked softners which become part of the fibre thus imparting permanent internal lubrication.

5.7.8 *Flocculants for coal washery :* (ATT-167/70)

Flocculants are used as sedimentation aids for clarification of coal washery water. The project has been undertaken in collaboration with CFRI, Dhanbad. The estimated demand of flocculants according to CFRI is of the order of 200 TPA by 1974-75. A series of samples made from indigenous polysaccharide raw materials and agricultural wastes were evaluted at CFRI and three of them were reported superior to the presently used product. More samples are under evaluation and on the basis of results, the best sample will be tried on pilot plant scale with different types of coal slurries.

5.7.9 *Hydroxyethyl starch :* (AB-60/68)

Out of the five paper mills contacted for testing hydroxyethyl starch (HES) for its utilization in paper industries, two mills have sent their reports. The results indicate that samples of HES cannot be used as a substitute for starch (Tapioca) for the level of starch addition usually used in paper industries. Samples are also sent to SASMIRA, Bombay, to conduct trial runs for sizing with different textile materials.

Since it has been observed that the HES samples are not suitable for textile printing as well as in paper industries, the project is kept in abeyance till industry shows real interest in sponsoring further work in the project.

5.7.10 *Cashew nut shell gum (CNS) gum :* (AB-76/69)

In India about 1,50,000 tons of cashew nuts are processed to produce cashew kernels and cashew nut shell liquid. The outer shell comprises two-third of the whole cashew nut and it is estimated that 1,00,000 tons of cashew nut shells are available, which at present are only used as a fuel. A process has been developed to utilize this waste material to obtain a water soluble polysaccharide (CNS gum). The CNS gum is a highly water soluble, colourless, non-toxic, free flowing powder which possesses good thickening properties. It can be used as thickening agent for icecream, binder for tablets in pharmaceutical industry, drilling muds in mining industry, etc.

The process developed on 1 kg./batch scale of CNS gum consists of extracting CN shells with water; concentrating the extract to a small volume; precipitating the gum with a suitable solvent and filtering and drying the gum.

The process is offered to industry.

5.7.11 *L-Arabinose :* (AB-87.1/70)

L-Arabinose is an important rare sugar required in biochemical research. It is usually obtained from mesquito gum, available in Mexico and southern parts of America. A cheap and economical process to prepare this rare sugar from an indigenous gum has been developed.

The indigenous gum is partially hydrolysed and the degraded gum is separated from the aqueous extract containing different sugars. L-Arabinose is isolated as a crystalline white material from the aqueous extract by fractional crystallization.

Laboratory experiments have been conducted on 5 kg. of raw material (or 500 g. of pure L-Arabinose).

The product has been analyzed and found to conform to the required specifications. The process is offered to industry.

5.7.12 *2-Amino-D-glucose hydrochloride :* (AB-87.2/70)

D-Glucosamine hydrochloride (2-amino-D-glucose hydrochloride) is one of the important chemicals required in biochemical research and pharmaceutical industry. It is generally prepared abroad from shells of lobsters and crabs. A cheap and economic process to prepare this chemical from a waste obtained in sea-food canning industry has been developed.

The waste from sea-food canning industry is dried, powdered and treated with dilute acid. The residue, on hydrolysis and subsequent careful separation yields D-glucosamine hydrochloride (GAH) as a white crystalline material.

The process has been developed on laboratory scale on 2.5 kg. of raw materials/batch (250 g. of GAH/batch). The product has been tested and found to conform to standard specifications. The process is offered to industry.

5.7.13 *D-Galactose :* (AB-87.3/70)

Conditions have been established to obtain pure D-galactose [m. p.

158 °, $\alpha_{+} = + 82$ (water)] in a crystalline form in about 20% yield from CNS gum. Further work to establish the optimum conditions to increase the yield of D-galactose is in progress.

5.7.14 *Glycosides :* (B-7.16/71)

It has been found earlier that stannic chloride catalysed glycosidation of phenols leads to the formation of either of the anomers predominantly depending on the conditions employed. On the basis of further studies a possible mechanism has been put forward for this reaction. A number of mannosides have been prepared by this reaction and it has been found that aryl α-D-mannoside is predominantly formed as anticipated due to the presence of an axial hydroxyl group in the 2-position of D-mannose.

This glycosidation reaction has been successfully employed in the preparation of several new and known aryl alkyl- and alkyl-glycosides, which has established its wide applicability.

5.7.15 *Synthesis of D-fucose*

D-Fucose is one of the rare and costly sugars required in biochemical research. The current price of D-fucose is of the order of $ 20 for 5 g. in USA. D-fucose has some export potential.

The starting material for the synthesis of D-fucose is D-galactose. A process has been developed for the preparation of D-galactose from an indigenous agricultural waste.

D-Galactose was converted to its 1,2,3,4-di-*o*-isopropylidene derivative and the crude reaction product was directly tosylated to obtain pure 1,2,3, 4-di-*o*-isopropylidene-6-*o*-*p*-toluenesulphonyl- α-D-galactopyranoside,m.p.103°, in 40% yield. Work is in progress to convert this product to 6-iodo derivative followed by acid hydrolysis to D-fucose.

6. STUDIES IN ORGANIC SYNTHESIS AND INDUSTRIAL ORGANIC CHEMICALS

6.1 *Intermediates*

6.1.1 *p-Nitroacetophenone :* (SP-57/70)

p-Nitroacetophenone is the starting material for the manufacture of chloramphenicol. *p*-Nitroacetophenone is at present imported and it is estimated that its requirement by 1973-74 would be around 250 TPA, valued at Rs. 65 lakhs.

Starting from ethylbenzene a two-step process involving nitration and oxidation has been worked out on 0.5 kg./batch scale of the finished product. The first step has been standardized on 4 kg./batch scale. The recovered *p*-nitroethylbenzene from the first step has been successfully reused in the oxidation step. A final report has been submitted to the sponsor and the project successfully completed.

6.1.2 *p-Phenetidine :* (SP-66/70)

A scheme on the preparation of *p*-phenetidine, an important intermediate for the manufacture of phenacetin- a pharmaceutical, was undertaken on sponsored basis. Several alternative routes for its preparation were studied and a commercially attractive process has been optimized and demonstrated to the sponsor. The project is concluded.

6.1.3 *Technical preparation of organic intermediates :* (PP-6/69)

This multi-purpose pilot plant project for scale up of batch type processes developed in the laboratory is continued.

Pilot plant runs of processes on catechol and tert-butyl catechol have been taken. These processes have been released to industry.

Process for the preparation of IP grade benzoic acid from crude methyl benzoate has been standardized. The process has been released to IPCL, Baroda.

Work on monochloroacetic acid and trichloroacetic acid has been concluded. The process has been released to industry.

Pilot plant batches on *p*-nitroacetophenone have been carried out.

Besides above, work on chlorinated paraffin wax; *N*-dimethylacetamide; dichloropropionic acid; and antidiabetic biguanide has also been carried out.

6.1.4 *Monochloroacetic acid (MCA) and Trichloroacetic acid (TCA) :* (ATT-154/70)

Monochloroacetic acid and trichloroacetic acid are used in the manufacture of weedicides and herbicides. Other requirements for MCA are in the manufacture of carboxymethyl cellulose (CMC) and for intermediates such as, ethyl chloroacetate, glycine, thioglycollic acid, EDTA, etc.

An economic process for the preparation of MCA and TCA by direct chlorination of acetic acid in presence of a suitable catalyst has been developed and optimized. The process has been released to industry and the project is concluded.

6.1.5 *2,2,4-Trimethyl-6-ethoxy-1,2-dihydroquinoline :* (ATT-162/70)

This compound is used as an antioxidant and flex-cracking inhibitor in rubber industry. It is estimated that the future demand of this product may be around 500 TPA.

A process for the preparation of this compound has been developed on 1 kg./batch scale and offered to industry. The process consists of condensing *p*-phenetidine with acetone in presence of a catalyst.

6.1.6 *Thioglycollic acid (Mercaptoacetic acid) :* (ATT-164/71)

Thioglycollic acid is used in cosmetics in hair fixation preparations and as an essential additive in the technical preparation of bisphenol-A. The annual requirement of this chemical is estimated at about 60 TPA.

An economical process for the synthesis of this chemical has been standardized on 1 kg./batch scale, and the know-how is being offered to industry. The project is now concluded.

6.1.7 *m-Phenylenediamine :* (ATT-180/71)

m-Phenylenediamine is an intermediate used as a coupling component in the synthesis of direct dyes. Its import during 1969-70 was 58 tonnes valued at Rs. 6 lakhs. Estimated demand for the intermediate by the end of the 4th Five Year Plan is around 100 TPA.

A process for the production of *m*-phenylenediamine has been standardized on 1 kg./batch scale (yield- 92%). The product obtained conforms to the specifications for the commercial product.

6.1.8 *Bisphenol-A :* (ATT-184/71)

Bisphenol-A is an important intermediate for making epoxy resins. Estimated demand by the end of the 4th plan is about 1000 TPA.

A process has been standardized for the preparation of Bisphenol-A in 90% yield starting from phenol and acetone. The product obtained meets the specifications of the technical grade product for epoxy resins. The project is concluded and the process will be offered to industry after a few large scale runs.

6.1.9 *A new synthesis of chloropheniraminemaleate*

2-*p*-Chlorobenzylpyridine is one of the important intermediates for the synethesis of chloropheniraminemaleate.

Attempts to synthesize 2-benzylpyridine, by the action of phenylmagnesium bromide on 2-chloromethylpyridine and 2-pyridinemethanol tosylate ed to a mixture containing 2-benzyl pyridine and bi-picolyl. 2-Benzylpyridine was selected as a model compound for these studies.

6.1.10 *Utilization of o-nitroethylbenzene*

Work on this project was undertaken with a view to find out a suitable commercial use of *o-nitroethylbenzene which is obtained as bye product in* the manufacture of *p*-nitroacetophenone.

During the attempts to use *o*-nitroethylbenzene for conversion into Indigo, the action of aqueous alkali on *o*-nitroethylbenzene was studied. The following products were isolated : (1) *o*-aminoethylbenzene, (2) *o*-aminoacetophenone, (3) 2-(*o*-aminophenyl) quinoline-4-carboxylic acid and (4) 2-(*o*-aminophenyl)-4-methylquinoline An intermolecular oxidation reduction is involved in this reaction.

Several experiments were conducted to obtain a better yield of *o*-aminoacetophenone. The maximum was 15% and it was found that longer time and high temperature converted it into bimolecular condensation product

Work on the project has been concluded.

6.2 *Dyes and pigments*

6.2.1 *Quinacridone pigments :* (SP-49/69)

Work on the preparation of quinacridone pigments which are characterized by their high all-round fastness properties comparable to phthalocyanines, was conducted on a sponsored basis earlier. Since the termination of sponsorship, further developmental work on this project was continued as NCL project.

A method for the preparation of linear trans-quinacridone red has been developed and standardized on 1 kg./batch scale. The crude quinacridone so obtained has been converted into pigmentary form which is slightly weaker in strength than the imported quinacridone red. Work on the project has been concluded

6.2.2 *Cationic dyes for acrylic fibres :* (SP-55/70)

Cationic dyes are not manufactured in India at present. A process for the preparation of blue cationic dye standardized earlier in NCL is being scaled up on a pilot plant scale by the sponsor. Work is being continued for the preparation of other cationic dyes.

6.2.3 *Carbazole Dioxazine Violet :* (ATT-139/69)

This pigment gives a characteristic bright violet shade with excellent fastness properties comparable to that of phthalocyanine pigments. Such pigments are not manufactured in India and the requirement is met through imports. The fourth plan target for the pigment is estimated at ca 12 TPA.

Starting from technical grade carbazole of 95-97% purity, a process for the synthesis of Carbazole Dioxazine Violet pigment base, was standardized on 1 kg./batch of the product, earlier.

Conversion of the above base into Pigment Violet RL has been undertaken. A pigmentary form has been obtained which compares well with the commercial pigment. The process has been offered to industry and the project is now concluded

6.2.4 *Sonobuoy fluorescent dye marker :* (ATT-147/70)

A suitable substitute for the imported dye marker composition was developed earlier and a 10 kg. sample was sent to the Naval Physical and Oceanographic Laboratory, Cochin, for trials.

The product has been tested and found suitable in the laboratory tests as well as in trials in the sea.

6.2.5 *Chlorinated Copper Phthalocyanine :* (ATT-155/70)

This pigment possesses excellent tinctorial and fastness properties and is widely used in paints, plastics and printing inks. After the termination of sponsored scheme, further developmental work on the technical preparation of Chlorinated Copper Phthalocyanine has been undertaken. An economically attractive process has been standardized on a laboratory scale and a patent has been applied for. Work on the project has been concluded.

6.2.6 *New synthetic dyes and pigments :* (ATT-157/70)

Disperse dyes containing sulphone morpholide group were prepared and evaluated earlier. The dyes showed good sublimation fastness properties on synthetic fibres but their tinctorial properties were not satisfactory. Several similar dyes, but containing insulating groups like a methylene group inserted in between the chromophore and the sulphonyl morpholide groups have been prepared. These dyes showed good sublimation fastness and affinity for polyester fibres. The tinctorial properties of these dyes were satisfactory for polyester and cellulose acetate fibres.

A new pyrocoline derivative, 1-(*p*-nitrophenyl)-2, 3-phthaloylpyrocoline was prepared, which gave brilliant red dyeings on polyester with good sublimation fastness but fastness to light was poor. The corresponding *p*-amino derivative gave violet dyeings on polyester.

Three new pigments having 2,3-phthalozlpyrocoline ring system were prepared and evaluated

Greenish coloured sulphur and sulphur-nitrogen heterocylic compounds from anthraquinone derivatives have been prepared and characterized. Their properties are being evaluated.

6.2.7 *Studies in synthetic dyes :* (B-8 8/62)

The Marschalk reaction : This reaction consists of *C*-alkylation of hydroxy and aminoanthraquinones by treatment with aqueous sodium dithionite, sodium hydroxide and an aldehyde. The mechanism of the reaction has been studied. Using aromatic aldehydes and α-aminoanthraquinone, new compounds have been prepared, whose dyeing properties are under study.

Polycyclic quinones : In two recent papers (*Ind J. Chem.* 1971,**9**,921) the mass spectra and NMR spectra of the methyl ethers of the leuco compounds of violanthrones have been discussed in relation to the structures of dyes such as CI Vat Blue 16 and CI Vat Green 2.

Arylanthraquinones : The structures for the nuclear arylation product obtained from CI Acid Blue 45 (4, 8-diamino-1, 5-dihydroxyanthraquinone-2,6-disulphonic acid) and of 4, 8-dinitro-1, 5-dihydroxyanthraquionone, described in an old IG and recent Bayer patents, have been revised (*Ind. J. Chem.* 1971, **9**, 1060). The mechanisms of the reactions are under investigation.

Sulphur dyes : The structure suggested earlier for Cibanone Orange R has been revised, but it will be desirable to confirm it by X-ray crystallography. Spectral data have also led to a new tentative structure for Cibanone Yellow R. Exploratory work on the structures of some sulphur dyes is in progress.

6.3 *Drugs*

6.3.1 *Glyceryl-α-mono-para-aminobenzoate :* (SP-56/70)

This chemical is used in the manufacture of certain cosmetic products. Trial batches have been conducted on 1 kg./batch scale, using commercial grade raw materials and the product obtained has been sent to the sponsor for evaluation.

Work on the preparation of the above intermediate will be continued.

6.3.2 *Propoxyphene hydrochloride :* (SP-73/71)

This is a drug known for its analgesic activity. Its analgesic activity mainly resides in its dextro-isomer. It has the same analgesic potency as that of codeine; but has no addictive action. It can be usefully administered to patients with chronic pain (rheumatoid arthrities and migraine). Based on preliminary laboratory work, a six-step process for the preparation of propoxyphene hydrochloride on 0.5 kg. scale was worked out as required by the sponsor. After preparation of pure dextropropoxyphene, the work will be concluded.

6.3.3 *Chlorohydroxyquinoline* (*Chloroiodoquin*) *:* (SP-75/71)

Chloroiodoquin is used as an amoebicide. 4-Chloro-2-nitrophenol, one of the compounds obtained during the synthesis of chloroiodoquin, is used as germicide.

A four-step process for the production of 5-chloro-8-hydroxy quinoline has been standardized on the laboratory scale, starting from *p*-dichlorobenzene. Further scale up work is in progress.

6.3.4 *Synthesis of potential pharmacologically active substances :* (SP-77/71)

The objective of the project is to synthesize new compounds which may posses better pharmacological activity.

Earlier, work on the synthesis of heterocyclic compounds as potential analgesic and antiviral compounds was conducted. Ten new compounds were synthesized and some of them were sent for testing.

Syntheses of compounds with potential anti-inflamatory activity have been undertaken and further work is in progress.

6.3.5 *Vitamin* B_6 *:* (ATT-16/66)

Vitmain B_6 is not manufactured in India. The landed cost of imported Vitamin B_6 would average at Rs. 245/kg. Import of this item for the year ending March 1971 was 2750 kg. at an average landed cost of Rs. 6.74 lakhs.

A four-step synthesis of Vitamin B_6 (starting from paraldehyde) on a laboratory scale has been completed. Cost of Vitamin B_6 by this route has been tentatively estimated as Rs. 425/kg. For a 10 TPA plant the estimated capital cost has been worked out to Rs. 32.25 lakhs.

The process is being released to industry through NRDC and scale-up trials are at various stages of progress.

6.3.6 *N, N-Dimethylbiguanide HCl and Phenethylbiguanide HCl:* (ATT-75 68

These compounds are valuable anti-diabetic drugs and are at present being imported under the trade names Metformin (DMBG-HCl) and

Phenformin (PEBG-HCl). The imports during 1969-70 for these drugs were of the order of 670 and 900 kg. valued at Rs. 0.9 and 1.23 lakhs. The requirements for these products will be more if they are freely available in the country.

A commercially feasible process for production of these drugs has been developed and offered to industry for commercial exploitation

6.3.7 *Vitamin D_3 :* (ATT-140/69)

Vitamin D_3 is used in the pharmaceutical and food industry since it has anti-rachitic activity. Imports of Vitamin D (D_2 and D_3) during 1970-71 were of the order of Rs. 2.03 lakhs. It is estimated that the total demand for Vitamin D_2 and D_3 is worth Rs. 10 lakhs/annum.

Cholesterol has been converted to 7-dehydrocholesterol in satisfactory yields. A number of experiments have been carried out to find optimum conditions for the photochemical conversion of 7-dehydrocholesterol (20g. per batch) to vitamin D_3, and it has been possible to obtain certain fractions which, though not crystalline, are rich in Vitamin D_3. Work on this project has been concluded.

6.3.8 *Camphene to camphor :* (ATT-143/69)

Work has been undertaken to simplify the currently used 3-step route for preparation of camphor from camphene obtained by isomerization of α-pinene, a constituent of turpentine oil. In this connection, a single-step direct oxidation of camphene to camphor using $Na_2Cr_2O_7$-aq.H_2SO_4 has been investigated in detail. The method has finally been standardized for a 0.5 kg. batch size of camphene to yield 80-82% of camphor (purity 90%). On economic considerations a 10% increase on the above yield appeared desirable but this could not be realized by the usual isolation procedure (steam distillation). The targetted yield has been however realized in small scale experiment (50 g.) when a different isolation procedure *viz.* solvent extraction was employed. However, this was not pursued further since this method appeared economically unattractive. The project is concluded.

6.3.9 *Potassium guaiacol sulphonate :* (ATT-183/71)

This drug is used as expectorant in many cough remedies and is not made in this country. The preparation of guaiacol from *o*-anisidine was standardized earlier. Several sulfonation experiments were carried out on guaiacol and a product conforming to nearly all the specifications (National Formulary, XI, p. 291) were obtained. Attempts to improve the product to pass all the tests are being made.

6.3.10 *Folic acid :* (ATT-191/71)

Folic acid is used in medicine and nutrition, and there is a substantial demand for this product in India.

Folic acid molecule is composed of two main fragments (i) pteridine part and (ii) *p*-aminobenzoyl glutamic acid and the success of the synthesis of folic acid mainly depends upon practical synthesis of these two fragments and their condensation. Work has been undertaken to synthesize these two fragments separately.

Synthesis of pteridine fragment : Initial exploratory work has been carried out to utilize 2,4-diamino-5-nitroso-6-oxo-pyrimidine (the intermediate obtained from IDPL) for its conversion into 2-amino-4-hydroxy-6-aldehydropteridine. Along with this the established route for the synthesis of folic acid is being standardized.

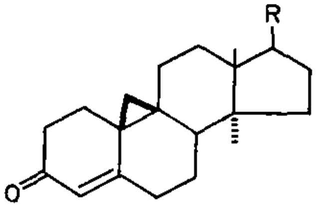
Synthesis of folic acid (*Pteroyl glutamic acid*) *:* The condensation of *p*-aminobenzoyl glutamic acid with 2-acetamido-6-formyl pteridine is the key step in the synthesis of folic acid. The work has been undertaken with a view to improve the yield of the condensation either by (1) increasing the reactivity of glutamic acid part or (2) by condensing these components in solid phase. Both the approaches are under investigation.

6.3.11 *Utilization of the bye products of opium alkaloid industry :* (AB-21/67 (

Opium marc- a waste product of the Indian opium alkaloid industry contains considerable quantities of cyclolaudenol and cycloartenol. Possible utilization of these compounds will aid the economics of opium alkaloid manufacture.

The objective of this project is to prepare the hitherto unknown 9, 19-cyclosteroid hormones (I) and (II) from the triterpenes cycloartenol and cyclolaudenol and to evaluate their biological importance.

The triterpenes are now readily accessible from the non-saponifiable fraction of opium marc.



(I) R = $-COCH_3$
(II) R = =O

An efficient sequence of reactions has been developed for degradation of the triterpene side chain into either C-17 methyl ketone or ketone. Conversion of 3-β-hydoxy-4,4-gemdimethyl system of ring A into 3-oxo-4-ene system has also been completed. Though the operations involved in the latter transformation were subsequently simplified and yields improved in certain steps, the sequence is rather long and hence a new shorter approach to ring A modification is underway.

6.3.12 *Synthesis of compounds with potential biological activity :* (B-8.31/63)

Tetra and pentacyclic phenothiaphosphine oxide derivatives have been synthesized. The synthesis of dihydrothiazole (3, 2-a) quinoline and dihydrothiazole (2, 3-a) isoquinoline has now been accomplished, starting from quinoline and isoquinoline *N*-oxides respectively. A number of compounds in these series are being tested for their biological activity.

6.4 *Fine chemicals*

6.4.1 *Fine Chemicals Project :* (PP-7/64)

For the third year in succession the Fine Chemicals Project has shown profits. Comparative production, and sales figures(in lakhs of rupees) for the past three years are tabulated below :

Year	*Production*	*Sales*	*Profit*
1969-70	2.35	2.71	0.22
1970-71	2.07	2.20	0.23
1971-72	2.05	2.17	0.39

6.4.2 *Polyesters as stationary phases in GLC analysis :* (AB-33/63)

With the growing tendency of using GLC techniques in different fields the demand for cloumn filling material (solid support) and liquid phases (substrates) will be considerably increased. All the materials are, so far, being imported. This is a long-term project, exploratory type and development work is continued along with routine analyses of samples.

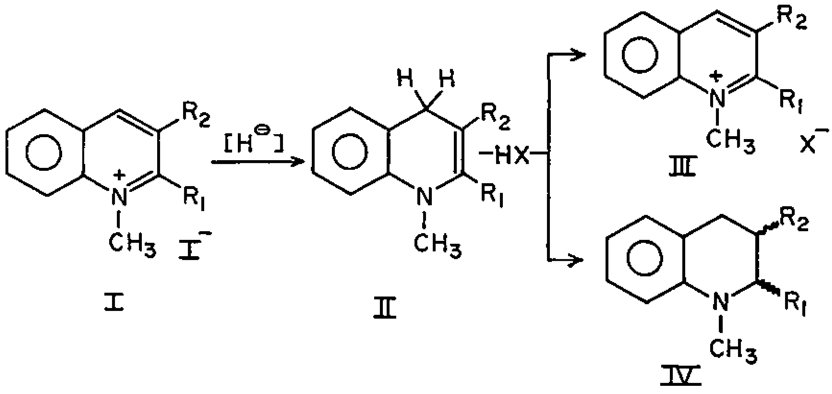
Studies on fire-brick support undertaken earlier has been completed.

Most of the commonly available stationary phases are not very effective for separation of substituted aromatic isomers. In order to get practical separation of such compounds, several new polyesters containing aromatic and substituted aromatic nuclei have been synthesized and tested as liquid substrates. Some of them are found suitable for separation of isomeric phenols, cresols, alcohols, aldehydes and esters; while others render effective separation of nitro-, nitro-halogeno-aromatic isomers. Partial separation of toluidines is also obtained. Further improvements are in progress.

6.5 *Studies in organic synthesis and reactions*

6.5.1 *Studies in heterocyclic chemistry :* (B-8.7/65)

Studies in hydride transfer : Sodium amalgam has been found to be a reagent of choice for effecting the reduction of various 2,3-disubstituted quinolinium methiodides (I) to 1,4-dihydroquinoline derivatives(II). These compounds on acid catalysed disproportionation yield 2,3-disubstituted quinolinium salts (III) and 2,3-disubstituted-1,2,3,4-tetrahydroquinoline derivatives (IV). The steric preferences in the formation of the latter have been investigated.



6.5.2 *Nitrogen heterocyclics :* (B-8.50/69)

The finer details of the rearrangement observed during the cyclodehydration of *cis*-2-arylaminomethylenecyclohexanones have been studied further. A few more derivaties of α- and β-quinidine derivatives have been prepared. These compounds represent non-alternant,non-benzenoid 14 π-electron systems.

6.5.3 *Reactions of α-sulphonyl carbanions :* (B-8.56/71)

Reactions of vinylogous α-halosulphonyl compounds with base were carried out in order to investigate the mode of reaction in analogy with the Ramberg-Backlund rearrangement for α-halosulphones. The model compounds *cis*-(I) *trans*-3-bromo-2-pentenylbenzyl sulphone(II)have been synthesized. The major products obtained in both the cases are *cis*- and *trans*-1-phenyl-2-methyl-1, 3-pentadienes. The *cis*-compound (I) affords a *cis*-diene (λ_{max}262 mμ; log ε 4.3) while the *trans*-compound (II) gives the *trans*-diene (λ_{max}267 mμ; log ε 4.4). In addition to the diene a compound containing sulphonyl group is also obtained from (I) and is probably a cyclic sulfone.

The mechanism of these base-catalyzed reactions involves an intramolecular allylic nucleophilic displacement (SN_2 type) with subsequent expulsion of SO_2 from an intermediate thiirane dioxide to yield the pentadiene. Further work is in progress to elucidate the stereochemistry of the reaction.

6.5.4 *Functionalization of saturated aliphatic and alicyclic compounds:* (B-8.46/66)

Work on the functionalization of saturated hydrocarbons (*p*-menthane and methylcyclohexane) is of considerable theoretical and practical interest especially for the synthesis of alcohols, ketones and acids. It involves irradiation of the hydrocarbon in ethylacetate in the presence of a peracid.

p-Menthane : After hydrolysis and oxidation of the reaction product GLC analysis showed the presence of six compounds. Separation of these by column chromatography on alumina was carried out. The pure compounds thus isolated were characterized as menthone, isocarvomenthone, dihydroterpineol-4 (trans) and dihydroterpineol-1.

Methylcyclohexane : The reaction product was subjected to hydrolysis and oxidation. Upon GLC analysis it was found to contain four compounds which were identified as 1,1-methylcyclohexanol, 2-methylcyclohexanone, 3-methylcyclohexanone and 4-methylcyclohexanone.

Assignment of stereochemistry for the three secondary alcohols obtained above (before oxidation) is underway.

7. BIOCHEMICAL STUDIES

7.1 *Applied microbiology*

7.1.1 *National collection of industrial microorganisms :* (AB-62/68)

The national collection of industrial microorganism (NCIM) maintained in the NCL contains about 1400 non-pathogenic yeasts, bacteria and fungi. Cultures are supplied free of charge to research institutions and industries. Isolation and identification of cultures for screening programmes for specific applied projects, research on preservation of cultures and routine subculturing of organisms and biochemical tests on important cultures have been continued during the year. Two hundred and fifty six new cultures received from abroad are being subcultured and tested before addition to the collection. Seven hundred and eighty four cultures were despatched to different institutions in the country.

7.1.2 *Industrial enzymes*

The project includes at present screening for suitable organisms for the production of industrially important enzymes such as alkali stable protease, acid stable amylase, cellulase and for 2-keto-L-gulonic acid which is the key intermediate for the manufacture of vitamin C.

Alkali stable protease : (AB-67.4/70)

Addition of small quantity of alkali stable protease in domestic washing powders and detergents is being extensively practised in advanced countries. These enzymes which are stable in presence of the different constituents of washing powders and detergents enhance considerably their washing power particularly against protein strains.

A microbial strain was isolated which was found to produce alkali stable protease. The stability of the enzyme in the presence of tripolyphosphate and perborate detergents etc., was found to be comparable to similar commercial preparations. At room temperature the enzyme remained stable at pH 8.5 to 10.5 for 48 hours. Attempts to increase the activity of the enzyme are underway.

Acid stable amylase : (AB-67.1/68)

The idea of incorporating acid stable amylase into pharmaceutical digestive compositions is to enhance the activity of the enzyme at the prevailing pH of stomach viz. 1.8 to 2.4.

Seven strains from among those investigated earlier (*Aspergilli*) were chosen for a detailed examination, especially resistance and stability towards acidic conditions. All of them were found to produce extracellular amylase which showed varying degrees of acid stability i.e. stability to incubation at pH 2.5 and 37° for half an hour. This stability could be further increased by the use of stabilizing agents like calcium chloride, starch, etc. A few among the seven strains exhibited amylase activity at a pH 2.5 but at much reduced levels as compared to their activity at pH 6.9. For comparison commercially available indigenous and imported amylase enzymes were obtained. It was observed that under the conditions employed in the laboratory tests, all commercial samples lost their activity on incubation at pH 2.5.

Work to obtain mutant strains of these organisms with enhanced resistance to acid pH as well as with increased activity is continued.

Cellulase : (AB-67.2/68)

Studies on the isolation and screening of microorganisms for obtaining high cellulase yielding strains were continued. Thirty new isolates of fungi growing at 30° and one thermophilic strain belonging to *Humicola* species growing at 50° were added to the collection. Out of the 120 fungal isolates so far obtained, three gave appreciably high cellulolytic activity both for C_n and C_1 enzymes in shake flasks.

Studies regarding influence of yeast extract, pH conditions, cellulase concentrations in the growth media etc., has made it possible to increase the yields in certain strains from 50 to 150 'Cx' units. Growth of *Sclerotium* species on semi-solid media like wheat bran and saw dust moistened with nutrient solutions also yielded high enzyme activity. Twenty two mutants have been obtained by UV irradiation and chemical mutagens from selected isolates. None of the mutants (survivors) got so far has given higher cellulase activity than the parent strain but the period of fermentation required for maximum activity has been reduced by a week in some mutants.

7.1.3 *Vitamin C :* (AB-67.3/68)

The technical know-how for the production of ascorbic acid from sorbitol has been worked out in this laboratory and is being taken up for commercial exploitations by Hindustan Antibiotics Ltd., Pimpri.

The possibility of improving the process by eliminating one or more of the steps is being explored by screening for suitable microorganisms that can convert sorbitol or sorbose directly to 2-keto-L-gulonic acid, which is the immediate precurssor of ascorbic acid.

Screening for suitable microorganism is in progress.

7.1.4 *Oxidation of ammonia in soils* : (AB-67.5/70)

Work on organisms which convert ammonia to nitrite and nitrate in soil and on a screening for suitable compounds which retard this oxidation has been undertaken. This group of organisms is difficult to maintain in subcultures and has not been extensively investigated inspite of their great economic importance with reference to the problem of loss of nitrogenous fertilizers by nitrification by soil microorganisms

The isolation of different strains of *Nitrosomonas*, *Nitrococcus*, etc., and study of their nutrition and growth conditions has been undertaken. Six strains of *Nitrosomonas* were isolated from irrigated and other soils from different agricultural stations and their growth and inhibition are under investigations.

7.2 *Plant and animal tissue cultures :* (AB-96/72)

During the last five years viable tissue cultures of maize, wheat, rice and sorghum have been established and a considerable amount of information has been obtained regarding their nutritional and other growth requirements.

Studies on the nutrition and differentiation of the above four cereal cultures established in this laboratory were continued. It was established by repeated subculture on media containing different vitamins that thiamine and pyridoxine are the only vitamins required by maize and wheat. Abscisic acid

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was found to have a selective effect in inhibiting the growth of roots but not of callus tissue of wheat and maize. This is the first report of a selective effect of this plant hormone in inhibiting root growth.

Whole plants were obtained from cultures of wheat even after twelve subscultures. Whole plants were also obtained from callus tissue of hybrid cabbage and sugarcane explants. Work on determining of optimum conditions for raising plantlets is in progress.

Facilities for animal tissue culture work were established and two animal cell lines (He La and Vero) are being maintained. Preliminary work on insect tissue culture for the study of insect hormones was undertaken.

7.3 *Enzyme chemistry and technology*

The study of enzymes is closely related to the study of intermediary metabolism. Work on this project deals with the isolation, chemistry, regulatory properties and biochemical role of various enzymes.

7.3.1 *Hexokinase :* (B-12.1/68)

Animal tissue hexokinases have been studied only to a limited extent owing to their instability or insolubility.

The particulate hexokinase of ox heart was solubilized by a new procedure and obtained in highly purified form for the first time in this laboratory and its properties and kinetics were studied. The partial purification of two isoenzymes from the soluble fraction of heart muscle was also carried out.

The project has been completed.

7.3.2 *Phytase :* (B-12.2/64)

A bacterial phytase which is specific for inositol hexaphosphates was obtained for the first time in this laboratory. It was found to be homogenous in the ultracentrifuge. Its sedimentation coefficient is 3.5 S and its molecular weight 35,000.On acrylamide gel electrophoresis it showed two bands both of which had enzyme activity. The study of its kinetic properties and its use for the estimation of inositol hexaphosphates is in progress.

7.3.3 *DPNase :* (B-12.4/68)

The DPNase of ox brain was solubilized and purified and its K_m and V_m at different pHs and the effect of different inhibitors on the enzyme over a wide pH range were determined. There was no significant change of K_m or K_I with pH.

7.3.4 *Acylphosphatase* : (B-12.7/70)

Acylphosphatase of plant tissues has not been studied hitherto. The enzyme from *Vigna catjang* has been shown to be a true acylphosphatase which does not act on other phosphate esters or on ATP etc. The enzyme was obtained in homogenous form and found to be an unusually small protein with a sedimentation coefficient of 1.15 S and molecular weight of 6000. It showed a single band on acrylamide gel electrophoresis.

7.3.5 *Enzyme inhibitors* : (B-12.7 1/70)

During the course of the purification of acetyl phosphatase a crystalline protein with trypsin inhibitor activity was obtained. On recrystallization the activity was, however, found in the supernatant liquid. DEAE chromatography gave homogeneous preparation. The ratio of its activities with trypsin and chymotrypsin was 5. It is stable to boiling at pH 3 to 7 and its molecular weight is 15,000.

7.3.6 *Bacterial degradation of (–) citramalate* : (B-12.8.1/68)

The metabolic pathway of degradation of (–)citramalate was established using a *Pseudomonas* grown on citraconate or (–)citramalate. (–)Citramalate was converted to citramalyl-CoA by the action of succinate-citramalyl CoA transferase. Citramalyl-CoA was cleaved to pyruvate and acetyl CoA by a second enzyme. Both the enzymes were specific for (–) citramalate and had no action on (+) citramalate derivatives. By the use of C^{14} labelled pyruvate it was conclusively established that the product of the condensation of pyruvate and acetyl CoA is (–) citramalyl CoA. The two enzymes and succinate activating enzyme were separated and partially purified. It was also established that the product of the reaction is citramalyl-CoA and not citramalate and CoA. The equilibrium for the cleavage of citramalyl-CoA was found to be approximately 2.0 M. at pH 7.4 at 30°.

7.3.7 *D-Malate dehydrogenase* : (B-12.8.2/68)

A specific D-malate dehydrogenase, which requires K^+ and Mg^{2+} for its activity, was partially purified. Several isoenzymes were found to be present on acrylamide gel electrophoresis, but they were shown to be artefacts formed during the exposure of the enzyme to pH 4 during purification.

Both the above projects have been terminated.

7.3.8. *DFP-susceptible enzyme and antidote* : (AB-65/68)

The problem of toxicity due to organo-phosphorus insecticide residues is closely related to the inhibition of several carboxyl esterases in the animal system. Diisopropyl phosphorofluoridate (DFP) and diethyl *p*-nitrophenyl phosphate (paraoxon) are the most well known prototypes of organo-phosphorous esters, which have been employed in this project for study of their influence on the Na/K ratio of animal tissues.

DFP and paraoxon when injected to mice at approximately one LD50, effect a decrease in the intra-cellular Na content of the liver. The K is unaffected. The same effect could be demonstrated *in vitro* by incubating liver slices in isotonic media containing 10^{-5} M organophosphates. The effects were shown to be unrelated to inhibition of acetylcholinesterase as eserine, a reversible cholinesterase inhibitor had no diminishing influence on the Na. Likewise, acetylcholine, butyrylcholine with or without eserine and ouabain had no effect.

Hitherto unrecorded findings mentioned above may have a bearing on the chronic toxicity due to organo-phosphorus insecticides. The project is now concluded.

7.3.9 *Citrate oxaloacetate-lyase* (*citrase*) : (B-12.6/65)

The project comprises of the studies on the structure-function relationships of the enzyme from *Aerobactor aerogenes*.

Studies on the detection and estimation of the N-terminal amino acid of the enzyme were carried using the dansyl chloride procedure. Methionine was identified as the only N-terminal amino acid and quantitative estimation by fluorescence spectrophotometry showed 1-N-terminal residue per subunit.

A new enzymatic method was developed for the preparation of specifically labelled citrate from fumaric acid-2, 3-C14. Conditions were standardized for the covalent tagging with eight C14-labelled residues per mole enzyme by sodium borohydride reduction of the *A. aerogenes* enzyme inhibited by oxaloacetate produced in the cleavage of the C14-labelled citrate.

Chloromalates, which were synthesized as possible active site directed irreversible inhibitors, were found to have no effect on the enzyme.

The enzyme from *A. aerogenes* has been obtained free from magnesium in its native state of aggregation. In presence of divalent metal ions, the enzyme has been shown to bind 1 g. atom of metal per subunit.

The pure enzyme isolated from a streptococcal source has been shown not to come under inhibition by oxaloacetate.

7.3.10 *Metabolism of nitrate by A. fischeri* : (B-12.6/65)

The activity of denitrifying bacteria in soil accounts for substantial losses of nitrate containing fertilizers. The project aims at biochemical studies with isolated enzymes which may help understand the basic mechanisms involved in the denitrifications.

Studies on nitrate metabolism in *A. fischeri* were continued. The formation of both nitrate and nitrite reductases was inhibited by oxygen. Nitrate and nitrite reductions by the growing cells occurred only when the respiratory enzymes were not saturated with oxygen. Nitrite reduction also did not occur so long as there was nitrate in the growth medium. Nitrate metabolism in *A. fischeri* was found unique in that the product of nitrate metabolism was ammonia but it resembled the respiratory type in that the metabloism of nitrate and the formation of nitrate and nitrite reductases was proloundly inhibited by oxygen.

Ultracentrifugal studies were carried out to investigate the subunit nature of nitrite reductase. The enzyme in urea system sedimented as a single symmetrical peak. The molecular weight of urea-treated enzyme was found to be the same as that of untreated enzyme. The end group analysis of nitrite reductase indicated that methionine is the amino terminal residue. A value of one amino terminal residue per mole of enzyme, as obtained by quantitative experiment with dansyl chloride, indicated that nitrite reductase consists of a single polypeptide chain.

7.3.11 *Enzyme technology : Matrix-bound enzymes :* (AB-97/72)

Insolubilized enzymes obtained by covalently binding the protein to solid support materials hold promise as elegant industrial catalysts, since they can be manipulated easily, removed readily from the reaction solution and reused repeatedly in relatively large amounts of substrate.

The primary objective of this project is to develop novel insolubilized enzyme systems which can profitably replace existing water-soluble enzymes as industrial catalysts. Preliminary studies on matrix-bound penicillin acylase systems for 6-amino-penicillanic acid production have been initiated in collaboration with the Research Division of Hindustan Antibiotics Ltd., Pimpri.

8. POLYMERS, RESINS AND ELASTOMERS

8.1 *Polymers*

8.1.1 *Expandable polystyrene :* (ATT-31/66)

Preparation of expandable polystyrene beads involves suspension polymerization technique. Polystyrene foam is presently manufactured in the country using the imported know-how. Attempts are underway to establish indigenous know-how in this field.

Efforts to obtain polystyrene beads having desired bulk density and foaming characteristics were continued

With the use of a new suspending agent and a change in stirrer design, it was possible to obtain 90% of beads of the proper characteristics. The thermal insulation and compression strength of the sheets made from these beads were found comparable to similar products in the market.

The technique of blowing expandable beads into foam sheets will be standardized and scaled up for easy transfer to industry

8.1.2 *Polymers for oil well drilling :* (ATT-115/68)

Oil and Natural Gas Commission (ONGC) has shown interest in developing a polymeric product which can be used as a substitute for an imported product known as ' Halad '. The product was identified as hydroxyethylcellulose, with some sodium sulphate content.

Efforts were continued to prepare a completely water soluble hydroxyethylcellulose with high ethoxy content (1.8 to 2 0) and containing 15-18% of sodium sulphate.

Test samples prepared by ethoxylation of soda cellulose followed by neutralization of excess alkali contained 40-45% of sodium sulphate. This was reduced by leaching with water.

The samples with reduced sulphate content (15-20%) and ethoxy content (1.5) were tested by M/s. Oil India, which indicated that our product has low degree of polymerization (DP) and should further be ground to 200 mesh to make it readily water soluble.

8.1.3 *Can lining composition :* (ATT-131/69)

Lining material based on synthetic rubber latex have better properties of adhesion, storage stability, film formation as compared to compositions based on natural rubber latex These compositions are chiefly meant for use in metal can manufacture suitable for storing mineral oils, greases, food, etc. Imports of these lining compositions are around 100 TPA valued at Rs. 10 lakhs.

Based on evaluation reports received from metal can manufacturers, the process and the product were modified and later standardized on 5 kg./batch scale.

The know-how is now available to industry.

8.1.4 *Nylon*-12 : (ATT-188/72)

Nylon-12 is a latest addition to the polyamide family. It possesses high dimensional stability at extreme temperatures and under conditions of extreme stress, water repellancy, exceptional toughness, impact strength, chemical resistance, etc., over other known polyamides. These properties make it very useful in the manufacture of precision instrument parts, glass reinforced tough sheets, several kinds of extruded and moulded articles, for coating reaction kettles and for sheathing underground cables, etc

Preliminary experiments on the oligomerization of butadiene to 1,5, 9-cyclododecatriene have been carried out using diethyl aluminium bromide and titanium tetrachloride complex as catalyst. The cyclic triene has been identified by physico-chemical methods. Using the same complex as catalyst, 700-800 g. lot of the cyclic compound has been prepared. Attempts to prepare lauryl lactam from the cyclic compound are being made and reaction conditions are being established.

8.1.5 *Polyurethane rigid foams* : (AB-95/71)

There is a good demand for polyurethane based foams for insulation purposes and as a substitute for wood. At present this product is not manufactured in the country.

Polyester, based on shellac and CNSL was prepared and reacted with different isocyanates. Evaluation of these rigid foams is under study.

8.2 *Resins*

8.2.1 *Thermosetting resin for industrial laminates* : (SP-68/71)

Thermosetting resin which impart cold punching properties to the industrial laminates are not at present produced in the country. These laminates have special significance in electrical insulation industry and for electronic components used in T. V. These cold punching laminates are based on kraft and bleached kraft paper of higher water absorbancy and tensile strength and oil modified phenolic resins.

The technical preparation of two types of resins as desired by the sponsor was standardized on 3 kg./batch scale. The laminates made by using these resins were tested and compared with imported standard cold punching laminate showing a generally favourable comparision. Large scale preparation of both the resins was conducted and the resins tested by the sponsor at their factory. The laminates made by using these resins were found to compare well with the imported product.

8.2.2 *Polyurethane coating compositions for textiles and other substrates :* (ATT-46/74)

Polyurethane coatings on textiles posses excellent flexibility at low temperatures, water repellancy and water proofing properties. The coated fabrics are also light in weigh . Coating compositions suitable for coating nylon fabrics which are useful for high altitude tents, etc., and coatings for gumboots had been earlier developed and the process is being released to a firm.

Water proofing on cotton fabrics for rainwears using shellac based polyurethanes and polyurethane rubber for textile from adipic acid, glycols and isocyanates were prepared and found to be satisfactory. Polyurethane lacquers suitable for gumboots and rubberized fabric were processed on 40-45 kg./batch and large samples were sent to industry for evaluation. The product was evaluated by Central Leather Research Institute, Madras, and found acceptable to leather industry.

Large scale trials for preparation of coatings for textile rainwears and work on preparation of polyurethane lacquers for wood and metal substrates is in progress.

8.2.3 *Linseed oil emulsion paint :* (ATT-49/66)

Water thinnable emulsion paints are becoming increasingly popular because of their versatile properties. These paints are quick drying, odourless, non-inflamable, non-toxic, easy to clean, easy to apply and have good covering property. They can be used on wood, plaster, fibreboard and concrete surfaces.

Preparation of a low cost water thinnable emulsion paint based on polymerized linseed oil has been standardized on 30 lit. scale. Samples were evaluated by industry and found satisfactory for drying properties, water resistance and mild alkali resistance.

The process is available to industry.

8.2.4 *Resin bound microfilters :* (ATT-87/68)

The product in view is known to be used in advanced countries for wi e variety of filteration jobs in domestic practice and industry where efficient, clean and compact filteration is desired. Such microfilters are not at present used in the country and there are no known imports.

A large number of filter cartridges (3" dia. and 10") using different types of cellulose pulps and resin binders were prepared and tested for filteration of water, milk and organic solutions, etc. Performance of these cartridges was tested by an entrepreneur interested in the technology.

The process is now being offered to industry for commercial implementation.

8.2.5 *Wet strength resin for paper* : (ATT-185/71)

These resins impart wet strength to paper and are not manufactured in the country. They are used for making paper for napkin, table cloth, wrapping paper for food stuff, milk bottles, etc.

Resin samples based on epichlorohydrin, polyamine condensates were prepared on small scale. The samples submitted for testing to a local filter paper manufacturing unit showed encouraging results.

Large scale preparation of resin will be undertaken if some paper manufacturer shows interest in this project.

8.2.6 *Ketone resins* : (ATT-187/72)

Ketone resins are suitable for air drying lacquers with good adhesion to metal, wood and paper. They are used in printing inks and for paper coatings.

Starting from cyclohexanone and paraformaldehyde a resin with softening point of 105° to 115° and good solubility in alchol has been prepared. Standardization of the preparation and testing and evaluation of the final product is in progress.

8.3 *Elastomers*

8.3.1 *Nitrile rubber* : (ATT-52/67)

Nitrile rubber is a copolymer of butadiene and acrylonitrile. It is commonly available in 3 grades depending on the acrylonitrile content. Nitrile rubber finds applications in various rubber compositions, where oil resistant property is desired.

Nitrile rubber is not manufactured in the country and the present requirement (500 TPA valued Rs. 50 lakhs) is entirely met by imports. Out of the two major raw materials, butadiene is already being manufactured in the country while acrylonitrile will be shortly produced by IPCL, Baroda.

Bottle experiments for the preparation of medium grade nitrile rubber were undertaken wherein soap prepared from tallow fatty acids was used as an emulsifier. These studies were suspended as efforts were directed to use a standard commercial soap in place if tallow fatty acid soap.

Finally a process for preparing medium grade nitrile rubber in 70-75% yield, having Mooney viscosity around 50 has been standardized on 7 kg./batch of the finished product. The process has shown good reproducibility in regard to the rate of conversion, total conversion at the end of 8 hrs, bound nitrile content of the rubber and its Mooney viscosity. Coagulating conditions so as to get good size porous crumbs have been optimized. The minimum soap retained in the rubber is about 1.5 percent. Experiments on further reducing the soap content are in progress.

Preliminary experiments have also been carried out on the preparation of high nitrile content rubber.

8.3.2 *Sulphochlorinated polyolefin elastomers (SCPE)* : (ATT-90.1/68)

The estimated present demand of SCPE in the country is around 200 TPA valued at Rs. 50 lakhs. If available indigenously the requirement will be still more.

These rubbers possess high outdoor weatherability, heat resistance and chemical resistance in addition to toughness and abrasion resistance in mechanical wear and tear. Main applications of these rubbers are in chemically resistant lining for storage tanks and other plant equipment and protective coatings to different substrates such as fabrics, cables, gaskets, wood, shoe soles, etc.

A one-step modification of the previous process (Ind. Pat. Appln. No. 130254) was evolved and a product comparable to 'Hypalon-20' in mechanical and chemical properties was obtained. It has been found that the economics of the process some what depends on the solvent recovery.

Large scale trials are proposed for study of solvent recovery and evaluation of the product by industry.

8.3.3 *Chlorinated polyethylene (CPE)* : (ATT-90.2/71)

Chlorinated polyethylene is a valuable commercial polymer primarily used for improving the impact strength of rigid PVC. It is also used as a vulcanizable elastomer for cable covering and for ozone resistant rubber goods and as a flexible tough thermoplastic. These products are yet not manufactured in the country.

Experiments were performed on a small scale to chlorinate low density polyethylene in aqueous suspension at different temperatures. The chlorinated product has about 20% chlorine as against the required 40%.

Further experiments are in progress.

9. STUDIES IN POLYMER CHEMISTRY

9.1 *Stereospecific polymerization* : (B-13.1/60)

The mechanism of polymerization of methyl methacrylate by chromium acetylacetonate and aluminium triisobutyl catalyst was studied in presence of inhibitors like hydroquinone and triethylamine. The rate of polymerization dropped considerably but the molecular weight of polymer did not change, indicating that the catalyst system behaved by free radical mechanism. This was later confirmed by studying NMR spectra of polymers obtained either in presence or in absence of inhibitor. The polymers were found to contain only heteroblock structure. This clearly showed that aluminium triisobutyl in excess of complex of catalyst system, polymerized methyl methacrylate by a free radical mechanism.

Another catalyst system of vanadium tetrachloride and aluminium diethyl bromide has been taken up to study polymerization of different monomers in presence of CCl_4 medium, which may be able to preserve transition metal in the higher valency state.

Isoprene, methyl methacrylate and vinyl acetate were polymerized, with catalyst system. Polyisoprene obtained was partially soluble in solvent, indicating the formation of cyclic structure of polymer. Products obtained with methyl methacrylate and vinyl acetate were brown in colour and soluble in chloroform. Work on kinetic studies and characterization of the products obtained will be continued.

9.2 *Polymer characterization* : (B-14.14/69)

Thermodynamic aspects : In continuation of the work on calculation and compliation of thermodynamic properties on monomers and polymers, the new bond-energy scheme was extended to hydrocarbons and hydrocarbon-polymers. Two papers have been published. The third one on alicyclic hydrocarbons (non-aromatic rings) and ring-opening polymerization has been communicated. Further work on halocarbons, oxygen-nitrogen- and sulfur-compounds etc., is in progress.

Copolymerization : The monomer reactivities in copolymerization are important kinetic constants that control the copolymer composition, an aspect of importance from both theoretical and practical angle.

A new analytical solution for copolymer composition equation was evolved and published. The procedure has been extended to the integrated copolymer equation and a computer program drafted with a view to computing the reactivities of monomers with high accuracy.

10. CHEMICAL ENGINEERING AND PROCESS DEVELOPMENT

10.1 *Studies in chemical engineering*

10.1.1 *Reaction models and reactor design :* (B-14.8/68)

Slurry reactors : The complete heterogeneous modelling of the hydrogenation of glucose on suspended Raney nickel catalyst was done using statistical methods of non-linear estimation of model parameters and the latest methods (including two new methods) of model descrimination.

Solid catalysed reactions : Kinetic study of the vapour phase esterification of acetic acid with normal aliphatic alcohols was undertaken to establish generalized models for esterification reactions and to find out rate and adsorption constants from physico-chemical data. Work on ethyl alcohol acetic acid system was carried out at length. Many runs were taken at different conditions. Difficulties were experienced in the analysis of the product and the performance of the reactor. Both have been overcome and new series of runs are being taken.

Gas-solid reactions : As a part of the application of the diffusion model for the sphere, the treatment was extended to various geometrical configurations like long cylinder, right circular cylinder, infinite cylinder and flat plates (one face sealed and both faces open). Mathematical expressions for the diffusion model for these shapes have been derived and linearized to facilitate graphical representation. The McKewan-Levenspiel model for the kinetic region for the sphere was also extended to include these shapes.

Using right circular cylindrical pellets prepared from zinc sulphide at various pressures of compression, the critical temperature was located as a function of porosity. The same relationship was then used for infinite cylinder and flat plates. The models for diffusion control and kinetic control were found to hold good in the respective zones for the oxidation of zinc sulphide pellets of other shapes also. Experimental values of D_e were in good agreement for all the shapes at a particular temperature, and the K_g values also agreed closely with those estimated from mass transfer correlations.

The activation energy for the kinetic regime was 7.55 kcal/mole and for the diffusion regime 1.92 kcal/mole. The Aris approximation for the diffusion length was applied for the first time to non-catalytic solid-gas reactions and was found to be satisfactory.

Combined reactors : The optimality criteria for a mixed reactor followed by a tubular reactor, the MT combination, under adiabatic conditions have been varified for a simple reaction. The criteria for optimality have been developed for parallel reactions and for other complex reaction networks. These equations are being tested with available kinetic data.

Efforts are being made to extend the above theories to the case of an adiabatic MT reactor with cooling in the mixed reactor and intercooler between the mixed and tubular reactors.

Catalyst fouling : In continuation of the earlier work, two theoretical models, one for a competitive reaction system and another for a competitive consecutive reaction system were developed. Computation work is in progress.

Mixed catalysts : The performance of dual functional catalysts is being studied in a fixed bed reactor. The system chosen for analysis is the dehydrogenation of cyclohexane to benzene and reduction of nitrobenzene to aniline.

10.1.2 *Fluidization :* (B-14.5/68)

A device for measuring the frequency of vibrations in the fluidized bed was developed, tested and found to be very satisfactory. Incorporation of this in the system would complete the work of instrumentation. This is in progress.

10.1.3 *Continuous fractional crystallization :* (B-14.14/70)

The column was modified incorporating sample points and temperature measurement points. The stirrer was also modified to improve the scraping action. A long pitched screw conveyor is being fabricated. Work is in progress with the present set-up.

10.1.4 *Ammoxidation of hydrocarbons :* (B-14.15/71)

Aromatic nitriles are industrially important chemicals which can be prepared by a number of ways. Ammoxidation technology opens up a one-step direct route for their manufacture. Work on the development of catalysts from indigenously available materials is in progress. A catalyst for the ammoxidation of *o*-xylene to phthalonitrile has been developed and its assessment is in progress.

10.2 *Process development*

10.2.1 *Ketene from acetone :* (SP-58/70)

Ketene is a highly reactive chemical and is used as a starting material for the manufacturing of many intermediates. It is specially useful in the manufacture of acetic anhydride, acetoacetic ester and other acetyl derivatives. These chemicals are imported at present (imports around 500 TPA). Ketene can be prepared either from acetone or acetic acid. The former route has an advantage of providing an industrial outlet for excess quantity of acetone available from petrochemical sources.

The sponsored work was undertaken essentially to collect data for the design of a 200 TPA plant. Accordingly, as a first step, a process was standardized on a laboratory scale achieving 70% yields based on acetone. The sponsor, however, intended to continue further work at the factory.

During the last year, at the instance of the sponsor, a rector of 10-15 kg/hr was completely designed, and along with the equipment list, estimated sizes of the equipment etc., has been submitted to the sponsor.

The project has now been concluded.

10.2.2 *Propylene oxide :* (SP-62/70)

Propylene oxide, a major industrial chemical has manifold uses in the manufacture of propylene glycol, polyester resins, polyglycol ethers, nonionic surfactants, and alkanolamines. This chemical is not presently manufactured in the country. Import statistics are not available.

The starting material (propylene), not being available, was prepared by the dehydration of isopropanol over a basic alumina catalyst and compressed in a cylinder. Laboratory scale data was collected for the chlorohydrination step on three different types of reactors and laboratory scale work on this step is practically completed. A few experiments were carried out in a semi-continuous reactor for the dehydrochlorination of propylene chlorohydrine to propylene oxide. Steps have been taken to procure the necessary material and equipment for the pilot plant unit for chlorohydrination steps.

10.2.3 *Acrylic acid and acrylates from acrylonitrile :* (SP-63/70)

Indian Petrochemical Corp. Ltd. (IPCL), Baroda, is putting up a plant for the production of 24,000 TPA of acrylonitrile. Although a major portion of the production will be utilized for the manufacture of acrylic fibres and elastomers (nitrile rubber) a portion of it is to be utilized for the production of acrylic acid and its esters which find extensive use in leather and textile finishing; paints, paper, plastics, adhesives, ion-exchange resins; and as additives to drilling mud and lubricants.

The estimated demand at present is about 2000 TPA valued at Rs.2 crores which is likely to be doubled in the next few years.

Acrylic acid

Initially a batch process for the production of acrylic acid in the form of aqueous solution was developed on a laboratory scale. The process was modified later to make it continuous giving yields of the order of 90% based on acrylonitrile.

The system of acrylic acid, ethylene dichloride and water was studied experimentally to determine equilibrium data for the partition of acrylic acid. Studies on the extraction of aqueous acrylic acid by ethylene dichloride to obtain glacial acrylic acid will be continued. This data will be used in designing a counter-current extraction column

Methyl acrylate

Various batch and continuous methods for the preparation of methyl ester were tried. Finally a method based on the reaction of acrylamide sulphate (prepared from acrylonitrile) with methanol in a counter-current reactor cum distillation unit was standardized. The yields obtained are of the order of 85%.

Studies on the extractive distillation of the ester-alcohol mixture using suitable solvent for isolating the pure ester will be undertaken.

10.2.4 *Oxyurea :* (SP-64/70)

Dimethylol ethylene urea is a well-known anti-crease reagent in textile finishing. A scheme on sponsored basis was undertaken to develop a commercial process for the chemical. The process involves two steps : (a) preparation of ethylene urea from urea and ethylenediamine and (b) reacting ethylene urea with formaldehyde to yield dimethylol ethylene urea.

The first step was fully standardized last year on 1 kg/batch scale, giving yields of 95-97% on the basis of chloroform soluble matter.

The process has now been completely developed on a pilot plant scale. Ethylene urea is developed on 13 kg/batch scale, while the second step of dimethylol ethylene urea on 10 kg/batch scale. After concluding the developmental work, a complete project report was prepared and has been forwarded to the sponsor. The finished product, about 100 kg. was supplied to the sponsor for their in-plant trials. The product was found acceptable.

10.2.5 *Oxalic acid :* (SP-80/72)

The bark waste of a timber tree-*Terminalia tomentosa* is known to contain considerable quantity of oxalic acid. The wood is available in plenty in Chandrapur district of Maharashtra state. Work was undertaken on behalf of a private firm for establishing process conditions and plant designs for the extraction of oxalic acid.

A successful process to extract as high as 30% of the bark weight has been developed. The necessary engineering data for scaling up the process has been collected.

A plant of 900 TPA capacity is being erected by the sponsor and is expected to go in production by the end of 1973.

10.2.6 *Terephthalic acid (TPA) and benzoic acid :* (PP-9/69)

TPA is a key intermediate for polyester fibre. In view of the increasing demand for polyester in the country, it has become necessary to develop an indigenous technology for this chemical.

Earlier a bench scale work on TPA was carried out following both the Henkel processes; i. e. (I) isomerization of phthalic acid,as also of phthalic anhydride and (II) disproportionation of benzoic acid. Encouraging results were obtained in both the cases. On economic consideration, however, the later route of disproportionation of benzoic acid was selected as the one to be followed up for further investigation on a large scale. This process has certain advantages over the other processes. The basic raw material toluene, is cheap and abundantly available. Secondly the by-product obtained in the process is benzene which is costlier than the starting chemical toluene.

The reactor design happens to be the most important and critical feature of the whole process. A reactor was designed and fabricated in the laboratory, and several runs on 1.5 kg. scale were carried out satisfactorily.

Further improvement in the design of the stirrer and some other mechanical details of the reactor were found desirable. In this connection MERADO, Poona, was contacted for assistance in the design and fabrication of a reactor of 10-15 litre capacity. Pilot plant runs would be carried out on this reactor.

10.2.7 *Methyl chlorosilanes :* (ATT-13/66)

Methyl chlorosilanes are used for making dimethyl polysilicones which have extensive applications such as dumping fluids in instruments, hydraulic fluids in precision tools, an efficient liquid dielectric in transformers, lubricants for rubber and plastic surfaces, moisture repellent etc. These are also used in polishing agents in automobile and furniture polishes. The market for silicones and silicone formulations is rapidly increasing. The current demand is estimated to the order of 100 TPA valued at Rs. 40 lakhs.

A reaction of methyl chloride and ferrosilicon in the presence of a catalyst to give chlorosilanes was developed on a laboratory scale.

Reactors with different types of stirrers have been studied with a view to arriving at a suitable design for this gas-solid reaction. Based on this study a pilot plant to convert about 20 kg./batch of ferrosilicon to Rochow liquid (a mixture of chlorosilanes) has been fabricated and installed. The scale-up and engineering data will now be collected on this assembly.

10.2.8 *Ethylenediamine* : (ATT-58/68)

The range of applications for ethylenediamine is extremely wide. It finds use in the synthesis of surface active and chelating agents; as inhibitors, rubber accelerators, fungicides, insecticides; in synthetic waxes, asphalt wetting agents, resinous polymers with formaldehyde, etc. The entire need of the country is presently being met by imports. The demand is estimated around 700 TPA valued approximately at Rs. 33 lakhs (c.i.f.) which is likely to go up to 1500 TPA in the next five years.

In order to economize the working of the process developed earlier, efforts were directed from a medium pressure reaction to a high pressure technology. Accordingly, experiments were carried out at 1000 psig batchwise in a high pressure autoclave using liquid ammonia to establish the reaction conditions with minimum steam requirements. The results showed higher conversion and increased yield of ethylenediamine with higher molar ratio of ammonia to ethylenedichloride.

On the basis of the preliminary material balance and equipment flow sheet submitted earlier to the project engineering firm, a feasibility report of the process was prepared by the firm on behalf of a party, for which all possible assistance was provided.

A few aspects of the process, not studied so far, are being investigated. If encouraging results are obtained the present process may be slightly altered

10.2.9 *Silicon tetrachloride and ethyl silicate* : (ATT-120/68)

Silicon tetracholride is used in the manufacture of ethyl silicate and microsilica. The demand is expected to be around 2,000 TPA. Ethyl silicate is used as a binder in precision casting and as a bonding agent for commuted materials. The best known compound is ethyl silicate-40 (ES-40) which contains 40% silica by weight. The demand is expected to be around 100 TPA valued at Rs. 8.50 lakhs (c.i.f.).

Starting with silicon tetrachloride, the preparation of ES-40 was standardized earlier on 1 kg./batch scale.

A pilot plant to chlorinate 2-3 kg./hr. of enriched ferrosilicon to yield $SiCl_4$ has been designed, fabricated and is being assembled.

10.2.10 *Ethanolamines* : (ATT-160/70)

There has been a consistent demand for ethanolamines in recent years in view of their various uses in detergent manufacture, gas scrubbing, textile chemicals, cosmetics and toilet products. About 1000 tonnes of the various ethanolamines costing about Rs. 26 lakhs have been imported during the period 1970-71.

Kinetics of the formation of mono- di- and tri-ethanolamines in the reaction of ammonia and ethylene oxide was studied. Based on the data analysis, batch experiments were designed to establish the conditions for obtaining products rich in either monoethanolamine or triethanolamine. The reactions were carried out in a 14 litre mild steel reactor and the individual amines were separated by fractional distillation. Triethanolamine rich mixture containing about 60% of triethanolamine and 40% of di- and monoethanolamines have been obtained. Similarly monoethanolamine rich mixture contains about 60% of monoethanolamine and 40% of remaining two amines. Overall yield in each case is 85%.

The project is expected to be completed shortly. Few more experiments will be conducted to test the reproducibility of the results.

10.2.11 *Diphenyl and chlorinated diphenyl :* (ATT-177/71)

Diphenyl is mainly used in the manufacture of ' Dowtherm ' (heating medium) and chlorinated diphenyl which has various industrial applications. The manufacturing process involves specialized high temperature technology. The chemical is not being produced in the country at present. The imports are to the tune of 600 TPA.

A special bench scale unit (capacity 200g./hr.) to convert benzene to diphenyl at high temperature has been designed and installed wherein critical factors of the reaction such as residence time, conversion temperature control, etc., will be studied to obtain optimum reaction conditions and the process design data.

Chlorinated diphenyl is used as heating media, impregnating materials for paper capacitors, plasticizers and as solvents. The imports are about 500 TPA.

A process involving direct chlorination of diphenyl has been developed on a laboratory scale of 1 kg./batch. The products obtained are being compared with the specifications of the imported samples to confirm their suitability for various uses.

The process will be scaled up before release to industry.

10.2.12 *Tetrakis (hydroxy-methyl) phosphonium chloride (THPC) :* (ATT-178/71)

THPC is used as an ingredient of certain resins which impart flame resistance to cotton fabrics. It is prepared by treating phosphine with aqueous formaldehyde and hydrochloric acid. At present it is not manufactured in the country.

Preliminary work on the preparation of this compound was undertaken. Several experiments were carried out on a laboratory scale and yields of over 90% were obtained. A model for this gas-liquid reaction system is under development.

Further work on this project will be undertaken if sponsored by industry.

10.2.13 *Sorbitol*

A medium pressure process for the preparation of sorbitol from dextrose was standardized and offered to industry. On inquiry from a party interested in a high pressure technology, experiments with high pressure were carried out on 500 g./batch scale with desired results, wherein reuse of catalyst could be avoided and time cycle considerably reduced. The high pressure process is also now being offered to interested parties.

10.2.14 *Selective alkylation of naphthalene :* (AB-92/71)

Development of processes, analogous to the cumene-based process for phenol as new routes for production of α-, β-naphthols, cresols and polyhydroxy benzenes is of wide industrial interest. These processes may open new utilization pattern for some of the surplus petrochemicals.

Propylation of naphthalene with a view to obtain primarily mono-isopropylnaphthalene has been studied on laboratory scale. A vapour phase catalytic dehydration of corresponding alcohol using indigenous bauxite catalyst was carried out to obtain 98% pure propylene.

Alkylation was done on naphthalene dissolved in a suitable solvent after addition of desired quantity of catalyst by complete absorption of known amounts of propylene. Several runs were taken and reaction variables such as concentration of naphthalene in solvent, temperature, propylene to naphthalene molar ratio, catalyst (H_2SO_4) to naphthalene molar ratio, time for absorption and reaction, etc., were studied.

Analytical procedures have been worked out to estimate the two mono (α and β) isomers, polyalkyl and unreacted naphthalenes and determine the conversions and selectivities.

Attempts are being made to obtain conditions for a technically feasible process for α and β isomers.

10.2.15 *Molecular sieve catalyst for alkylation reactions :* (AB-84/70)

The project aims at development, testing and evaluation of various molecular sieve type catalysts for vapour phase alkylation of aromatics. This may open up new areas of utilization for presently surplus bulk petrochemicals.

Based on X- and Y-type synthetic zeolites, fifteen catalysts (12 of X-RE and three of Y-RE type) containing different active materials were prepared and evaluated for the vapour phase alkylation of benzene using 1:3 and 1:5 alcohol/benzene mixture at different temperatures. The reaction products were analysed by the VPC.

Conversions of alcohol to ethylbenzene taken as an index of catalyst activity for this reaction indicated that mostly catalysts X-RE 9 to 12, all belonging to one sub-group of X-RE, performed better, under all conditions. The effect of temperature, feed composition and feed rate were then investigated with X-RE-12. The remaining three, X-RE 9 to 11, were then tested under chosen combination of the three reaction parameters.

The adsorption properties and the X-ray crystal structure of these catalyst samples are being simultaneously studied.

The unusual catalytic properties exhibited by the synthetic zeolites are often attributed to the nature of active sites present in the materials. With a view to understand and correlate this with the alkylation properties of such catalysts, a series of rare-earth substituted zeolite catalysts have been prepared and study of the nature of their active sites both by adsorption and infra-red techniques is being undertaken.

10.2.16 *Maleic anhydride :* (B-14.16/71)

This is a key intermediate for a number of ester type resins. Although few parties in the country have been licensed to produce maleic anhydride, none has yet made any appreciable progress. Imports of maleic anhydride during 1970-71 were 700 tonnes valued at Rs. 36 lakhs. Estimated demand for 1975-76 is of the order of 3000 TPA valued at Rs. 1.5 crores.

The present investigation aims at the maximum conversion of benzene to maleic anhydride and at the development of a suitable catalyst for this highly exothermic oxidation reaction. Several experiments were carried out to assess various catalysts. A catalyst giving 60% conversion of benzene to maleic anhydride was prepared from locally available materials.

Experiments were conducted to recover the unreacted benzene by absorbing over activated carbon. Further runs are in progress for benzene recovery. The chief aspect of the process to be finalized is the recovery of benzene to obtain yields of 85% or more.

10.2.17 *Follow-up actions on the processes already developed at the laboratory*

Hexachloroethane (*HCE*) : A continuous process earlier developed has been released to M/s. Industrial Oxygen Ltd., Poona for industrial implementation. A pilot plant of 1 kg./hr designed and commissioned by the laboratory was run at the factory and hexachloroethane of the required specifications was produced. The field trials of the product carried out by the consumer have been reported to be satisfactory. Design work for a 300 TPA plant is likely to be undertaken on behalf of the party.

Chlorobenzene : A continuous process developed earlier in a sponsored scheme is being implemented by M/s. Hindustan Organic Chemicals Ltd, Rasayani. Assistance in providing engineering designs for the commercial plant is being rendered to the engineering firm to whom the tender has been given by HOC.

Chloromethanes : The process for carbon tetrachloride and chloroform initially developed at the laboratory on a bench scale is now being further developed on a semi-commercial scale, jointly by the laboratory and M/s. Standard Mills Co. Ltd., Chemical Division, Thana. Several Continuous runs were carried out on an integrated plant of capacity 300 TPA at the factory site. Distillation trials on the crude product were also carried out to obtain pure carbon tetrachloride. The necessary modifications in the reactor have been implemented to avoid run-away reactions and to obtain a product of the desired composition.

Dimethylaniline : Training and demonstration runs on 70 kg. batch were successfully completed. The process has been taken over by M/s. Sahyadri Dyestuffs & Chemicals (P) Ltd., Poona, who has started production of the chemical since January 1972 with the assistance of the laboratory in designing the plant and carrying out trial runs. The present plant capacity is 150 TPA and the product obtained in the plant meets the defence requirements and specifications.

11. NEW ANALYTICAL METHODS

11.1 *Chemical and electro-chemical properties of free and co-ordinated organic ligands*

Basic work in this field is being undertaken with two primary objectives : (i) to develop new analytical methods for organic compounds relating to the projects in the NCL, (ii) to explore new electro-chemical routes for organic compounds of commercial significance. It may be recalled that basic work in this area has already provided methods of analysis for nitrobenzene in aniline, assay of and impurities in *p*-nitrophenol, and purity of *p*-aminosalicylic acid.

11.1.1 *Analytical aspects*

The nitro prusside ion shows interesting properties presumably because the nitric oxide (NO) can behave as a positively charged, negatively charged or neutral ligand. In alkaline solution sodium nitroprusside reacts slowly with acetone to give an intense violet colour. The polarographic data on this reaction suggests the following steps. Oximino acetone is formed which is in equilibrium with its nitroso tautomer. The equilibrium is pH dependent, the nitroso form predominating in acid pH. Iron appears to be coordinated both to the cyano and oximino groups.

It is observed that sodium nitroprusside under the influence of ultraviolet radiation reacts with primary aromatic amines in alkaline medium to give intensely green coloured products. The mechanism of this reaction is still obscure, but it has applications in analysis. For instance, aniline in acetanilide can be readily detected and determined.

The differential polarographic behaviour of ethylenediamine, diethylene triamine and triethylene tetramine in presence of transition metal ions can be exploited to provide an elegant method for the determination of the individual amines in their mixtures. The differential behaviour arises from the fact that the higher amines coordinate with transition metals more strongly than the lower ones. Consequently the polarographic half-wave potentials of reduction of the coordinated metals are shifted to more negative potentials, different amines displacing the potentials to different degrees.

It is reported that pentavalent vanadium gives intensely coloured solutions with iso-nicotinic acid hydrazide. A critical examination of this reaction suggests that vanadium (V), is initially coordinated to the ligand. This species is unstable and by a process of intra-electron transfer the ligand is oxidized giving rise to nitrogen gas and the pentavalent vanadium is reduced to the quadrivalent state.

Electro-reduction of nitrobenzene and nitrophenols in presence of suitable metals and supporting electrolytes is being investigated. The reduction product of nitrobenzene is greatly influenced by the electrolyte acidity and metal. Direct reduction to *p*-amino phenol is favoured in presence of certain metal ions.

To understand the rather complex reaction mechanism between nickel(II) and diacetyl monoxime, the reaction between diacetyl monoxime and copper(II) was investigated in the first instance. Spectrophotometric and potentiometric studies indicated only weak coordinate bonding, though data by the two methods were at variance with each other. Depending on the concentration of the ligand and pH, 1 : 1 and 1 : 2 species were formed with formation constants 5.4 and 2.7 for log K_1 and log K_2 respectively. The 1 : 2 species was not extracted into polar or non-polar solvents indicating a planer configuration or hydration. Hydration is suggested by the fact that in the presence of aromatic nitrogen bases such as pyridine and quinoline, the species was extracted to some extent. This is consistant with the idea that the nitrogen base replaces the coordinated water molecule, making the coordination compound less hydrophilic and therefore more readily extractable.

11.1.2 *Synthetic aspects*

An attempt is being made to exploit the data on basic studies relating to redox behaviour of organic ligands in preparative organic chemistry. For instance, direct reduction of nitrobenzene to *p*-amino phenol rather than to aniline was observed under certain conditions. Work on a 25 g. scale has demonstrated that by the use of appropriate transition metal ions, the chemical reduction of nitrobenzene to *p*-amino phenol is possible. Difficulties are encountered in the separation of the end product without further decomposition.

The oxidation of γ-picoline to isonicotinic acid with nitric acid requires rather higher temperature which puts a strain on glass-lined equipment. Laboratory experiments indicate that the reaction temperature can be substantially brought down in the presence of copper salts without impairing the yield.

11.2 *Microanalysis*

11.2.1 *Simultaneous estimation of chlorine and bromine in organic compounds:* A rapid method for halogen estimation has been standardized using the Schoniger flask combustion technique and argentometric titration. Attempts were made to modify the method for the simultaneous estimation of chlorine and bromine in organic compounds. It has been possible to estimate the number of atoms of halogen in the molecule but determination of exact percentage is not yet satisfactory.

11.2.2 *Estimation of sulphur in organic compounds containing phosphorus as additional element :* A method has been standardized and results on seven compounds indicate its successful application. After analysing some more compounds (which are being prepared) the method will be reported for publication.

11.2.3 *Studies to use silica gel as an adsorbent for moisture in the microdetermination of carbon and hydrogen :* It was observed that the adsorption efficiency of silica gel drops down after a couple of estimations. Hence it cannot be used for the purpose indicated above in microanalysis. The work has since been discontinued.

11.2.4 *Quantitative separation of chloride and bromide ions by ion exchange chlomatography :* About 2 mg. quantities of each of sodium chloride and sodium bromide were separated quantitatively on a column of anion exchange resin, Amberlite IRA-400. Attempts are being made to apply the process to mixtures containing sodium iodide also.

11.2.5 *Isolation, identification and estimation of isopropylnaphthalenes :*

This work has been undertaken in connection with the project on synthesis of isopropylnaphthalenes.

The products from various batches were analysed for unreacted naphthalene, mono- and di-isopropyl naphthalenes by VPC. As it was necessary to know the proportion of α-isopropyl naphthalene and β-isopropyl naphthalene in the mixture, the mixture was first subjected to preparative GLC. After isolating the mono-isopropyl naphthalenes from the rest of the material, attempts were made to separate α-and β-isopropyl naphthalenes. However, the available GLC facilities seem to be inadequate for the purpose. This separation can be achieved by using capillary columns in GLC. Attempts are being made to prepare glass capillary columns. As an alternative, it has been possible to use NMR to have some rough idea about the proportion of these components in a mixture.

12. INSTRUMENTATION TECHNOLOGY

12.1 *Electron spin resonance (ESR) spectrometer :* (ATT-64/68)

Designing of 100 kc system and construction of magnet power supply using chopper comparator and drift free a. c. amplifier was reported earlier.

Flat bed recorder assembly is nearing completion and the magnet sweep circuitry is being tested. Cavity for the microwave bridge has been fabricated and it is to be tested on a standard set-up. Efforts are being made to fabricate a sophisticated cavity for 100 kc modulation system.

Feasibility tests will be conducted after procuring optical parts from NPL, New Delhi.

12.2 *DC Recording polarograph :* (ATT-134/69)

The development work on this project has been completed and one prototype has been fabricated. Performance tests have been carried out and they prove to satisfy the following specifications :

Sensitivity	..	0.003 μa/mm to 1.5 μa/mm. in 20 steps
Time required for one complete bridge rotation	..	20 minutes
Full deflection of the recorder	..	2.5 mV
Response time of the recorder	..	10 seconds
Chart speed of the recorder	..	40 inches per hour.

The know-how is available for commercial exploitation.

12.3 *UV-visible spectrophotometer* : (ATT-173/71)

An imported instrument of this type is being handled for the last several years and enough scientific knowledge about its circuitry, optics and working principles etc., has been collected. The present imported cost of the instrument is around Rs. 60,000/- (c.i.f.). It is further estimated that more than 50 units of the instrument will be required in the country every year, for the use of research institutions, universities and quality control laboratories of the industry.

Efforts have been made to procure the optical components such as plane mirrors, toroid mirrors, paraboloid mirror etc., from indigenous sources and it is hoped that they will be received in 2 to 3 months time. Efforts are also being made to procure silica prism and grating from abroad, which are absolutely essential as they constitute the dispersing elements forming the heart of the spectrophotometer.

Meanwhile approximate designing of optical and electronic units is in progress. A prototype unit is expected to be ready by end of 1973

12.4 *Infra-red spectrometer* : (ATT-190/72)

Preliminary work on spectrometer base, prism and mirror mounts and experimentation with solid state circuitry has been carried out. Appropriate components are ordered from various indigenous sources. Few components received are under rigorous testing for quality. Basic optical layout is complete. Few components are still to be imported and the work on optics is held up for these critical components.

Meanwhile efforts are continued for the better design of wavelength drives, slit mechanisms and recording system.

It is anticipated that by the end of the year all components will be at hand and the fabrication work for the prototype could be undertaken.

12.5 *Instruments for research in solid state*

Fabrication of an instrument for automatic recording of current voltage curves for heterojunction devices was reported earlier. An advanced version of the unit has been designed and fabricated for the study of thermally stimulated current in semiconductors. The all solid state circuit incorporates a high gain feedback loop which effectively computes the difference between a present linear reference signal and the sample temperature and produces a correcting power input proportional to the difference. Maximum power handling capacity is nearly 120 watts (peak). Linearity of 1% or better has been attained over the temperature range-180°

...10

to 100°. Careful alignment of heaters and sensors have eliminated hunting which is sometimes detected in high gain control systems.

Photothermoelectric effect : An instrument for the study of the photothermoelectric effect has been fabricated. This apparatus includes a vacuum cryostat, controlled d. c. power for the heaters and vacuum monitor. Studies on thin layer of CdS and CdSe are in progress using this technique.

13. INFRASTRUCTURE ACTIVITIES

13.1 *Analytical groups*

13.1.1 *Physico-chemical analysis*

This group is primarily engaged in the chemical examination of raw materials, intermediates and finished products relating to the various projects in progress in the laboratory. Besides routine analysis, developmental work on new analytical procedures is another important activity of this group.

Analytical methods have been developed and standardized for the following projects.

Ethanolamines: Potentiometric methods for the determination of individual amines i.e. a mixture of mono- di- and tri-ethanolamines have been developed. Selective reactions of these amines with acetic anhydride and salicyl aldehyde have been taken advantage of in these procedures.

Ethylenediamine: Methods have been worked out for the estimation of ethylenediamine, diethylene triamine and triethylene tetramine in their mixtures.

The basis for these methods are :

(i) Preferential distillation of ethylenediamine with *n*-octanol.

(ii) Measurement of the colour of the copper complexes at varying and controlled pH.

(iii) Forming the nitroso amines of the higher amines with nitrous acid followed by polarographic analysis of the nitroso amines.

(iv) pH controlled polarographic reduction of the copper complexes.

A copper coating on aluminium metal or aluminium base alloy has definite advantages in the electrical and electronic industry. A dip process has been developed for the adherent coating of copper on aluminium surfaces. It is essentially based on copper deposition from alkaline borate-phosphate baths.

13.1.2 *Microanalysis*

The major activitiy of this group includes microanalysis of organic and organometallic compounds for various elements (C,H,N, etc.) functional groups and other estimations like molecular weight determination, neutralization equivalent determination, etc. Microanalytical work of various compounds is also accepted in this group from other research organizations, universities, on payment basis depending upon the internal work load.

Two thousand one hundred and forty samples (2140) were analysed during the period under report for various elements (2082) and functional groups (58).

A number of organic compositions and auxiliaries available under trade names find special applications in various industries. Many of these are imported. Identification of these compositions is usually undertaken on payment basis on behalf of interested parties.

During the period under review, four such products were studied and reports submitted to parties concerned.

13.1.3 *Mass spectrometry*

As an analytical tool special techniques were standardized for the analysis of raw materials, intermediates and finished products related to the various projects of the laboratory.

(i) The starting material-ethylene, required in the manufacture of hexachloroethane was analysed using mass spectral techniques on behalf of M/s. Industrial Oxygen Ltd., Poona, the licensee. It was found that the material contained acetylene, apparantly which has been the cause of getting low yield of hexachloroethane.

(ii) The purity of methyl chloride used in the chlorosilane project has been determined using this technique.

(iii) Finished product analysis was done for phthalate project.

One thousand two hundred and sixty (1260) samples were analysed using mass spectral techniques for various research groups in the laboratory

13.1.4 *Spectrochemical and other analyses*

With the help of various physico-organic techniques such as UV, IR, NMR, Visible spectra, VPC, GLC, etc., analytical and structure elucidation work was carried out in the laboratory for various research disciplines.

Number of samples studied :

NMR	..	3447
UV	..	503
IR	..	4116
VPC/GLC	..	6060
X-ray powder patterns	..	100

13.1.5 *Ultracentrifugal analyses*

During the year, 45 samples were analysed.

13.1.6 *Biological testing of organic compounds*

Under this scheme, 12 selected organic compounds isolated or synthesized in the NCL were sent to Bristol Laboratories, Syracuse, New York, USA, for biological testing and evaluation.

13.2 *Instrumentation section*

The section is primarily engaged in the maintenance and upkeep of electronic, optical and electromechanical instruments such as NMR spectrometer, IR, UV and visible spectrophotometers, X-ray spectrometer, gas chromatographs, colorimeters, servorecorders, electrical furnaces, flame photometers, microbalances, various types of meters, gauges and indicators. The work also includes tracing of the faults and rectification to avoid further occurences; and to improve the existing designs and fabricate spare parts for replacement.

Various types of spares required particularly for the servo controls and spectrometers which are manufactured in the country are routinely tested in this section. A new T-60 NMR spectrometer has been installed.

Total number of jobs including test reports that were attended to is about 400.

Special instruments fabricated

(i) At the request of Armed Forces Medical College, Poona, Electro Cardiograph (ECG) pre-amplifier for twin beam oscilloscope using integrated op-amplifires was fabricated and supplied to them.

(ii) Another instrument namely ' Automatic Peritonial Dialysis ' was designed and fabricated for Armed Forces Medical College, Poona.

13.3 *Engineering section*

13.3.1 *Mechanical engineering*

Following special installations are routinely operated and maintained :

Liquid air plant, liquid nitrogen plant, telephone exchange, steam boilers, ice plant, cold storage plant, air conditioning plants, window type air conditioners, refrigerators and water coolers.

Apart from rendering day to day maintenance and engineering services such as steam, compressed air, vacuum, gas, water and electricity to the laboratory, several pilot plants were designed in collaboration with the scientists concerned and fabricated.

The total job orders executed by this section during the year were over 3150.

A few of the pilot plant fabrication jobs carried out during the year are mentioned below :

(i) Coil type M. S. reactor for diphenyl project.

(ii) Reactor and vapouriser for monoethylaniline project.

(iii) Modifications to workshop-fabricated coating machine for cold punching laminates, sponsored by Formica India Ltd., Poona.

Special equipment fabricated : This section also undertakes developmental work on fabrication of equipment and components which save foreign exchange. Following have been fabricated during the period under review :

(i) Oscilating type (180°) motorised shaker, complete with stand' bottle holder, etc.

(ii) Photo cell holder, graduated up to 1/2 degree accuracy for electron diffraction work.

13.3.2 *Civil engineering*

During the period under review, following major civil constructions were completed :

Community block; Staff quarters (H-type 10 Nos. and G II/A type 10 Nos.); Hostel (20 rooms and 10 suits) and water storage reservoir (10,000 gal. capacity).

Total floor area of the above constructions was about 24,000 sq. ft. and the corresponding cost was about Rs. 5.57 lakhs.

13.4 *Glass blowing section*

This section primarily undertakes repairs of glass apparatus for various research groups in the laboratory. During the period under report, 1540 jobs were completed.

Glass assemblies of various types are fabricated as per the specific drawings furnished by scientists. About 2000 fabrications of glass assemblies were carried out during this year. Of these, 330 fabrication jobs were related to the sponsored projects work. About 6380 standard ground glass joints were fabricated for internal use.

13.5 *Library*

The library houses about 56,000 accessioned items consisting of books, periodicals, publications, patents, standards, technical reports, etc., in its stock. 2448 volumes comprising of (i) bound volumes of journals and (ii) 724 books were added to the library during the period under review. 760 periodicals are received currently in the library.

Loan and consultation facilities are extended to outside persons from private concerns, Govt. departments, universities, colleges and other research institutes on the basis of membership etc. Nearly 250 persons from these organizations made use of the library during 1971-72.

NCL is a Patent Inspection Centre for Indian Patents. The Library receives and maintains a complete and up-to-date holding of Indian patents.

13.6 *Division of Technical Services*

Commercial intelligence

As a follow-up of Advisory Panel recommendations, market data on following items was collected : Digitonin and *Digitalis purpurea,* cellosolves, diphenyl, chlorinated diphenyl and morpholine.

Compilation and documentation of techno-economic, market and scientific data relevant to NCL research programme was regularly carried out by referring to newspapers, journals and through correspondence.

Industrial liasion and coordination

About two thousand and five hundred enquiries pertaining to NCL projects and general technical enquiries from different individuals, industries, Govt. organizations and starred questions from the Parliament were attended to. One hundred and seventy applications in chemical and allied industries for industrial licences with foreign collaboration were scrutinized with a view to ascertain the necessity of foreign collaboration for the technical know-how. Techno-economic data from the licence applications received since 1965-66 was indexed.

One thousand six hundred and fifty (1650) visitors were shown round the laboratory during the year. Enterepreneurs visiting NCL were attended to and information on know-how available at NCL was furnished.

Follow-up of the processes referred to NRDC was carried out. Non-technical notes on 21 processes (approved by PRC) along with cost-estimates were prepared. The total number of non-technical notes which are now available for circulation to interested parties is about seventy five.

Liaison with CSIR

Twenty nine NCL processes cleared by the NCL PRC for release to industry were referred to CSIR/NRDC for licensing to the interested parties.

Reports relating to the activities and achievements of NCL were sent every month for inclusion in the monthly report to the Union Cabinet, CSIR News and Research Utilization Data.

Success story for acetanilide was prepared and forwarded to DGSIR for publicity.

Internal coordination

A close contact with the programme and activities of the laboratory was maintained by associating one member of the DTS to each division.

The division continued to render statistical, photographic and draftsman services to other divisions in the laboratory.

Reports

(*i*) *Reports on NCL*

Information regarding Annual Plan 1972-73 in the prescribed proforma was sent to CSIR under the heads (a) pilot plants in operation (b) new pilot plants to be set-up (c) major R and D projects related to the socio-economic objectives of the fourth-five year plan and (d) new major R and D projects to be started in the next year and related to socio-economic objectives of the plan.

A quarterly progress report for July-Sept. 1971 on achievements of socio-economic objectives was sent to CSIR in prescribed profroma.

Report on foreign visits by NCL scientists was sent to CSIR for onward transmission to Dept. of Science and Technology. Views of NCL scientists regarding international collaboration were communicated to CSIR.

(ii) *Other reports and review articles*

1) Proceedings of the UNESCO Regional Seminar on ' Problems of transition from laboratory research to industrial application' were compiled, edited and produced.

2) NCL Annual Report 1970-71 and Achievements 1970-71

3) NCL Half-Yearly Report (April-Sept. 1971)

4) NCL Research Programme (1972-73)

Review articles

1) Rajan, J. V. and Lele, A. M.
Role of reseach in the creation of modern industries.
Silver Jubilee Souvenir of AOSW, Feb. 1972.

2) Kelkar, D. D. and Lele, A. M.
The role of chemical research in national defence.
Vigyan Pragathi, Special Defence Number, Jan.–Feb. 1972.

3) Saraf, C. U. and Lele, A. M.
Small scale industries and the National Chemical Laboratory.
Paper presented in the Seminar on 'Chemical Industry' organized by the Mysore Small Industries Corpn. Ltd., Bangalore, Feb. 1972.

4) Adke (Mrs.), S. S., Seth., N. D. and Lele, A. M.
Planning and programming in R and D institute.
Paper presented in the HOC seminar on 'Development of indigenous know-how in chemical and allied industries' HOC, Rasayani.

5) Lele, A. M.
Invention, innovations and technology transfer to small industries.
F. A. S. I. I. bulletin for small industry, X, 73 (1971).

14. RESEARCH ANALYSIS AND RESEARCH MANAGEMENT

During the year under review, preliminary techno-economic feasibility reports on 21 completed R & D projects were submitted for the consideration of the Process Release Committee. Studies related to the following projects : (1) Benzoic acid I.P. from methyl benzoate, a bye product of dimethyl terephthalate plant of IPCL, Baroda (2) Thioglycollic acid (3) L-Arabinose C.P. (4) D-Glucosamine hydrochloride (5) D. C. recording polarograph (6) Direct reading spectrophotometer/colorimeter (7) Can lining composition (8) Linseed oil emulsion paint (9) Flexible magnets (10) Radiosonde thermistors (11) 2,2,4-Trimethyl-6-ethoxy-1,2-dihydroquinoline (12) Cashewnut shell gum (13) Cellulose powder (14) Foundry core binder (Sinol core binder) (15) Morpholine (16) Tamarind kernel powder-phosphate and borate (17) Gum arabic substitute (starch based) (18) N,N-Dimethylbiguanide-HCl and phenethylbiguanide-HCL (19) Microfilters (20) Monoethylaniline and (21) Clofibrate.

Cost estimates were prepared in respect of modified processes for the production of phenylacetic acid starting directly from toluene; a process for alkali stable protease (10 TPA); quinidine; ethylene dichloride and ethylenediamine. Terms and conditions were finalized for releasing ethylenediamine project as a turn-key plant to a firm of project engineers. Draft agreement for release of technology on nitrile rubber was prepared.

Contractual research terms were prepared for the following sponsored projects : conversion of bauxite into anhydrous aluminium chloride, xylit, chlorohydroxyquinoline, propoxyphene, oxalic acid from bark of Ain tree, fractionation of turpentine oil, molecular sieves, re-burning of precipitated calcium carbonate from causticizing plant, vinyl acetate, guar gum derivatives, keryl benzene, ketone resin, N,N-dimethyl acetamide and terephthalic acid.

Input-output analysis

Norms for the calculations of financial inputs for research projects were revised for the year 1972-73. Financial inputs were calculated for all the research projects included in the Research Programme 1972-73 and also for projects undertaken during the period April 1968-72.

Research planning and management

In connection with the formulation of Science and Technology Plan for the chemical industry, several background notes were prepared relating to the methodology to be followed, criteria for identification and selection of R & D projects of national importance, and a PERT time schedule for the formulation of Science and Technology Plan.

Two notes, one relating to the management of research and the other on utilization of indigenous research, were submitted for the consideration of the Minister for Planning, Science and Technology. Literature survey on (1) Personnel policy for R & D (2) Transfer of technology and (3) Patents and research productivity were carried out.

A survey on ' Migration of Talents' for Poona area was conducted wherein 40 respondents from the industries and educational institutes in the Poona region were contacted personally. This work was undertaken for the United Nations Institute of Training and Research, (in collaboration with CSIR).

RESEARCH UTILIZATION

TABLE 1 : PRODUCTS MANUFACTURED ON THE BASIS OF NCL KNOW-HOW

T=Metric tonnes

S. No.	Name of the process & Indian patent No.	Field of utilization	Name of the manufacturer (year of commencement of production)	Production		Remarks
				1971-72 Qty./Value in Rs. lakhs	Upto March 1971 Qty./Value in Rs. lakhs	
1	2	3	4	5	6	7
1.	Acetanilide	Intermediate	M/s. Hindustan Organic Chemicals Ltd., Rasayani, through project engineers M/s. R. L. Dalal & Co., Bombay-18 (1969).	1574.75 T 119.64	1211 T 76.43	Exclusive to the project engineer for engineering.
2.	Acriflavine (sponsored)	Pharmaceuticals	M/s. Western India Fine Chemicals, 83, Lalbahadur Shastri Marg, Bombay-80 (1969).	927 kg. 4.00	1752 kg. 7.82	Exclusive.
3.	Adhesives for decorative laminates (sponsored)	Laminates	M/s. Swastik Rubber Products Ltd., Poona-3 (1969).	22 T 2.36	30.75 T 2.90	Exclusive.

1	2	3	4	5	6	7
4.	Anion exchange resin from melamine (71190)	Demineralization of liquids	M/s. Tulsi Industries, Bhosari Industrial Area, Poona-26 (1963).	—	565 cft. 1.75	Non-exclusive.
5.	Antipriming compositions (77081)	Antipriming in locomotives	Research, Design and Standard Organisation, M & C Wing, Chittaranjan (1964).	9 T 0.28	89.70 T 2.32	Non-exclusive.
6.	Bacterial diastase (66096)	Textile desizing	M/s. Chemaux (P) Ltd., Sitaladevi Temple Rd., Mahim, Bombay-16 (1967).	93 T 5.94	248.58 T 11.31	Exclusive.
*7.	tert-Butylcatechol	Synthetic rubber	M/s. Percynic Chemicals, United Bank of India Bldg., Sir P. M. Road, Bombay-1 (1972).	1.57 T 0.77	—	Non-exclusive.
8.	Berberine hydrochloride (sponsored)	Pharmaceuticals	M/s. Nitin Pharmaceuticals, 180/82, Samuel Street, Bombay-9 (1965).	10 T 20.00	6.30 T 12.36	Exclusive.
9.	Cadmium sulphide photoconductive cells	Electronics	(i) M/s. Chinoy Electronics, 64, Koregaon Park, Poona-1 (1971).	631 Nos. 0.08	— —	Non-exclusive.
			(ii) M/s. Gera Engineering Co., 8/352, Boat Club Rd., Poona-1 (1969).	—	0.50	Non-exclusive.

1	2	3	4	5	6	7
10.	Calcium hypophosphite (sponsored)	Pharmaceuticals	M/s. Hypophosphite & Co., 79-F, Princess Street, Bombay-2 (1967).	12 T 7.00	23.25 T 12.30	Exclusive.
11.	Calcium silicate (sponsored)	Low density insulators	M/s. Newkem Products Corp., Harganga Mahal, Bombay-14 (1968).	264.21 T 9.20	503 T 17.80	Exclusive.
12.	Can sealing composition (66194)	Metal can industry	M/s Arya Chemical Works, 114/2A, Dharmatolla Street, Calcutta-13 (1962).	25.9 T 1.66	157.50 T 8.48	Exclusive.
13.	Carbimazole (sponsored)	Pharmaceuticals	M/s. Indian Schering Ltd., Sion-Trombay Road, Chembur, Bombay-71 (1970).	28 kg. 2.32	10 kg. 0.83	Exclusive.
*14.	Catechol	Pharmaceuticals	M/s. Percynic Chemicals, Bombay-1 (1972).	1.95 T 0.34	—	Non-exclusive.
15.	Cation exchange resin from CNSL (59497, 59606)	Demineralization of liquids	M/s. Tulsi Industries, Poona-26 (1960).	—	7, 310 cft. 6.14	Exclusive.

1	2	3	4	5	6	7
16.	Chloral hydrate (sponsored)	Industrial chemicals	M/s. Hindustan Insecticides Ltd., Industrial Area, New Delhi-15 (1963).	0.82 T 0.19	19.91 T 1.94	Exclusive.
17.	Diethyl-*m*-amino-phenol (sponsored)	Dye intermediate	M/s. Sahyadri Dyestuffs & Chemicals (P) Ltd., 117, Vithalwadi Road, Poona-9 (1970).	9.64 T 3.86	4T 2.40	Exclusive.
18.	Dihydroisojasmone	Perfumery	M/s. S. H. Kelkar & Co. (P) Ltd., Bombay-Agra Road, Bombay-80 (1965).	172 kg. 0.60	597 kg. 2.05	Non-exclusive; Technical aid.
19.	Ethylene oxide condensates (sponsored)	Surface active agents	M/s. Hico Products (P) Ltd., Mogal Lane, Bombay-16 (1965).	420 T 52.43	1439 T 165.45	Exclusive.
*20.	Ferrites-Hard	Electronics	M/s. Semiconductors Ltd., Nagar Road, Poona-14 (1968).	2.6 lakh Nos. 2.8	—	Non-exclusive;
21.	Geraniol, citronellal and citronellol (sponsored)	Perfumery	M/s. Radhakishan Rajaram & Co., 141, Samaldas Gandhi Marg, Bombay-2 (1970).	3 T 3.50	2 T 2.60	Exclusive.

1	2	3	4	5	6	7
22.	Hexylresorcinol	Pharmaceuticals	M/s. Unichem Laboratories Ltd., Jogeshwari, S. V. Road, Bombay-60 (1964).		96.40 kg. 0.42	Exclusive.
23.	4-Hydroxycoumarin (62890, 63083)	—do—	—do— (1964).	142 kg. 0.306	347.70 kg. 0.99	Exclusive.
24.	β-Ionone (77225)	Perfumery, Pharmaceuticals	M/s. Industrial Perfumes Ltd., Army & Navy Bldg., M. G. Road, Bombay-1 (1968).	16.30 T 26.08 (approx.)	35.03 T 22.86	Non-exclusive.
25.	Liquid rubber (60555)	Adhesives, Rubber rollers	M/s. K. N. Chari Rubber & Plastics (P) Ltd., 144, Nyneappa Naick Street, Madras-3 (1962).	—	2.2 T —	Non-exclusive; Production for captive use.
26.	Orthotolyl biguanide	Soap	M/s. Industrial Perfumes Ltd., Bombay-1 (1970).	2.20 T 0.60	2.75 T 0.74	Exclusive.

1	2	3	4	5	6	7
27.	Perfumery products based on longifolene (Capinone) (sponsored).	Perfumery	M/s. Camphor & Allied Products, Bareilly (1968).	6. 032 T 4.98	16.121 T 13.30	Exclusive.
28.	Perfumery products based on Δ -carene (Meracene) (sponsored).	—do—	—do— (1968).	1.705 T 0.54	2.797 T 1.05	Exclusive.
29.	Nicotine sulphate	Insecticides	M/s. Urvakunj Nicotine Industries, Petlad-Cambay Road, Dharmaj (Dist. Kaira) (1963).	12 T 2.20	27.81 T 4.44	Exclusive on zonal basis.
30.	Peach aldehyde	Perfumery	M/s. S. H. Kelkar & Co. (P) Ltd. Bombay-80 (1965).	1.23 T 2.14	3.17 T 5.68	Non-exclusive; Technical aid.
31.	β-Phenethyl alcohol (sponsored)	Perfumery	M/s. Sunanda Aromatic Industries, K. R. S. Road, Mettagalli, Mysore (1970).	25 T 8.15	9 T 3.15	Exclusive.

1	2	3	4	5	6	7
32.	Phthalates-dioctyl and dibutyl	Plasticizers	(i) M/s. Alta Laboratories Ltd., Giri Vihar, Khapoli (Dist. Kolaba) (1969).	784.5T(DOP) 47.07	934T(DOP) 69.19	Non-exclusive.
			(ii) M/s. India Carbon Ltd., Dr. A. B. Road, Worli, Bombay-18 (1971).	1860T(DOP) 107.00	318T(DOP) 24.17	Non-exclusive.
33.	Phthalates-diethyl and dimethyl	Plasticizers	M/s. Mysore Acetate and Chemicals Co. Ltd., ' Mysugar Buildings ', Sri J. W. Road, Bangalore-2 (1970).	6.40 T (DEP) 0.48	48.62T(DEP) 3.72	Non-exclusive.
34.	Polyurethane printing rollers (86991)	Printing	M/s. Sree Saraswathy Printing Press Ltd., 32, Acharya P. C. Road, Calcutta-9 (1965).	—	—	Non-exclusive; Reported in production.
35.	Radio opaque dyes (sponsored)	Pharmaceuticals	M/s. Unichem Laboratories Ltd., Bombay-60 (1970).	—	—	Exclusive; Under trial production.
36.	Rigid filters (59608, 66966)	Tube wells	M/s. Ashim Filters, 196, Defence Colony, New Delhi (1965).	1145 Mtrs. 2.90	5275 Mtrs. 10.26	Exclusive.

1	2	3	4	5	6	7
37.	Rubber base contact adhesive (65977)	Adhesives	M/s. K. N. Chari Rubber & Plastics (P) Ltd., Madras-3 (1962).	—	16,925 Ltrs. 1.37	Non-exclusive.
38.	Rubber blowing agent	Rubber chemicals	M/s. Swastik Rubber Products Ltd., Poona-3 (1968).	76 T 11.81	93 T 11.90	Non-exclusive; Technical aid
39.	Rubberized cork sheet	Gaskets	M/s. Bharat Casements (P) Ltd., P. B. No. 89, Baroda (1966).	13.56 lakh Nos. 5.00	32.67lakh Nos. 10.88	Non-exclusive.
40.	Rubber reclaiming agent	Rubber chemicals	M/s. Swastik Rubber Products Ltd.. Poona-3 (1968).	5.06 T 0.80	13.32 T 1.66	Non-exclusive
*41.	Sachets-Hot and Cold	Substitute for hot water bag & ice bag	M/s. Thermo Chem Laboratories, A-39, H-Block, MIDC, Pimpri, Poona-18 (1972).	—	—	Non-exclusive; Trial production started.
42.	Silica gel (sponsored)	Humidity control	M/s. Minco Products, 301/27, T. H. Road, Madras-21 (1963).	11 T 0.88	72 T 4.83	Non-exculsive.
43.	Sisal wax (64958, 65440)	Cosmetics, Polishes	M/s. Aphali Pharmaceuticals Ltd., Station Road, Ahmednagar (1966).	—	15.30 T 1.64	Non-exclusive.

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1	2	3	4	5	6	7
44.	Sorbide nitrate (sponsored)	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay-71 (1969).	91 kg. 1.26	237 kg. 5.37	Exclusive.
45.	Thermistors	Electronics	(i) M/s. Semiconductors Ltd., Poona-14 (1963).	6.28 lakh Nos. 4.4	30.39 lakh Nos. 27.28	Non-exclusive; Technical aid.
			(ii) M/s. Tempo Semiconductors, Subhash Road, Vile Parle (E), Bombay-57 (1963).	10,000 Nos. 0.40	35,000 Nos. 1.41	Non-exclusive; Technical aid.
46.	Vapour phase chromatograph	Instruments	M/s. Associate Instruments Manufacturers (India) (P) Ltd., 26-27, Asaf Ali Road, New Delhi (1969).	27 Units 5.40	38 Units 7.55	Exclusive.
47.	Warfarin	Rodenticides	M/s. Unichem Laboratories Ltd., Bombay-60 (1968).	79.20 kg. 0.44	100.40 kg. 0.60	Exclusive.

* During the period under review, production has been commenced on these four new items.

Table 1 : Continued

Value of some major chemicals produced in Fine Chemicals Project

		Value in Rs.
48.	Alumina (column & TLC grade)	17,665/-
49.	Benzimidazolethiol	12,888/-
50.	Chemibond adhesives	10,922/-
51.	Silica gel (column & TLC grade)	97,270/-

VALUE OF PRODUCTIONS BASED ON NCL KNOW-HOW

Year	No. of items manufactured (including major FCP items)	Value of productions (Rs. in lakhs)
Upto 1967	26	53.22
1967-68	29	39.98
1968-69	47	69.55
1969-70	53	130.22
1970-71	55	283.84
1971-72	*51	471.20
	Total	1,048.01

* Seven items on which no production is reported for more than two consecutive years are dropped from this table. Productions on 4 new items have been established during this year.

TABLE II (A) : PROCESSES RELEASED AND AWAITING PRODUCTION

S. No.	Name of the process & Indian Patent No.	Field of utilization	Name of the party & year of release	Nature of licence/present state of development
1	2	3	4	5
1.	Benzoic acid from toluene (sponsored)	Pharmaceuticals	M/s. Aniline Dyestuffs & Pharmaceuticals (P) Ltd., 28, Apollo Street, Bombay-1 (1969).	Exclusive.
2.	Benzoic acid (IP) from crude methyl benzoate	— do —	Indian Petrochemicals Corpn. Ltd., P. O. Jawaharnagar, Baroda (1972).	Non-exclusive.
3.	Substitutes for Bostik sealants	Adhesives	M/s. Swastik Rubber Products Ltd., Poona-3 (1972).	Non-exclusive.
4.	BD-catalyst (sponsored)	Catalyst for synthetic rubber	M/s. Synthetic & Chemicals Ltd., New Great Insurance Bldg., J. Tata Road, Bombay-20 (1969).	Exclusive.
5.	Butyl titanate	Varnishes, Enamels	M/s. Synthochem, 33-A, Laxmi Nagar, Indore-1 (1970).	Non-exclusive.

1	2	3	4	5
6.	Butylated hydroxyanisole (sponsored)	Antioxidant	M/s. Mhatre Art Studio, Mahant Road, Vile Parle, Bombay-57 (1970).	Exclusive.
7.	Calcium silicate from Wollastonite (sponsored)	Industrial inorganic chemicals	M/s. Chemicals India, Chetak Bldg., P. B. No. 4, Udaipur (1971).	Exclusive.
8.	Cation exchange resin, polystyrene base (74356)	Demineralization of liquids	M/s. Tulsi Industries, Poona-26 (1965).	Non-exclusive.
9.	Chlorinated copper phthalocyanine (sponsored)	Dye intermediate	M/s. Sudarshan Chemical Industries (P) Ltd., 162, Wellesley Road, Poona-1 (1968).	Exclusive.
10.	Chlorides from bauxite residue (sponsored)	Industrial inorganic chemicals	M/s. Dharamsi Morarji Chemicals Co. Ltd., Prospect Chambers, 317/21, Dr. D. N. Road, Bombay-1 (1972).	Exclusive.
11.	Chloromethanes	Industrial chemicals	M/s. Standard Alkali, Chemicals Division, The Standard Mills Co. Ltd., Mafatlal Centre, P. B. No. 1038, Bombay-1 (1969).	Semi-commercial (300 TPA) plant commissioned at the works.

1	2	3	4	5
12.	Chlorobenzenes (sponsored)	Industrial chemicals	M/s. Hindustan Organic Chemicals Ltd., Rasayani (1969).	Exclusive.
13.	Coating for oil filter papers	Oil filtration	M/s. White Cloud Paper Mills, 412, Gultekdi Road, Poona-9 (1968).	Non-excluside; Technical aid.
14.	Coumarin (sponsored)	Perfumery	M/s. Lona Industries (P) Ltd., Ladivali, Tal. Panvel, Dist. kolaba (1970).	Exclusive.
15.	Covering materials from CNSL (74451)	Chemically resistant flooring	M/s. Natson Manufacturing Co., Bhadra, Ahmedabad (1964).	Exclusive.
16.	DDT—Water dispersible	Insecticides	(i) M/s. Lotus Industries, 62/20, Luz Church Road, Madras-4 (1967).	Non-exclusive.
			(ii) M/s. Pesticides Ltd., 138-141, Govt. Industrial Estate, Kandivali (W), Bombay-67 (1967).	Non-exclusive.
17.	Dithranol	Pharmaceuticals	M/s. Indian Dyestuffs Industries Ltd., Kalyan (1968).	Exclusive.

1.	2	3	4	5
18.	Dimethylaniline	Industrial chemicals	M/s. Sahyadri Dyestuffs & Chemicals (P) Ltd., Poona-9 (1972).	Non-exclsusive.
19.	Heat sealable coating composition	Adhesives	M/s. Chandmal Fakirchand Jain, Khajuri Bazar, Indore-2 (1963).	Non-exclusive; Technical aid.
20.	Hexachloroethane	Industrial chemicals	M/s. Industrial Oxygen (P) Ltd., Off Nagar Road, Poona-14. (1971).	Non-exclusive.
21.	8-Hydroxyquinoline (sponsored)	Pharmaceuticals	M/s. Alta Laboratories Ltd., Khopoli (1970).	Exclusive.
22.	Isolation of borneol (sponsored)	Perfumery	M/s. Camphor & Allied Products, Bareilly (1969).	Exclusive.
23.	Ketene from acetone (sponsored)	Intermediates	M/s. Sudarshan Chemical Industries (P) Ltd., Poona-1 (1971).	Exclusive.
24.	Liquid stabilizer for PVC (sponsored)	Plastics	M/s. Swastik Rubber Products Ltd., Poona-3 (1969).	Exclusive.

1	2	3	4	5
25.	Megimide (sponsored)	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay-71 (1969).	Exclusive.
26.	*p*-Menthane hydroperoxide	Synthetic rubber	M/s. Camphor & Allied Products, Bareilly (1969).	Exclusive.
27.	*l*-Menthol from dementholized peppermint oil (sponsored)	Fine chemicals, Drugs	M/s. Bhavana Chemicals Ltd., Laxmi Insurance Bldgs., Bombay-1 (1968).	Exclusive.
28.	Monochloroacetic acid	Weedicides, CMC	M/s. Hico Products (P) Ltd., Bombay-16 (1972).	Non-exclusive.
29.	*p*-Nitroacetophenone (sponsored)	Drug intermediate	M/s. Dey-Se-Chem. Ltd., 8, Rifle Range Road, Calcutta-29 (1971).	Exclusive.
30.	*p*-Nitrophenol	Insecticides	M/s. Hindustan Organic Chemicals, Rasayani (1972).	Non-exclusive.

1	2	3	4	5
31.	Nonylphenol (sponsored)	Surface active agents	M/s. Surfactants (P) Ltd., 28, Apollo Street, Bombay-1 (1969).	Exclusive.
32.	Opium alkaloids	Pharmaceuticals	Govt. of India, Ministry of Finance, New Delhi (1966).	Exclusive; Capacity planned 4.7 TPA—value Rs. 1 crore.
33.	Oxalic acid from bark of Ain tree (sponsored)	Industrial chemicals	M/s. Vidarbha Organic Chemical Industries, Dhote Bldg., Sitabuldi, Nagpur (1972).	Exclusive.
34.	Oxy-urea (sponsored)	Textile chemicals	M/s. Calico Chemicals & Plastics Division, Anik Chembur, Bombay-74 (1971).	Exclusive.
35.	Pentachlorophenol and other chlorophenolic compounds (sponsored)	Fungicides	M/s. Bombay Chemicals (P) Ltd., Bombay-1 (1969).	Exclusive.
36.	*p*-Phenetidine (sponsored)	Pharmaceuticals	M/s. Kabbur Industries Ltd., 3, Bastion Road, Bombay-1 (1971).	Exclusive.

1	2	3	4	5
37.	Potentiometric strip chart recorder	Recording instruments	M/s. Associated Instruments Manufacturers (India) (P) Ltd., New Delhi (1970).	Non-exclusive.
38.	Propoxyphene (sponsored)	Pharmaceuticals	M/s. Sarabhai Research Centre, Wadi Wadi, Baroda (1972).	Exclusive.
39.	Pulping of Kashmir softwoods (sponsored)	Dissolving grade pulp	M/s. J & K Minerals Ltd., The Bund, Srinagar (1971).	Exclusive.
40.	Quinacridone pigment (sponsored)	Dye intermediates	M/s. Lona Industries (P) Ltd., Ladivali, Dist. Kolaba (1970).	Exclusive.
41.	Removal of silica from black liquor (sponsored)	Paper industry	M/s. The Central Pulp Mills Ltd., 1183, Shivajinagar, Poona-4 (1971).	Exclusive.
42.	Styrene DVB-base cation exchange resin (65976, 98155, 98156)	Demineralization of liquids	(i) M/s. Industrial & Agricultural Engg. Co. (P) Ltd., 43, Forbes Street, Bombay-1 (1967).	Non-exclusive.

1	2	3	4	5
42.	(Continued)		(ii) M/s. Tulsi Industries, Poona-26 (1963). (iii) M/s. Bird & Co. Ltd., Chartered Bank Bldg., Calcutta-1 (1963).	
43.	Sulphacetamide and its sodium salt (sponsored)	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay-71 (1969).	Exclusive.
44.	Terpene G (sponsored)	Perfumery, Insecticides	M/s. Camphor & Allied Products, Bareilly (1968).	Exclusive.
45.	Terpineol	Perfumery	M/s. Dujodwala Industries, 14-1 Mile, Mathura Road, Faridabad (1972).	Non-exclusive.
46.	Titanium tetrachloride (sponsored)	Industrial chemicals	M/s. Travancore Titanium Products Ltd., Trivandrum-7 (1967).	Non-exclusive.
47.	Vat Golden Yellow G. K. (sponsored)	Dye intermediate	M/s. Amar Dye-Chem Ltd., P. B. No. 6471, Bombay-16 (1967).	Exclusive.
48.	Vitamin C	Pharmaceuticals	M/s. Hindustan Antibiotics Ltd., Pimpri, Poona (1966).	Non-exclusive.

TABLE II (B)

Following processes which have been mentioned in Table I are also licensed on non-exclusive basis to the following additional firms.

	Name of the process	Name of the party year of release
1.	Dihydroisojasmone	M/s. Sonebon Laboratories, Kottayam-5 (1970).
2.	Ferrites-Hard	M/s. Ajanta Enterprises, Mahalaxmi Industrial Estate, Gandhi Nagar, Bombay-13 (1971).
3.	β-Ionone	M/s. S. H. Kelkar & Co , Bombay (1964).
4.	Liquid rubber	M/s. Swastik Rubber Products Ltd., Poona-3 (1962).
5.	Nicotine sulphate	(i) M/s. Tabocco By-Products (P.) Ltd., Fort Narsarawpet, Guntur (1963). (ii) M/s. Coromandal Tobacco Bye-Products, P. O. Gannavaram, Andhra Pradesh (1970).
6.	Peach aldehyde	M/s. Sonebon Laboratories, Kottayam-5 (1970).
7.	Phthalates-dioctyl and dibutyl	M/s. Synthetic Organics, Amar Hill, Saki-Vihar Road, Bombay-72 (1971).
8.	Polyurethane printing rollers	(i) M/s. United Ink & Varinish Co., P. B. 6862, Vile Parle, Bombay-57 (1965). (ii) M/s. Dabholkar Bros., Poona-1 (1965).
9.	Rubber base contact adhesive	M/s. Bhagsons Paint Industries, 16-A DLF, Industrial Area, New Delhi-15 (1967).

10.	Sachets-Hot and Cold	(i) M/s. Vasant Industrial Corporation, 356, Great Nag Road, Nagpur-2 (1971).
		(ii) Andhra Pradesh Small Scale Industries Development Corporation Ltd,, Hyderabad-4 (1972).
11.	Sisal wax	(i) M/s. Shalimar Rope Works, 14, Netaji Subhash Road, Calcutta (1966).
		(ii) M/s. Deccan Paint & Chemical Industries, 289, G. T. Road, North Calcutta (1968).
		(iii) M/s. Industrial & Engg. Corpn., 84-B Bondel Road, Calcutta (1966).
12.	Thermistors	M/s. Adept Laboratories, Karve Road, Poona-4 (1963).

TABLE - III KNOW-HOW AVAILABLE

S. No.	Name of the process/product	Field of utilization	Capacity envisaged	Imported raw material	Capital (Rs. in lakhs) Fixed/ Working	Scale of development	Terms of release (Provisional)	Remarks
1	2	3	4	5	6	7	8	9
1.	Acetanilide	Drugs and dye intermediate	2000 TPA Rs. 1.73 crores	—	32/ 38	60 kg./batch	Available on request	Turnkey plant available through Project Engineer.
2.	Aniline	Organic intermediate	—	—	—	5 kg./hr. continuous	Available on request	Catalyst indigenous; Know-how and designs for 600 TPA plant available.
3.	L-Arabinose (CP)	Biochemical research	3 TPA Rs. 3.7 lakhs	—	1/ 1.6	500 g./batch	Non-exclusive; Premium Rs. 15,000; Royalty—2%.	Export potential.
4.	Atrazine	Herbicide	200 TPA of 50% Atrazine	Monoisopropylamine	3.45/ 13	1 kg./batch	Non-exclusive; Premium Rs. 3,500; Royalty—2.5%.	—
5.	Substitute for Bostik sealant	Oil resistant adhesives for fuel tanks	2,500 kg./annum	Nitrile rubber	*1.2/ 1.1	20 kg./batch	Non-exclusive; Premium Rs.2,500; Royalty—2%.	*Capacity assumed is 15 TPA. Plant and equipment will produce these adhesives only for 50 days and for the rest of the year will be used for production of other allied items. Released to one party.
6.	tert-Butyl catechol	Stabilizer and polymerization inhibitor for synthetic rubber	25 TPA Rs. 8 lakhs	tert-Butyl alcohol	1.5/ 3.4	10 kg./batch	Non-exclusive; Premium Rs. 10,000; Royalty—50 P./kg.	Released to one party.
7.	Butyl titanate	Insulating varnishes, special paints, catalyst	20 TPA Rs. 4.71 lakhs	Titanium tetrachloride	1.5/ 1.24	10 kg./batch	Non-exclusive; Premium Rs. 10,000; Royalty—1%.	Released to one party.

1	2	3	4	5	6	7	8	9
8.	Cadmium sulphide photocells	Instruments, photo-electric devices	30,000 cells/annum Rs. 7.5 lakhs	Indium, Woods metal	0.70/ 0.44	50-100 cells/batch	Non-exclusive; Premium Rs. 12,000; Royalty—30 P./cell.	Released to two parties.
9.	*Can lining composition	Lining cans for storing mineral oils, greases, food	50 TPA Rs. 5 lakhs	Nitrile rubber	1/ 1.5	4 kg./batch	Non-exclusive; Premium Rs. 5,000; Royalty—3 ½%.	*Based on nitrile rubber latex.
10.	Carbazole Dioxazine Violet pigment	Organic pigment	2 TPA Rs. 6 lakhs	Carbazole, Chloranil, Diethylsulphate	4/ 4.63	1 kg./batch	Non-exclusive; Premium Rs. 25,000; Royalty—1%.	—
11.	Cashewnut shell gum	Binder, thickening agent in food and pharmaceuticals	300 TPA Rs. 30 lakhs	—	11/ 4.5	1 kg./batch	Non-exclusive; Premium Rs. 10,000; Royalty—2%.	—
12.	Catechol	Organic intermediate	45 TPA Rs. 8.1 lakhs	—	3/ 2	7-8 kg./batch	Non-exclusive; Premium Rs. 10,000; Royalty—50 P./kg.	Released to one party.
13.	Cellulose powder	Chromotography, coating for electrodes, filter media	30 TPA Rs. 3 lakhs	—	1.5/ 0.60	1 kg./batch	Non-exclusive; Premium Rs. 5,000; Royalty—2.5%.	—
14.	Clofibrate	Drug	3 TPA Rs. 5.25 lakhs	*p*-Chlorophenol	2/ 1.06	1 kg./batch	Non-exclusive; Premium Rs. 10,000; Royalty—2%.	—
15.	Coating for oil filter papers	Oil filters	5 TPA coated paper* Rs. 0.80 lakhs	—	0.60/ 0.10 (For resin only)	6 kg./batch of coating resin	Non-exclusive; Premium Rs. 5,000 as technical aid.	*This will need 1,800 kg. resin. Released to one party.
16.	Costus root oil	Perfumery	300 kg. Rs. 4.8 lakhs	—	1.50/ 0.50	1 kg. of costus root oil	Non-exclusive; Premium Rs. 25,000 for costus root oil, additional Rs. 5,000 for fructose and chamazulene.	Kashmir costus roots yield more oil but less fructose. *From Punjab costus roots.
	*Fructose	Medicine	3045 kg. Rs. 1.8 lakhs	—		200 g./batch		
	Chamazulene	Cosmetics		—		4 g./batch		
17.	D. C. Recording polarograph	Polarographic analysis	25 units/annum Rs. 3 lakhs	—	*0.90/ 3	1 unit/batch	Non-exclusive; Premium Rs. 30,000 or Premium Rs. 15,000 +Royalty—3%.	*A capacity of 150 units per year has been assumed. The plant will produce 25 units in 2 months

1	2	3	4	5	6	7	8	9
17.	D. C. Recording polarogph	(Continued)						and for the rest of year will be used for making other instruments.
18.	Dihydroisojasmone and Peach aldehyde	Perfumery chemicals	—	—	—	—	Non-exclusive, Premium Rs. 5,000 as technical aid.	Released to two parties.
19.	Dimethylaniline	Dyestuff and explosives intermediate	600 TPA Rs. 60 lakhs	—	8/ 15	9 kg./batch	Non-exclusive; Premium Rs. 1 lakh for 600 TPA.	Released to one party.
20.	N-N-Dimethyl-biguanide-HCl (DMBG-HCL)	Anti-diabetic drugs	1 TPA 2.2 lakhs	Dicyandiamide, Dimethylamine	*2/ 2	500 g./batch	Non-exclusive; Premium Rs. 7,500; Royalty—2%.	*These estimates are based on a capacity of 12 TPA for each drug and it is assumed that the plant will produce each drug in one month and for the rest of 10 months will produce other products.
	Phenethylbiguanide -HCl (PEBG-HCl)		1 TPA 2.2 lakhs	Dicyandiamide, β-phenylethylamine	*0.90/ 2.95	100 g./batch		
21.	Direct reading spectrophotometer/ colorimeter	Biochemical research, spectroscopic analysis in visible range	100 units/annum Rs. 5.1 lakhs		*0.75/ 7.0	1 unit/batch	Non-exclusive; Premium Rs.10,000; Royalty–2%.	*A capacity of 1,200 units per annum is assumed. The plant will produce 100 units in one month and for the rest of the year will be used for making other instruments.
22.	Disperse dyes (Ind. Pat No. 116453)	The invention relates to a process for the production of new disperse dyes especially suitable for polyesters showing good affinity and sublimation fastness.		—	—	—	Non-exclusive; Premium Rs. 5,000; Royalty–1.5%.	Offering only right of exploitation of the patent (116453). Further developmental work, feasibility study and market survey will have to be done by the licncee.

1	2	3	4	5	6	7	8	9
23.	Dissolving grade pulp	Rayon, tyre cord	33,000 T/year of oven dried pulp	—	16.4 crores 1.6 crores	100 kg. of chip charge per batch	Negotiable	The process could be offered on turn-key basis through Project Engineering firms.
24.	Ethylene from ethyl alcohol	Organic intermediate	—	—	—	10 kg./hr.	Non-exclusive; Premium Rs. 5,000; Additional fee of Rs. 7,500 if designs and drawings are required.	Will be released as technical aid.
25.	Ethylenediamine	Bulk organic chemical	1000 TPA Rs. 1.6 crores	—	78/ 22	5-10 kg./day continuous	Non-exclusive; Premium Rs. 50,000; Royalty—1½%.	Turnkey plant available through Project Engineers.
26.	Ethylene-dichloride from ethyl alcohol	Solvent, organic intermediate	3000 TPA Rs. 80 lakhs	—	11/ 13.5	3 kg./hr.	Negotiable	—
27.	Ferric oxide-calcined and precipitated	Fine chemicals	*500 kg./annum of each grade		—	100 g./batch	Non-exclusive; Premium Rs. 3,000 as technical aid.	*This indicates requirement only.
28.	Ferrites-Hard	Electronics	30 TPA Rs. 6.7 lakhs	—	7.1/ 0.57	1 kg./batch	Non-exclusive; Premium Rs. 5,000; Royalty—2%.	Released to two parties.
29.	Ferrites-Soft	Electronics	30 TPA Rs. 8.6 lakhs	—	6.5/ 1.1	1 kg./batch	Non-exclusive; Premium Rs. 5,000; Royalty—2%.	—
30.	Fine chemicals	Know-how available for 200 laboratory chemicals, fine chemicals and reagents used in research.		—	—	—	Non-exclusive; Rs. 1.5 lakhs as package know-how fees for all 200 chemicals. Know-how is also available on these 200 chemicals groupwise.	
							(a) Chromotrographic adsorbents & standardizing agents	Rs. 1,00,000

1	2	3	4	5	6	7	8	9
30.	Fine chemicals (Continued)						(b) Industrial organic pharmaceutical intermediates (c) Analytical reagents, indicators & laboratory chemicals	60,000 90,000
31.	Flexible magnets	Refrigerator gaskets, toys, educational kits	10 lakhs feet (2 mm. thick & 1 cm. wide) Rs. 8 lakhs	Chlorinated polythene	4/ 1.91	500 g./batch extrusion of small pieces of 1-2 ft. length.	Non-exclusive; Premium Rs. 5,000; Royalty—2%.	For parties who have already bought NCL know-how on hard ferrites, no premium will be charged.
32.	Foundry core binder (sinol core binder)	Core binder in steel foundries for high dimensional accuracy	300 TPA Rs. 10 lakhs	Catalyst	0.90/ 2.0	5 kg./batch	Non-exclusive; Premium Rs. 5,000; Royalty—2%.	—
33.	Gaskets from coir pith	Gaskets	5.3 TPA Rs. 1.1 lakhs	Neoprene	1/ 0.32	Gaskets of 18" × 18" × 1/8" size.	Non-exclusive; Premium Rs. 10,000.	—
34.	D-Glucosamine hydrochloride (C. P.)	Biochemical research, pharmaceuticals	3 TPA Rs. 1.56 lakhs	—	1/ 0.90	250 g./batch	Non-exclusive; Premium Rs. 10,000; Royalty—2%.	Export potential.
35.	Glyceryl guaiacolate	Drug-expectorant, intestinal antiseptic	2 TPA Rs. 1.5 lakhs	Epichlorhydrin, Sodium nitrite	*3.5/ 4.1	Laboratory scale	Non-exclusive; Premium Rs. 25,000.	Capacity assumed is 24 TPA. Plant and equipment will produce this drug only in one month and for the rest of the year will be utilized for production of other chemicals.
36.	Gum arabic substitute	Adhesives	180 TPA Rs. 5.4 lakhs	—	1/ 1.4	3 kg./batch	Non-exclusive; Premium Rs. 5,000; Royalty—2%.	—
37.	Hexachloroethane (Ind. Pat. No. 92997)	Pyrotechnics, smoke screen, veterniary medicine, fluxing agent in foundries	300 TPA	—	3/ 2.5	10 kg./hr.	Non-exclusive; No premium; Royalty—2½%.	Released to one party.

1	2	3	4	5	6	7	8	9
38.	β-Ionone (Ind. Pat. No. 77225)	Perfumery chemical, intermediate for Vitamin A	—	—	—	Laboratory scale	Non-exclusive; No premium; Royalty—2½%.	Released to two parties.
39.	Linseed oil emulsion paint	Emulsion paints	15 TPA Rs. 1.5 lakhs	—	0.75/ 0.36	8.5 kg./batch	Non-exclusive; Premium Rs. 10,000; Royalty—2%.	—
40.	Microfilters	Industrial filtrations	60,000 cartridges per annum Rs. 2.7 lakhs	Melamine	1.5/ 0.58	1 kg./of pulp per batch	Non-exclusive; Premium Rs. 15,000; Royalty—10P. per. cartridge.	— —
41.	Monochloroacetic acid	Organic intermediate for weedicides, CMC, etc.	600 TPA Rs. 33 lakhs	—	6.5/ 9.5	600 g./batch	Non-exclusive; Premium Rs. 30,000.	Released to one Party.
42.	Monoethylaniline	Intermediate for explosives	150 TPA Rs. 20 lakhs	—	3/ 5	15 kg./hr.	Non-exclusive; Premium Rs. 25,000 Royalty—2.5%.	—
43.	Morpholine	Intermediate for rubber chemicals, textile chemicals, optical brightners, etc.	300 TPA Rs. 46 lakhs	Diethanolamine	6/ 14	400 g./batch	Non-exclusive; Premium Rs. 50,000.	—
44.	Nicotine sulphate (40% nicotine)	Insecticide	300 TPA of tobacco waste (13.54 TPA nicotine sulphate)	—	2.5/ 0.75	Plant designs for processing 1 T of tobacco waste.	Non-exclusive on zonal basis; Premium Rs. 2,000; Royalty—1%.	Released to four parties.
45.	Nitrile rubber (Medium grade)	Oil resistant rubber formulations, adhesives	2000 TPA	Acrylonitrile	60/ 45	7 kg./batch	Negotiable	Know-how for low and high grades is also under development.
46.	*p*-Nitrophenol	Intermediate for parathion and paracetamol	300 TPA Rs. 40 lakhs	*p-Nitrochlorobenzene	3.9/ 9.9	25 kg./batch	Non-exclusive; Premium Rs. 40,000.	Released to one party (* To be produced by HOC shortly).

1	2	3	4	5	6	7	8	9
47.	Optical whitening agent for synthetic fibres	Whitening agent for synthetic fibres	5 TPA Rs. 6 lakhs	Acenaphthene, Methylamine	2/ 1.6	1 kg./batch	Non-exclusive; Premium Rs. 30,000.	—
48.	Perfumery chemicals (a) Civetone & Dihydrocivetone (b) Dihydroambrettolide & Isoamrettolide (c) Exaltone (d) Exaltolide (e) Neo-Lavandulol	Perfumery	—	—	—		Know-how for these developed on laboratory scale. Detailed costing not done. These have limited home market but good export potential. Terms of release negotiable.	
49.	Phenacetin	Drugs	100 TPA Rs. 40 lakhs	*p*-Phenetidine	6/ 7.6	60 kg./batch	Non-exclusive; Premium Rs.60,000.	—
50.	Phenylacetic acid	Perfumery, Penicillin G.	60 TPA Rs. 7.8 lakhs	Sodium cyanide, Catalyst	4/ 2.2	1 kg./batch	Non-exclusive; Premium Rs.10,000; Royalty–$1\frac{1}{2}\%$	—
51.	Phenoxyacetic acid	Penicillin V	20TPA Rs. 3.6 lakhs	—	1.5/ 0.89	50 kg./batch	Non-exclusive; Premium Rs.10,000.	—
52.	Phthalate-butyl octyl	Plasticizer in non-electrical applications	3000 TPA Rs. 200 lakhs	—	10.5/ 25	45 kg./batch	Non-exslusive, Premium : 0-1500 TPA-Rs. 35,000 1501-2500 TPA-Rs. 70,000 2501-3000 TPA-Rs. 1,00,000	For those who have already bought DOP/DBP technologies from the NCL, only $\frac{1}{3}$ of these premia will be charged.
53.	Phthalate-dibutyl/ dioctyl	Plasticizers	3000 TPA Rs. 200 lakhs	—	10.5/ 25	60 kg./batch (DBP) 70 kg./batch (DOP)	Non-exclusive, Premium : Upto 1500 T-Rs. 35,000 1501 to 2500 T-Rs.70,000 2501 to3500T- Rs. 1 lakhs; 3501 to 9500 T-Sliding scale from Rs. 1 3 lacs; to Rs. 2.4 lakhs. Above 9500 T-Rs. 2.5 lakhs.	Released to three Parties.

1	2	3	4	5	6	7	8	9
54.	Phthalate-dimethyl/ diethyl	Plasticizer	1500 TPA Rs. 105 lakhs		6/ 31.3 (DMP) 6/ 30 (DEP)	60 kg./batch (DMP) 40 kg./batch (DEP)	Non-exclusive; Premium Rs. 2 lakhs.	Released to one party.
55.	Polyurethane coatings	Leather, rubber, wood, glass, Nylon fabrics	10,000 liters per annum Rs. 1.6 lakhs 15 TPA Rs. 2.4 lakhs	Toluene disocyanate Adipic acid, Toluene diisoyanate	0.25/ 0.80 0.38/ 0.53	7 kg./batch	Non-exclusive; Premium Rs. 10,000; Royalty 1%.	—
56.	Potentiometric strip chart recorder	Instrument for use in research and industry	1200 units Rs. 1 crore	Chopper	4/ 9	One unit per batch	Non-exclusive; Premium Rs.10,000; Royalty upto 1,000 pieces—3%; above 1,000 pieces—2%.	—
57.	Radiosonde thermistors	Meterology	30,000 pieces Rs. 4.5 lakhs	Platinum foil	1.2/ 0.79	100 pieces/batch	Non-exclusive; Premium Rs.25,000; Royalty—5%	—
58.	Recovery of pyridine bases from their aqueous solutions (Ind. Pat. No.111311)	20% Aqueous pyridine base solutions are produced in manufacture of soluble vat dyes. Process involves separation & extraction of pyridine bases by suitable solvent.			—	Laboratory scale	Non-exclusive; Premium Rs. 10,000.	—
59.	Rubber blowing agent (Dinitroso-pentamethylene-tetramine)	Rubber chemical	30 TPA Rs. 3 lakhs	Sodium nitrite	0.60/ 0.52	10 kg./batch	Non-exclusive; Premium Rs.5,000 as technical aid.	Released to one party.
60.	Rubber reclaiming agent	Rubber chemical	100 TPA Rs. 10 lakhs	—	2.7/ 2.2	20 kg./batch	Non-exclusive; Premium Rs. 30,000.	Released to one party.
61.	Rubbrized cork sheets from cork waste	Gaskets	5.3 TPA Rs. 1.1 lakhs	Neoprene	1/ 0.32	Sheets of 6″ × 9″ × ⅛″ size	Non-exclusive; Premium Rs. 10,000.	
62.	Sachets—Hot and Cold	Substitute for Hot water bag/Ice bag	1 lakh sachets Rs. 1.5 lakhs	—	0.35/ 0.33	20 cm. × 10cm. size sachets	Non-exclusive; Premium Rs.2,000; Royalty 5 paise per sachet.	Released to three parties.

1	2	3	4	5	6	7	8	9
63.	Sealants for canvas bags	Sealant adhesive	500 kg. Rs. 22,000	*Nitrile rubber, Sulphur, Thiourea, Methyl ethyl ketone and resin	0.28/ 0.055	4.5 kg./batch	Non-exclusive; Premium Rs.2,500 as technical aid.	*Since the quantities of these raw materials are small they could be procured in market.
64.	Simazine	Herbicide	100 TPA (92% simazine) or 184 TPA (50% simazine) Rs. 35 lakhs	—	3.45/ 14.8	1 kg./batch of 50% simazine	Non-exclusive; Premium Rs. 3,500; Royalty—2.5%.	—
65.	Sisal wax	Cosmetics, polishes	—	—	—	2kg./batch	Non-exclusive; Royalty—2 ½%.	Released to four parties.
66.	Mannitol/sorbitol from cane sugar	Pharmaceuticals (Mannitol) Pharmaceutical syrups, Humectant (Sorbitol)	300 TPA (48.75 TPA Mannitol, 251.25 TPA 100% Sorbitol or 359 TPA of 70% Sorbitol) Rs. 33 lakhs	Catalyst (Raney Nickel)	15/ 7	5kg./batch of sugar	Non-exclusive; Premium Rs.50,000.	—
67.	70%Sorbitol from dextrose monohydrate	Pharmaceuticals, Vit. C synthesis	500 TPA Rs. 30 lakhs	Catalyst (Raney nickel)	12/ 6.7	75kg./batch of dextrose monohydrate	Non-exclusive; Premium upto 500 T-Rs.50,000; 501-600T-Rs.55,000; 601-700 T-Rs.60,000; 701-800T-Rs.65,000; 801-900 T-Rs.70,000; 901 T & above-Rs. 75,000.	
68.	Staple pins adhesive	Adhesive for staple pins	5TPA Rs. 80,000/-	—	0.5/ 0.09	10 kg./batch	Non-exclusive; Premium Rs.6,000 as technical aid.	—
69.	Tamarind kernel powder-phosphate and borate	Textile sizing, substitute for hydrolyzed maize starch	300 TPA Rs. 5.6 lakhs	—	1.4/ 1.28	5 kg./batch	Non-exclusive; Premium Rs. 5,000; Royalty—2%.	—
70.	Terpineol	Perfumery	100 TPA Rs. 10 lakhs	α-Pinene	3.25/ 3.66	1 kg./batch	Non-exclusive; Premium Rs.35,000.	Released to one party.

1	2	3	4	5	6	7	8	9
71.	Theophylline, Aminophylline, Caffeine	Drugs (Caffeine also used in beverages)	20 TPA of each Total 60 TPA Rs. 44 lakhs	1,3-Dimethylurea, Raney nickel, Sodium nitrite, Sodium cyanide, Dimethylsulphate, Ethylenediamine	9/ 14	Theophylline, Caffeine-1kg./ batch, Aminophylline-500 g./batch	Non-exclusive; Premium-Rs.25,000; Royalty—1½%.	—
72.	Thermistors	Temperature measurement and control, electronic devices, etc.	—	—	—	1 kg./batch	Premium Rs.10,000 as technical aid.	Released to three parties. —
73.	Thioglycollic acid	Cosmetics, catalyst	50 TPA (80%) Rs. 10.13 lakhs	Isopropyl ether	1.6/ 3.4	1 kg./batch	Non-exclusive; Premium Rs.25,000 or Premium Rs.10,000 + Royalty—1 %.	—
74.	2,2,4-Trimethyl-6-ethoxy-1, 2-dihydroquinoline	Rubber antioxidant	100 TPA Rs.22 lakhs	*p*-Phenetidine	8/ 7.2	1 kg./batch	Non-exclusive; Premium Rs.50,000.	—
75.	Vitamin B_6	Drug	10 TPA Rs. 42.5 lakhs	Sodium cyanide, Phosphorus pentoxide, 2-Butene 1,4 diol, Paraformaldehyde	19/ 11.75	Laboratory scale (1 kg./batch in progress)	Non-exclusive; Premium Rs. 50,000; Royalty—1%.	—
76.	Vitamin C	—	—	—	—	—	This is an important drug. Process released to M/s. Hindustan Antibiotics Ltd. (HAL), who are setting up a 125 TPA plant. The process could also be offered through NRDC to other interested parties in collaboration with HAL.	
77.	Xanthates-Potassium ethyl (PEX) and Potassium amyl (PAX)	Froth-flotation	150 TPA Rs. 11 lakhs (PEX & PAX)	—	2.5/ 3 (PEX)	500 g./batch	Non-exclusive; Premium Rs.40,000.	

COST-BENEFIT ANALYSIS FOR 1971–72

ACTUAL EXPENDITURE

	Rs. in lakhs
1. *Recurring expenditure*	
(a) Pay and allowances	46.615
(b) Chemicals & apparatus	10.377
(c) Maintenance & contingencies	13.420
Total (1)	70.412
2. *Capital expenditure*	
(a) Building	0.260
(b) Equipment & apparatus	8.209
(c) Services & miscellaneous	3.955
Total (2)	12.424
3. *Pilot plant expenditure*	
(a) Pay and allowances	1.727
(b) Raw materials and miscellaneous	0.800
(c) Equipment, buildings and services	0.318
Total (3)	2.845
Total CSIR expenditure (1+2+3)	85.681
4. *Expenditure on account of sponsored projects*	8.676
Total	94.357

BENEFITS

Receipts

1. Premia and royalties (Figure refers to calender year 1971)	1.422
2. Receipts on account of sponsored projects	8.676
3. Analytical and testing charges, technical aid, etc.	0.329
4. Institutional consultancy (CSIR share)	0.384
5. FCP (Fine Chemicals Project) sales	2.672
6. Miscellaneous receipts	1.976
Total	15.459

Indirect benefits

1.	Total number of processes in production	51
2.	Value of production based on NCL know-how during 1971-72 (Rs. in lakhs)	471.20
3.	Estimated saving in foreign exchange on account of above production (Rs. in lakhs)	188.48
4.	Total cumulative value of production upto March 1972 (Rs. in lakhs)	1,048.01
5.	Research papers published during 1971-72	111
6.	Doctorate and Masters degrees awarded during 1971-72	20
7.	No. of recognized guides for Doctorate and Masters degrees	38
8.	Patents	
(a)	New patents filed	8
(b)	Total patents in force	36
(c)	Patents which are being commercially exploited	20
9.	Total number of technical enquiries on which information was supplied	2,500

APPENDICES

1. SPECIAL EQUIPMENT FACILITIES AVAILABLE

	Name of Equipment/Instrument	*Function*
1)	Abrasion resistance machine	Elastomer testing
2)	Analogue computer	—
3)	Autoclaves-capacity ranging 1 to 75 litres, working pressures 70 to 300 atm., working temperature-300°-500°	High pressure reactions
4)	BET apparatus	Surface area measurements
5)	Bleaching equipment-capacity 100 kg. chip charge,	Bleaching of pulp
6)	Chromatographic fraction collector-automatic	Chromatographic analysis
7)	Cobalt 60 (Gamma Cell 220) unit	Chemical reactions induced by radiations
8)	Conductivity measurement set up -thermally stimulated	Measurement of thermal conductivity
9)	Counter current extraction unit	Solvent extraction
10)	Derivatograph	Differential Thermal Analysis (DTA); Thermo Gravimetric Analysis (TGA) and Differential Thermo Gravimetric Analysis (DTGA)
11)	Distillation column-all glass, steam heated, bubble cap (50 lit. capacity)	Fractionation of common solvent mixtures
12)	Distillation unit-all glass, electrically heated (20 lit. capacity)	Vacuum distillation at low temperature

13)	Distillation unit-all glass, steam heated (100 lit. capacity)	Solvent distillation
14)	Electron diffraction camera (Finch type)	Studies in crystal growth and structure of thin films in solid state
15)	Electron microscope (EM-3 model)	Studies in the morphology and particle size of every small samples which can not be observed by optical microscope (below $\sim$ 1000 Å)
16)	Evaporator-all glass, climbing film type	Concentration of heat sensitive materials
17)	Flame photometer	Analysis of trace elements eg. Na, K, Ca, etc.
18)	Fractionation column-bubble cap, vaccum	Fractionation
19)	Gamma rays spectrometer-single channel	Gamma rays spectral analysis
20)	Hall coefficient measurement unit	Measurement of Hall coefficients-Magnetic field 6000 gauss, temperature range −180° to 150°
21)	Heats of mixing apparatus (Twin type calorimeter model)	Measurement of heats of mixing of solutions (aqueous and non-aqueous)
22)	Huppert muffle furnace	High temperature reactions upto 1200°
23)	Injection moulding machine	Plastics
24)	IR model 137E-PE	Spectral analysis (in the region 2.5-15 microns)
25)	IR model 221-PE	Spectral analysis (in the region 2-35 microns)
26)	Light scattering photometer	Determination of molecular weight of high polymers

27)	Liquid-liquid extraction unit-all glass, steam heated (20 lit. capacity)	Pilot plant work on liquid-liquid systems
28)	Microbalance-high temperature high vacuum	Measurement of the minute vapour pressure of the refractory materials
29)	Microphotometer (Lirepho-2 model)	Measurement of intensity of rings sports of X-ray and electron diffraction patterns for the structure analysis
30)	Mooney elasticity machine	Elastomer testing
31)	Mossabauer spectrometer	Nuclear resonance absorption spectral analysis
32)	NMR-model A-60 Varian	Spectral analysis (in the region 0-16 [δ])
33)	NMR-model T-60 Varian	Spectra of 1_H and 19_F compounds, spin decoupling and variable temperature studies
34)	Okay furnaces	High temperature reactions upto 1400°
35)	Padding mangle	Dyeing studies
36)	Photochemical reactor	Studied in photochemistry
37)	Pigment Muller	Pigment testing
38)	Podbielniak fractionation unit	Fractional distillation
39)	Polarograph	Study of reaction mechanisms, Standardization of electrolytic reduction
40)	Projection microscope	Particle size determination of solid substances
41)	Pulp making unit (2 kg./charge)	Pulp making

42)	Rayleigh interference refractometer	Differential refractive index measurements
43)	Rubber mixing mill (laboratory type)	Rubber compounding
44)	Solid-liquid extraction unit-all glass, steam heated (20 lit. capacity)	Extraction of solids with volatile solvents
45)	Spectrograph	Inorganic analysis (elements)
46)	Spray dryer	Spray drying
47)	Tensile testing unit	Elastomer testing
48)	Thermogravimetric unit	Thermogravimetric study of materials in semi-micro quantities (upto 10-30 mgm.) in vacuum or any gas atmosphere
49)	Ultracentrifuge-model E-analytical	Molecular weight and homogeneity studies on proteins
50)	Ultracentrifuge (Beckman Spinco model L)-preparative	Isolation and separation of cell, organocells and micromolecules
51)	UV/Vis/Nir-PE-350	Spectral analysis (in the region 185-2.7 μ-microns)
52)	Vacuum coating unit	Gives vacuum 10^{-5} mm. of Hg, fitted with high tension ionization cleaning equipment
53)	Vacuum coating unit-model No. 12A4	Provided with three different heating rates, temperature range -180° to 100°
54)	Verneuil furnace	Growing single crystals of materials with high melting point
55)	Viscometer-rotational	Viscosity measurements of monomolecular films
56)	Viscose making and spinning unit (Emil Blaschke) (2 kg. capacity)	Viscose making and spinning

57)	Weissenberg Gonimeter	Studies in single crystal structures
58)	Weissenberg Gonimeter-integrating	Single crystal structure studies at room and low temperatures
59)	Xenotest equipment	Testing light fastness of dyed samples
60)	X-ray diffraction unit (Philips PW-1010 and Philips PW-1008 model)	X-ray powder patterns
61)	Yarn testing equipment	Mechanical testing of yarn

2. SERVICES TO INDUSTRIES, RESEARCH INSTITUTES, UNIVERSITIES, ETC.

2.1 *Supply of cultures*

During the year under report, 784 cultures from the national collection of industrial microorganisms (NCIM) were supplied free of charge to various institutions in India and abroad.

2.2 *Analytical services*

A large number of analyses were done on payment basis for universities, research institutes, Govt. departments, private parties, etc.

The following analyses were carried out :

Microanalysis	..	120
NMR	..	63
IR	..	130
UV	..	7
VPC/GLC	..	4
Mass spectra	..	186
X-ray powder patterns	..	11

2.3 *Technical aid involving ad-hoc experimental, instrumental, engineering and glass blowing services*

1.	Identification studies for coating on scratch sensitive paper	M/s. Adept Laboratories, Poona-4.
2.	Drying of disperse dye slurry in spray dryer	M/s. Arlabs Ltd., Bhor.
3.	Analysis of heat treating salt	M/s. Kwality Papers, Kolhapur.
4.	Analysis of sodium silico fluoride	M/s. The Dharamsi Morarji Chemical Co. Ltd., Bombay.
5.	Analysis of mangenese dioxide (2 samples)	Industrial Toxicological Research Centre, Lucknow.
6.	Facilities were given for conducting experiments on catalytic reduction of aliphatic ester	M/s. Cyanamid India Ltd., Bombay.
7.	Supply of CdS photoconductive cells	M/s. The Oriental Science Apparatus Workshop, Haryana.
8.	Analysis of vinyl chloride acetate copolymer	M/s. Calico Chemicals and Plastics Divn., Bombay.
9.	Determination of particle size and distribution in PVC resin	—do—
10.	Analysis of pyritic carbonaceous clay samples (2 Nos.)	Petrography and Mineral Chemistry Laboratory, Ahmedabad.
11.	Pure and stoichiometric barium titanate prepared and supplied	M/s. Tempo Semiconductors, Bombay.
12.	Analysis of sample used in fire extinguishing equipments	National Metallurgical Laboratory, Jamshedpur.
13.	Analysis of a mineral sample	The Sikkim Mining Corpn., Sikkim.

14	Analysis of vacuum salt		The Octori Dept., Poona Municipal Corpn., Poona.
15.	Repair and servicing carried out by the instrumentation group :		
	(i)	Flame photometers (2 Nos.)	Soil Survey Office, Poona and Soil Testing Lab., Dhulia.
	(ii)	pH Meters (3 Nos.)	Soil Testing Lab., Parabhani and Dapoli Regional Cane Research Centre, Kolhapur.
	(iii)	Craftman colorimeter	Soil Survey Office, Dhulia.
	(iv)	Conductivity bridges (2Nos.)	Soil Testing Lab., Parabhani.

2.4 *Training*

	Name of the trainee and place	*Field of training*
1.	Major N. V. Kamat, Military Dental Centre, Jabalpur.	Electrophoresis and immunological techniques
2.	Mr. S. P. Misra, Agricultural Univ., Pantnagar (UP) and Mrs. V. T. Surve, Marathwada Univ., Aurangabad.	Mirobiological techniques with special reference to maintenance of cultures
3.	Mr. A. D. Pawade and Mr. B. R. Korde, Public Health Institute, Nagpur.	Microbiological assay of Vitamins B_1 and B_2
4.	Prof. S. H. Chavan, Shivaji Univ., Kolhapur.	IR spectroscopy
5.	Mr. K. Jadhav, Mr. V. S. Kulkarni Shivaji Univ., Kolhapur and Dr. P. K. Aditya, Regional Engineering College, Kurukshetra.	Instrumentation
6.	Mr. S. E. Pathak, Shivaji Univ., Kolhapur.	Thermogravemetric measurement techniques

7.	Dr. S. C. Jain, Muzaffar Inst. of Technology, Muzaffarnagar.	NMR, IR, and Mass spectral techniques
8.	Mr. C. J. Shishoi, L. M. College of Pharmacy, Ahmedabad.	Spectroscopic analysis

3. SPONSORED PROJECTS

3.1 *Sposnored projects concluded during* 1971-72

1.	Calcium silicate from wollastonite	M/s Chemicals India Ltd., Udaipur.
2.	High temperature chlorination of bauxite residue	M/s. The Dharamsi Morarji Chemicals Co. Ltd., Bombay.
3.	Constitution of lac	Indian Lac Cess Committee, Ranchi.
4.	Ketene from acetone	M/s. Sudarshan Chemical Industries (P) Ltd., Poona.
5.	*p*-Nitroacetophenone	M/s. Dey-Se-Chem. Ltd., Calcutta.
6.	Oxalic acid from bark of Ain tree	M/s. Vidharbha Organic Chemical Industries, Nagpur.
7.	Oxy-urea	M/s. Calico Chemicals & Plastics Divn., Bombay.
8.	*p*-Phenetidine	M/s. Kabbur Industries (P) Ltd. Bombay.
9.	Propoxyphene	M/s. Sarabhai Research Centre, Baroda.
10.	Pulping of Kashmir soft-woods	M/s. J. & K. Minerals Ltd., Srinagar.
11.	Removal of silica from black liquor	M/s. The Central Pulp Mills Ltd., Poona.

12.	Wood phenolics (PL-480 scheme)	U. S. Dept.of Agriculture, Washington.

3.2 *Sponsored projects newly unertaken during* 1971-72

1	Conversion of bauxite into anhydrous aluminium chloride	M/s. Tata Chemicals Ltd., Bombay.
2.	High temperature chlorination of bauxite residue	M/s. Dharamsi Morarji Chemicals Co. Ltd., Bombay.
3.	Chlorohydroxyquinoline	M/s. Biological Evans Ltd., Hyderabad.
4.	Flavonoids, tannins, stilbenes, lignans and quinones in some Indian forest trees (PL-480 scheme)	U. S. Dept. of Agriculture, Washington.
5.	Oxalic acid from bark of Ain tree	M/s. Vidharbha Organic Chemical Industries, Nagpur.
6.	Synthesis of potential pharmacologically active substances	M/s. Sarabhai Research Centre, Baroda.
7.	Propoxyphene	M/s. Sarabhai Research Centre, Baroda.
8.	Solvent extraction of sandalwood oil	Govt. Sandalwood Oil Factory, Bangalore.
9.	Thermosetting resin for industrial laminates	M/s. Formica India Co., Poona.
10.	Production of xylit	M/s. Unichem Laboratories Ltd., Bombay.

3.3 *Sponsored projects continued from* 1970-71

1.	Arylic acid/acrylaes from acrylonitrile	Indian Petrochemicals Corpn. Ltd., Baroda.
2.	Cationic dyes for acrylic fibre	M/s. Sahyadri Dyestuffs & Chemicals (P) Ltd., Poona.

3.	Composite drug research scheme on Indian medicinal plants	Ministry of Health, Govt. of India, New Delhi.
4.	Glyceryl-α-mono-para-aminobenzoate	M/s. Indian Schering Ltd., Bombay.
5.	Extraction of mohrpine and other alkaloids from lanced poppy-straw	Ministry of Finance, Govt. of India, New Delhi.
6.	Perfumery products based on Δ^3-carene	M/s. Camphor & Allied Products, Bareilly.
7.	Perfumery products based on logifolene and isolongifolene	M/s. Camphor & Allied Products, Bareilly.
8.	Propylene oxide	Indian Petrochemicals Corpn. Ltd. Baroda.
9.	Production of rutile titania from Indian ilmenite	M/s. Asian Paints, Bombay.

3.4 *Review of sponsored work*

Period	No. of sponsored schemes completed during the period	R & D inputs (Rs. in lakhs)	No. of items in production	Value of production (Rs. in lakhs)
Upto 1960	—	0.18	—	—
1960-65	10	5.98	2	1.88
1965-70	44	36.45	9	155.91
1970-71	10	6.68	15	92.07
1971-72	12	8.68	15	119.79
TOTAL	76	57.97	15	369.65

Note :- Out of the total 76 sponsored schemes completed upto March 1972, commercial production is establised on 15 products/processes. 28 are in various stages of commercial implementation (awaiting production), 18 are of basic nature wherein no production is envisaged. In case of remaining 15 schemes, commercial implementation has been suspended by sponsors for various reasons not necessarily connected with the R & D work done in the NCL.

4. TECHNOLOGY TRANSFER 1971-72

4.1 *Demonstrations*

Following processs were demonstrated :

	Process	*Name of the party*
1.	Substitute for Bostik sealants	M/s. Swastik Rubber Products Ltd., Poona
2.	Ferrites-Hard	M/s. Ajanta Enterprises, Bombay
3.	Phthalates-dioctyl and dibutyl	M/s. Synthetic Organics, Bombay
4.	Sachets - Hot and Cold	(i) M/s. Vasant Industrial Corporation, Nagpur (ii) M/s. Thermo Chem Laboratories, Pimpri,Poona

4.2 *Processes leased out during* 1971-72

	Process	*Party*	*Remarks*
1.	Benzoic acid (IP) from methyl benzoate available with IPCL	M/s. Indian Petrochemicals Corpn. Ltd., P.O. Jawaharnagar, Baroda.	Non-exclusive; Premium-Rs. 40,000/-; Agreement in progress.
2.	Substitute for Bostik sealants	M/s. Swastik Rubber Products Ltd., Poona.	Non-exclusive; Premium-Rs. 2,000/-; Royalty-2%.

3. Dimethylaniline	M/s. Sahyadri Dystuffs & Chemicals (P) Ltd., Poona.	Non-Exclusive; Premium-Rs. 50,000/- for 300 TPA capacity; Rs. 1 lakh for 600TPA capacity; Agreement in progress.
4. Ferrites - Hard	M/s. Ajanta Entreprises, Mahalaxmi Industrial Estate, Bombay-13.	Non-Exclusive; Premium-Rs. 5,000/-; Royalty-2%.
5. Monochloroacetic acid	M/s. Hico Products (P) Ltd., Bombay.	Non-Exclusive; Premium-Rs. 30,000/-; Agreement in progress.
6. Phthalates-dioctyl and dibutyl	M/s. Synthetic Organics, Amar Hill, Saki-Vihar Rd., Bombay-72.	Non-exclusive; Premium-Rs. 5,000/-; Royalty-1%.
7. *p*-Nitrophenol	M/s. Hindustan Organic Chemicals, Rasayani.	Non-Exclusive; Premium-Rs. 40,000/-; Agreement in progress.
8. Sachets-Hot and Cold	i) M/s. Vasant Industrial Corporation, 356, Great Nag Rd., Nagpur-2.	Non-exclusive; Premium-Rs. 2,000/-; Royalty-5 paise per sachet.
	ii) M/s. Thermo Chem Laboratories, Pimpri, Poona.	—do—
	iii) Andhra Pradesh Small Scale Industries Development Corpn. Ltd., Hyderabad-7.	—do—
9. Terpineol	M/s. Dujodwala Industries, Faridabad.	Non-exclusive; Premium-Rs.35,000/-.

4.3 *Processes assigned to NRDC during* 1971-72

(1) L-Arabinose (CP)
(2) Atrazine
(3) Benzoic acid (IP) from crude methyl benzoate
(4) Can lining composition (based on nitrile rubber latex)
(5) Carbazole Dioxazine Violet pigment
(6) Costus root oil, fructose and chamazulene
(7) D. C. Recording polarograph
(8) Dimethylaniline
(9) Direct reading spectrophotometer/colorimeter
(10) Double boiled linseed oil substitute
(11) Ethylenediamine
(12) Fine chemicals project
(13) Gaskets from coir pith
(14) D-Glucosamine hydrochloride (CP)
(15) Glyceryl guaiacolate
(16) Linseed oil emulsion paint
(17) Monochloroacetic acid
(18) Optical whitening agent for synthetic fibres
(19) Phenacetin
(20) Phenyl acetic acid
(21) Phthalate—butyl octyl
(22) Recovery of pyridine bases
(23) Rubberized cork sheets from cork waste
(24) Simazine
(25) Sorbitol/Mannitol from cane sugar
(26) Sorbitol 70% from dextrose monohydrate
(27) Thioglycollic acid
(28) Vitamin B_6
(29) Xanthates-Potassium ethyl and potassium amyl

4.4 *Premia and Royalty received by NRDC through NCL processes during the year* 1971-72

S. No.	*Name of the process*	*Premia* (Rs.)	*Royalty* (Rs.)
1.	Stable gel for hot and cold sachets	8,000	—
2.	Butyl titanate	2,000	—
3.	Dioctyl phthalate	5,000	—
4.	Rubber reclaiming agent	7,000	—
5.	*p*-Menthane hydroperoxide	10,000	—
6.	Dimethylaniline	5,000	—
7.	*p*-Nitrophenol	40,000	—
8.	Acetanilide	20,000	—
9.	Terpineol	5,000	—
10.	Substitute for Bostik sealants	2,500	—
11.	Can sealing composition	—	5,122.99
12.	Nicotine sulphate	—	2,358.08
13.	β-Ionone	—	21,675.63
14.	Sisal wax	—	82.50
15.	Vapour phase chromatograph	—	28,193.75
16.	4-Hydroxycoumarin	—	6,272.38
17.	Catechol	—	248.75
18.	tert-Butylcatechol		
19.	Bacterial diastase	—	3.00
20.	Antipriming composition	—	431.94
	Total	1,03,500	64,388.60

5. SEMINARS AND LECTURES

5.1 *Short course on 'Interpretation of mass spectra'*

An American Chemical Society's five day course on 'Interpretation of mass spectra' which was sponsored by National Science Foundation (NSF), USA and National Council of Scientific Education (NCSE), New Delhi, was conducted at NCL from 14th to 19th June 1972. Thirty scientists from various research organizations, universities, private parties, etc. participated in the course.

5.2 *Post—IUPAC symposium*

A post-IUPAC symposium on 'The art and science of organic synthesis in the field of natural products,' was held at NCL on 15th and 16th Feb. 1972. The symposium was attended by eighteen eminent internationally known scientists in their fields. Notable amongst them were :

Prof. D. H. R. Barton-(Nobel Laurate) Imperial College, London, UK; Prof. A. J. Birch, The Australian National Univ., Canbera; Prof. H. Schmid, Univ. of Zurich, Switzerland and Prof. G. Gurisson, Univ. Louis Pasteur, Institute De Chimie, Strasbourg, France.

5.3 *Lectures*

In addition to the talks by several NCL scientists in weekly seminars, the following lectures were delivered by visiting scientists :

1.	Prof. A.F. Bocakov, Zelinsky Institute of Organic Chemistry, Moscow, USSR.	Synthesis of polysaccharides.
2.	Prof. A. Nickon, The John Hopkins University, Maryland, USA.	Stereochemistry of caryophyllene cyclizations.
3.	Dr. M.J. Thirumalachar, Hindustan Antibiotics Ltd., Pimpri, Poona-18.	Antifungal antibiotics in the treatment of fungal diseases of humans.
4.	Prof. Don C. De Jongh, Wayne State University, USA.	Some applications of mass spectrometry in natural products chemistry.
5.	Dr. P. Narasimha Rao, Southwest Foundation for Research and Education, San Antonio, Texas, USA.	Isotope lebelling of specific centres in steroid molecule.
6.	Prof. M. Heidelberger, New York University School of Medicine, New York, USA.	Immunological cross-reactivity : Bane or benefit.
7.	Prof. A. R. Verma, National Physical Laboratory, Delhi.	Physical standards.
8.	Dr. Nobusuke Kawano, Nagasaki University, Nagasaki, Japan.	Chemistry of pseudoanisatin.
9.	Dr. P. Koteswara Rao, Osmania Medical College, Hyderabad.	Chemistry and crime.

10.	Dr. D. B. Johnson, US Department of Agriculture, Washington D. C., USA	Directions and progress of US forest service research in the utilization of forest products wastes.
11.	Dr. J. W. James, Aspro-Nicholas Ltd., UK.	The synthesis and CNS activity of a group of basically substituted barbiturates.
12.	Prof. A. T. James, Unilever Research Laboratory, UK.	Some aspects of lipid and steriod biosynthesis in plants.
13.	Prof. Henry Feuer, Purdue University, Lafayette (Ind.), USA.	The alkyl nitration of active methylene compounds.
14.	Prof. A. Charlesby, F. R. S., Royal College of Military Science, London.	Modification of polymers with radiations.
15.	Dr. R.T. Parfitt, Nicholas Research Institute, UK.	Some aspects of medicinal chemistry of 6, 7-benzomorphans.
16.	Prof. Roy S. Whistler, Purdue University, USA.	Chemistry and biochemistry of sugars, sugar nucleosides and sulphur in sugar ring.
17.	Dr. F. Scheinmann, University of Salford, UK.	The structure and total synthesis of metabolites from guttiferae.
18.	Dr. Mihaly Nogradi, Technical University, Budapest, XI, Gellert-4, Hungary.	A conformational study of eight membered ring compounds by nuclear magnetic resonance.
19.	Dr. J. H. P. Tyman, Brunel University, UK.	Some work on hydrogenation with nickel catalysts.
20.	Prof. M. Kocor, Polish Acadamy of Sciences, Poland.	New sesquiterpenic lactones from *lactarius rufus*.

21.	Dr. K. S. Rajan, Illinois Institute of Technology, Chicago, USA.	Metal chelate equilibria in solution.
22.	Dr. Daniel L. Klayman, Walter Reed Army Institute, Washington D. C., USA.	Recent work in thiourea chemistry.

5.4 *Following NCL scientists delivered lectures at various institutes, universities, colleges, etc.*

	Name of the scientist	*Subject*	*Venue*
(1)	Dr. L. C. Anand	Chemistry of polymers (6 lectures)	University of Poona, Poona
(2)	Dr. J. L. Bose	The availability of hydroxyl groups of native and mercerized cotton cellulose	Southern Regional Lab., U. S. D. A., New Orleans, USA
(3)	Dr. K. G. Das	Mass spectrometry	University of Poona, Poona
(4)	Dr. A. B. Deshpande	Chemistry of polymers (2 lectures)	University of Poona, Poona
(5)	Dr. N. D. Ghatge	Condensation of polymers and catalyst in polymerization	—do—
(6)	Dr. V. N. Gogte	Chemistry of steroids (15 lectures)	S. P. College, Poona
(7)	Dr. S. Gundiah	Chemistry of polymers (2 lectures)	University of Poona, Poona
(8)	Dr. S. L. Kapur	i) Recent developments in paints	Technical Institute, Fergusson College, Poona
		ii) Chemistry of polymers (2 lectures)	University of Poona, Poona
(9)	Mr. V.S. Krishnamachar	Microbiology (4 lectures)	Ahmednagar College, Ahmednagar

(10)	Mr. P. S. Kulkarni	Physical chemistry	Wadia College, Poona
(11)	Dr. P. M. Nair	Study of conjugation by NMR	NMR workshop-Indian Institute of Science, Bangalore.
(12)	Dr. C. R. Narayanan	i) Restricted rotation around some carbon-carbon signle bonds	IIT, Kanpur
		ii) Restricted rotation around some carbon-oxygen and carbon-nitrogen single bonds	CDRI, Lucknow
		iii) Conformation of the primary hydroxyl group in terpenoids and conformation of the carbonyl group in secondary amides	Shivaji University, Kolhapur
(13)	Dr. L. M. Pant	Crystallographic work at NCL	Osmania University, Hyderabad
(14)	Dr. A.P.B. Sinha	Ferrites and related materials (4 lectures)	Winter School-Solid State Chemistry, IIT, Kanpur
(15)	Dr. C. SivaRaman	Chemistry of immunoglobulins	University of Poona, Poona
(16)	Mr. S.R. Srinivasan	Phenolic resin, Amino resin, Polyamides and Polyesters	University of Poona, Poona
(17)	Dr. P.R.SubbaRaman	Solvent extraction in analytical chemistry	Convention of Chemists, Bombay
(18)	Dr. S. P. Vernekar	Plasticizers and Antioxidants	University of Poona, Poona

6. STAFF STRENGTH AS ON 31st MARCH 1972

1.	Scientific	313
2.	Technical	202
3.	Non-technical	98
4.	Class IV technical	141
5.	Class IV non-technical	73
	Total	827

6. *Research fellows, Pool officers & Guest workers*

(a)	JRF and SRF	57
(b)	CSIR Pool Officers	12
(c)	Guest workers	27
	Total	96

7. *Scientific staff working on sponsored projects*

(a)	NCL staff	13
(b)	Staff deputed by sponsors	14
	Total	27

7. STAFF NEWS

7.1 *Foreign deputations/training, etc.*

(i) Dr. J. L. Bose worked as a N.R.C. (USA) Research Associate at the Southern Regional Research Laboratory of U.S. Dept. of Agriculture, New Orleans, in the field of ' Crosslinking of cotton cellulose' (January to August 1971).

(ii) Dr. L. K. Doraiswamy visited USA under Indo-USA Exchange of Scientists' Programme. He visited various research organizations in USA which are engaged in the research in the field of Chemical Engineering (October to December 1971).

(iii) Dr. S L. Kapur visited University of Mains, West Germany, under the Exchange of Scientists' Programme between the CSIR and German Academic Exchange Service, to acquaint himself, with the research work in the field of Polymers (May to July 1970).

(iv) Dr. S. L. Kapur attended IUPAC international symposium on ' Macromolecules ' held at Boston, USA and he also delivered a special lecture on ' Polymerization of polar monomers with Ziegler-Natta catalysts' (July 1971).

(v) Mr. S. Y. Kulkarni returned from Australia, after receiving training in the field of ' Research techniques in analytical chemistry' under Colombo Plan (April to December 1971).

(vi) Dr. J. C. Sadana visited U.S. Army Natick Laboratories, Natick, USA, and Louisiana State University, Baton Rouge, USA as a Visiting Professor (Sept. to October 1971).

(vii) Dr. B. D. Tilak toured Rumania and U.K as a leader of Indian Scientists' Delegation for formulating a programme for scientific and technological cooperation. He delivered several lectures in Rumania and later in U.K. He also gave a plenary lecture at symposium organized by the Chemical Society of U.K. at University of Aberdeen, U.K. (Sept. to October 1971).

(viii) Dr. B. D. Tilak toured West Germany, Holland, France, U.K., USA and Canada as a Member of the Selection Team for Indian Petrochemical Corpon. Ltd., Boroda (March to April 1972).

7.2 *Participation of NCL scientists in seminars*

Symposia, seminar, etc. and venue	*Name of the scientist*
1. Symposia on ' Crystallography', BARC, Bombay.	Dr. L.M. Pant, Mr. N. N. Dhaneshwar
2. Winter School in Solid State Chemistry, IIT, Kanpur.	Mr. M. S. Shetty, Miss P. G. Menon
3. Convention of Chemists, Bombay.	Dr. B. D. Tilak, Dr. P. R. SubbaRaman, Dr. V. N. Gogte, Dr. K. G. Das, Dr. C. R. Narayanan, Mr. A. R. Acharekar, Mr. A. G. Namjoshi
4. Symposium on ' Topics of current interest ', IIT, Kanpur.	Dr. C. R. Narayanan
5. 8th International symposium on ' Chemistry of natural products', New Delhi and Poona.	Dr. B. D. Tilak, Dr. C. R. Narayanan, Dr. J. L. Bose, Dr. K.G. Das, Dr. Sukh Dev, Dr. U. R. Nayak, Dr. A. P.

5.	(Continued)	Joshi, Dr.V. D. Patil, Dr.S.C. Sethi, Dr. A. S. Gupta, Dr. A. S. Narula, Dr. (Miss) R. Misra, Dr. A.S. Rao, Dr. S.N. Kulkarni, Dr.A.V. RamaRao.
6.	Short course on ' Interpretation of mass spectra,' Bombay.	Dr. J.L. Bose
7.	Gordan Research Conference on ' Textiles ', New Hampshire, USA.	Dr. J.L. Bose
8.	Symposium on ' Meterological aspects of air pollution ', Poona.	Dr. K.G. Das
9.	Short course in ' Interpretation of infra-red spectra ', Univ. of Poona, Poona.	Dr. V. S. Pansare
10.	International symposium on ' Morphogenesis in plant cell, tissue and organ cultures,' U. G. C. Centre, Delhi.	Dr. A. F. Mascarenhas
11.	International convention of Biochemistry, Indian Institute of Science, Bangalore.	Dr. V. Jagannathan, Dr. A. F. Mascarenhas, Miss F. S. Maskati, Miss V. K. Ranjekar
12.	Guha Research Conference, Dhikala (U.P.).	Dr. J. C. Sadana
13.	Symposium on ' Application of stainless steel in design and fabrication for chemical and process industries,' Durgapur.	Mr. A. D. Deshpande
14.	Symposium on ' Development of indigenous know-how in chemical and allied industries', HOC, Rasayani.	Dr. R. B.Mitra, Mr. A. M. Lele
15.	Seminar on ' Chemical industry ', Bangalore.	Mr. C. U. Saraf

7.3 *Awards and Honours*

Dr. B. D. Tilak was awarded ' Padma Bhushan' on the occasion of the Republic Day 1972.

7.4 *Post-graduate degrees received by NCL staff and research fellows*

S. No.	*Name*	*Degree*	*University*	*Subject of thesis*	*Name of the guide*
1.	Atchayya, M.	Ph. D.	Poona	Equilibrium studies of the reaction of aluminium chloride with rare earth oxides and oxygen absorption capacities of rare earth sesquisulphides	Dr. V. V. Dadape
2.	**Berde (Miss), H.V.	Ph. D.	Poona	Synthesis of nitrogen heterocyclic compounds	Dr. B. D. Tilak
3.	Bhasin, S. K.	Ph. D.	Poona	Studies on some metal complexes ligands, chelates and their reactivities	Dr. D. N. Sen
4.	Chakrabarty, D.	Ph. D.	Bombay	Studies in the vaporization of refractory material at high temperature	Dr. V. V. Dadape
5.	Deshpande, V.H.	Ph. D.	Poona	Isolation and structural elucidation of wood phenolics	Dr. K. Venkataraman
6.	*Goswami, N. N.	Ph. D.	Poona	Studies on some thin films by electron diffraction	Dr. A. Goswami
7.	Iqbal, S.H.	Ph. D.	Poona	Studies on the reactions of anhydrous inorganic halides	Dr. V. V. Dadape
8.	*Kolhe, D.B.	Ph. D.	Poona	Structural studies of vapour phase deposits of some oxides and alloys by electron diffraction	Dr. A. Goswami
9.	Mascarenhas, A.F.	Ph. D.	Poona	Metabolism of plant cell grown *in vitro*	Dr. V. Jagannathan
10.	Mehendale, A.R.	Ph. D.	Poona	Constitution and synthesis of naturally occuring hydroxy-anthraquinonoid pigment	Dr. K. Venkataraman
11.	Mittal, R. S. D.	Ph. D.	Rajasthan	Non-benzonoid aromatic compounds	Dr. Sukh Dev
12.	Murthy, B. R.K.	Ph. D.	Poona	Studies of high temp. chemical reactions involving meta-metal halides and metal oxides metal halides	Dr. V. V. Dadape
13.	Narula, A.S.	Ph. D.	Rajasthan	Modifications of tetracyclic triterpenes nitro steriod hormone analogues	Dr. Sukh Dev
14.	Paranjape, S. V.	Ph. D.	Poona	Studies of hexokinase from heart	Dr. V. Jagannathan
15.	Parulekar, A.D.	Ph. D.	Poona	The polarographic behaviour of some metal ions in sulphosalicyclic acid	Dr. P.R. SubbaRaman
16.	Rao (Mrs.), S. Suseela	Ph. D.	Poona	Study of magnetic and electrical properties of some spinels	Dr. A.P. B. Sinha
17.	Rao, V.S.	Ph. D.	Andhra	Application of nuclear magnetic resonance in the study of conjugated systems	Dr. P. M. Nair

S. No.	*Name*	*Degree*	*University*	*Subject of thesis*	*Name of the guide*
18.	Rathi, S. S.	Ph.D.	Poona	Studies in the chemistry of wood phenolics	Dr. K. Venkataraman
19.	Sawant B. M.	Ph. D.	Shivaji	Structural and conformational studies in terpenoids and related comopunds	Dr. C. R. Narayanan
20.	Surve A. S.	M.Sc. (Tech.)	Bombay	Technical preparations of organo-phosphorous compounds	Dr. B.D. Tilak

* These are Research Fellows/National Scholars.

7.5 *NCL scientists recognized by different universities as research guides*

Dr. Ayyangar, N. R.	Poona
Dr. Bose, J. L.	Bombay, Poona, Nagpur, Shivaji
Dr. Chakravarti, K. K	Poona, Shivaji, Karnatak
Dr. Dadape, V. V.	Bombay, Poona, Karnatak, Vikram, Banaras
Dr. Damodaran, V.	Shri Venkateswara University, Tirupathi
Dr. Das, K. G.	Poona, Bombay, Kerala, Marathwada, Kalyani
Dr. Doraiswamy, L. K.	Poona, Bombay, Nagpur, Calcutta, Jadavpur
Dr. Ghatge, B. B.	Poona
Dr. Ghatge, N. D.	Poona, Bombay, Shivaji
Dr. Gogte, V. N.	Shivaji
Dr. Goswami, A.	Calcutta, Poona, Punjab
*Dr. Gupta, J.	Bombay, Poona, Punjab, Madras
Dr. Ingle, T. R.	Poona
Dr. Jagannathan, V.	Bombay, Poona
Dr. Joshi, R. M.	Poona, Bombay
Dr. Kapur, S. L.	Bombay, Poona, Punjab
Dr. Katti, S. S.	Bombay
*Dr. Kelkar, G. R.	Poona
Dr. Kulkarni (Miss), S. B.	Poona
Dr. Kulkarni, S. N.	Poona, Bombay, Karnatak, Shivaji
Dr. Mathur, H. B.	Poona, Agra, Madras, Bombay, I.I.T.-Bombay
Dr. Nair, P. M.	Poona, Shivaji, Andhra
Dr. Narayanan, C. R.	Poona, Bombay, A. B. Univ., Zaria, Nigeria
Dr. Pai, M. U.	Bombay
Dr. Pant, L. M.	Poona
*Dr. Ramchandran, B. V.	Shivaji, Poona
Dr. Rama Rao, A. V.	Shivaji
Dr. Rangachari, P. N.	Poona, Shivaji
Dr Rao, A. S.	Poona, Shivaji
Dr. Roy Chowdhury, P.	Marathwada
Dr. Sadana, J. C	Poona, Aligarh
Dr. Sen, D. N.	Poona, Bombay

Dr. Sinha, A. P. B.	Poona, Banaras, Vikram, Bombay, Karnatak
Dr. SivaRaman, C.	Poona
Dr. SubbaRaman, P. R.	Poona, Bombay, Kerala
Dr. Sukh Dev	Poona, Bombay, Agra, Punjab, I. I. Science- Bangalore, Rajasthan
Dr. Tilak, B. D.	Bombay, Poona
*Dr. Venkataraman, K.	Poona, Bombay, Madras, Banaras, Karnatak

*Retired/Emeritus scientists

7.6 *Consultancy*

NCL scientists deputed as consultants

Name of the scientist		*Name of the firm*
1. Dr. Chakravarti, K. K.		M/s. Sunanda Aromatic Industries, Mysore
2. Dr. Ghatge, N. D.	i)	M/s. Dr. Beck and Co., Pimpri, Poona
	ii)	M/s. Swastik Rubber Products Ltd., Kirkee, Poona
3. Dr. Mitra, R. B.		M/s. Alta Laboratories Ltd., Khopoli
4. Dr. Rama Rao, A. V.		M/s. Poona Synthetic Co., Poona
5. Dr. Sukh Dev		M/s. Camphor and Allied Products Ltd., Bareilly
6. Dr. Tilak, B. D.	i)	M/s. Aniline Dyestuffs and Pharmaceuticals Pvt. Ltd., Bombay
	ii)	M/s. Indian Dyestuff Industries Ltd., Bombay

7.7 *Membership of the committees*

S. No.	*Name of the scientist*	*Position*	*Name of the committee*
1.	Dr. Ayyangar, N. R.	Member	Planning Group V : Dyes, intermediates for dyes, pharmaceuticals and other fine organic / inoraganic chemicals of National Committee on Science and Technology (NCST) Panel V
		Member	ISI—Food additives Sectional Committee—AFDC 19
		Member	Advisory Panel on Leather Auxiliaries of CLRI, Madras
2.	Dr. Bose, J. L.	Member	ISI—Textile sizing and finishing materials Sectional Committee—TDC—22
3.	Dr. Chakravarti, K. K.	Member	ISI—Natural and synthetic perfumery matrials Sectional Committee-CDC 11/A2.21
4.	Dr. Dadape, V. V.	Member	Planning Group III : Inorganic chemicals / electrochemicals of NCST Panel V
5.	Dr. Doraiswamy, L. K.	Member	Board of Directors, Indian Drugs and Pharmaceuticals Ltd., New Delhi
		Member	Board of Directors, Engineers India Ltd., New Delhi
		Member	Planning Group I : Bulk organic chemicals/petrochemicals/polymers of NCST Panel V
		Member	Planning Group IX : Chemical engineering research and design capability development of NCST Panel V
6.	Dr. Jagannathan, V.	Member	Planning Group IV : Organic fine chemicals including pharmaceuticals, drugs, perfumery chemiicals (essential oils),

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6.	Dr. Jagannathan, V.		physicochemicals, biochemicals, carbohydrates of NCST Panel V
7.	Dr. Mathur, H. B.	Member	Planning Group II : Fertilizers and other plant nutrients of NCST Panel V
8.	Dr. Mitra, R. B.	Member	Planning Group VII : Pesticides of NCST Panel V
		Member	ISI—Dye intermediate Sectional Committee—CDC 46
9.	Dr. Pant, L. M.	Member	Indian National Committee for International Union of Crystallography
10.	Dr. Sinha, A. P. B.	Member	Planning Group VIII : Marine chemicals—forward technologies relating to use of marine chemicals of NCST Panel V
11.	Dr. SivaRaman, C.	Member	Expert Committee on Biochemistry of Indian Council of Medical Research
12.	Dr. SubbaRaman, P. R.	Member	ISI—Structural and metal Divisional Council - SMDC-2 and Chemical standards Section Committee—CDC-I
13.	Dr. Sukh Dev	Member	Planning Group IV : Organic fine chemicals including pharmaceuticals, drugs, perfumery chemicals (essential oils), physicochemicals, biochemicals, carbohydrates of NCST Panel V
		Member	Planning Group VIII : Marine chemicals—forward technologies relating to use of marine chemicals of NCST Panel V
14.	Dr. Tilak, B. D.	Member	NCST, Govt. of India
		Convener	NCST, Panel V for Chemical Industry

14.	Dr. Tilak, B. D.	Director	Indian Petrochemicals Corporation Ltd., Baroda
		Chairman	Board of Directors, Hindustan Organic Chemicals Ltd., Rasayani
		Member	Board of Directors, Hindustan Antibiotics Ltd., Pimpri, Poona
15.	Dr. Unni, M. K.	Member	ISI—Industrial organic chemicals Sub-Committee-CDC 4 : 14

8. PUBLICATIONS

8.1 *Research Papers*

Solid state materials

1. Murthy, M. N. S. and Sinha, A. P. B.
Some practical considerations in the preparation of crystalline and polycrystalline barium titanate.
Indian J. Tech., **9**, 156 (1971).

Goswami, A. and Nikam, P. S.
Epitaxial growth and phase change in monoselenide of cobalt
J. Crys. Growth, **8**, 247 (1971).

Physico-chemical studies

3. Rao, Balachandra and Mathur, H. B.
Thermodynamics of the intersection of transition metal ions with histamine.
J. Inorg. & Nuclear Chem., **33**, 809 (1971).

4. Rao, Balachandra and Mathur, H. B.
Thermodynamics of *bis*-β-diketonato complexes of Cu(II) and Ni(II).
J. Inorg. & Nuclear Chem., **33**, 2919 (1971).

5. Mathur, J. N. and Mathur, H. B.
A Mossbauer study on the nature of Fe(II) bonding in *bis*-dithioacetylacetone tetrabromo-Fe(II) and *bis*-dithiobenzoylacetone-tetrachloro-Fe(II).
Indian J. Chem., **9**, 1348 (1971).

6. Gupta, M. P. and Mathur, H. B.
The nature of precipitated tin oxide in the internally oxidized Sn-Ag alloy.
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7. Pradhan, S. D., Katti, S. S. and Kulkarni (Miss), S. B.
Dielectric constants and dipole moments of *n*-long chain alcohols.
Indian J. Chem., **9**, 1345 (1971).

8. Pradhan, S. D., Katti, S. S. and Kulkarni (Miss), S. B.
Dielectric constants and dipolemoments of *n*-alkoxy ethanols and *n*-alkoxy propanols.
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9. Balakrishnan, I. and Reddy, M. P.
The effect of temperature in the gamma radiolysis of aqueous solutions. 1273
J. Phys. Chem., **76**, ~~8~~ (1972).

10. Bhowmik (Miss), S. and Roy Chowdhury, P.
Molecular characteristics and ultrasonic degradation of NeopreneAD.
J. Indian Chem. Soc., **48**, 699 (1971).

11. Kulkarni, M. P. and Dadape, V. V.
Sublimation pressure of cobalt and the study of the reaction $CO_{(c)} + COCl_{2(g)} = 2CO\ Cl_{(g)}$ at high temperatures.
High Temperature Science, **3**, 277 (1971)

12. Sen, D. N., Umapathy, P. and Budhkar, A. P.
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Indian J. Chem., **9**, 376 (1971)

Inorganic chemicals and products

13. Iqbal, S. H., Mahajan, S. S. and Dadape, V. V.
Chemical beneficiation and chlorination of ilmenite.
Chemical Engineering World, **VI**, 81 (1971).

Natural organic products

14. Landge, A. B. and Bose, J. L.
Structure of the disaccharide from *Asparagus racemosus* (Shatamuli).
Indian J. Chem., **8**, 588 (1970).

15. Nayak, U. R.
A convenient method for 8-oxoiosolongifolene from longifolene.
Indian J. Chem., **9**, 606 (1971).

16. Trivedi, G. K., Chakravarti, K. K. and Bhattacharyya, S. C.
Isolation and characterization of (—) -cadinene, (—)δ-cadenol and khusimol from north Indian vetiver oil.
Indian J. Chem., **9**, 1049 (1971).

17. Pai, P. P., Paknikar, S. K. and Chakravarti, K. K.
Revised structure for nardostachone, identity with 1,8,9,10—tetrahydroaristolan-2-one.
Indian J. Chem., **9**, 729 (1971).

Synthetic organic chemistry

18. Khan, H., Khan, A., Chetty, G. L., Gupta, A. S. and Sukh Dev
Cheilanthatriol—A new fundamental type in sesterterpenes.
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19. Patwardhan (Mrs.), S. A. and Sukh Dev
Cyclohexane-1, 4-dione.
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20. Audichya, T. D., Ingle, T. R. and Bose, J. L.
Studies in the stannic chloride catalysed glycosidation of phenols.
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21. Bapat, B. V. and Kulkarni, G. H.
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22. Tadwalkar, V. R. and Rao, A. S.
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23. Tadwalkar, V. R. and Rao, A. S.
Synthesis of *p*-menthenolides.
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24. Gogte, V. N.
Direction of cyclization reactions in the formation of polycyclic heteroaromatic compounds. A PMR assessment.
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25. Das, K. G., Gogte, V. N., Seetha (Miss) and Tilak, B. D.
Substituent effects in unimolecular ion decompositions under electron impact.
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26. Dingankar, P. R., Gore, T. S. and Gogte, V. N.
Synthesis of condensed furans-Mechanism of the formation of benzo-bis-benzofurans.
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27. Gogte, V. N., Namjoshi, A. G. and Tilak, B. D.
Synthesis of heterocyclic compounds; Part V : Synthesis of quinindine derivatives.
Tetrahedron Letters, 4305 (1971).

28. Berde (Miss), H. V., Gogte, V. N., Kulkarni, P. S. and Das, K. G.
Substituent effect in unimolecular ion decomposition in the gaseous phase.
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Synthesis of heterocyclic compounds; Part II : Phenothiaphosphine derivatives.
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31. Takwale, M. G. and Pant, L. M.
The structure of *p*-toluic acid.
Acta Cryst., **B 27**, 1152 (1971).

32. Barve, J. V. and Pant, L. M.
The structure of *p*-nitrotolune.
Acta Cryst., **B 27**, 1158 (1971).

32. Tavale, S. S. and Pant, L. M.
Further refinement of the structure of *p*-nitrobenzoic acid.
Acta Cryst., **B 27**, 1479 (1971).

34. Dhaneshwar, N. N. and Pant, L. M.
The structure of *n*-methylanthranilic acid.
Acta Cryst., **B 28**, 647 (1972).

35. Gujarathi, N. C. and Jose, C. I.
Infrared spectra and molecular structure Part I : 3-amino, 4-amino and 5-amino salicylic acids.
Indian J. Chem., **9**, 822 (1971).

36. Sen, D. N., Jose, C. I. and Umapathy, P.
Infrared spectral studies on acetoacetanilides.
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37. Narayanan, C. R. and Parkar (Miss), M. S.
Deshielding effect on adjacent protons on esterification of a hydroxyl group.
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38. Narayanan, C. R. and Lala, A. K.
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39. Nair, P. M., Gopakumar, G., Fairwell, T. and Rao, V. S.
Long range spin-spin coupling and π-electron distribution.
Indian J. Chem., **9**, 549 (1971).

40. Fairwell, T. and Nair, P. M.
α-Acetoxybenzyl perchlorates.
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41. Rao, J. M., Nair, K. R. R. and Nair, P. M.
Mutarotation of L-Rhamnosazone.
Indian J. Chem., **9**, 604, (1971).

42. Tadwalkar, V. R., Narayanaswamy, M. and Rao, A. S
Preparation and stereochemical characterization of 1, 6-epoxy-*p*-menthan-2-ols and 1,6-epoxy-*p*-menthan-2-ones.
Indian J. Chem., **9**, 1223 (1971).

43. Pandhare, E. D., Patil, V. B., Rao, A. V. R. and Venkataraman K.
Applications of NMR spectroscopy and mass spectrometry to some problems concerning synthetic dyes; Part VIII : Positions of the aryl group in two β (*p*-methoxyphenyl)4,8-diamino-1, 5-dihydroxy anthraquinones.

    Indian J. Chem., **9**, 106 (1971).

44. Das, K. G., Manjrekar, T. G., and Venkataraman, K.
Applications of NMR spectroscopy and mass spectrometry to some problems concerning synthetic dyes; Part IV : Mass spectra of violanthrone derivatives and structure of indanthrene Navy Blue G.

    Indian J. Chem., **9**, 921 (1971).

45. Nair, P. M., Manjrekar, T. G., Rao, A. V. R. and Venkatraman, K.
Applications of NMR spectroscopy and mass spectrometry to some problems concerning synthetic dyes; Part VII : NMR spectra of some violanthron derivatives.

    Indian J. Chem., **9**, 925, (1971).

46. Nair, P. M., Sinha, A. P. B. and Venkataraman, K.
Laser Raman spectroscopy of organic compounds.
Current Sci., **40**, 239 (1971).

Enzyme chemistry and technology

47. Paranjape, S. V. and Jagannathan, V.
Properties and kinetics of purified particulate ox heart hexokinase.
Indian J. Biochem. Biophys., **8**, 227 (1971).

48. Husain, M. and Sadana, J. C.
Simple gel electrophoresis procedure for purification of *Achromobacter fischeri* nitrite reductase.

    Analytical Biochem., (USA) **45**, 316 (1972).

49. Om Prakash and Sadana, J. C.
Purification characterization and properties of nitrite reductase of *Achromobacter fischeri.*

    Archio. Biochem. Biophys., **148**, 614 (1972).

Polymers, resins and elastomers

50. Ghatge, N. D. and Kulkarni, D. M.
Condensation of cashewnut shell liquid fraction with cyanuric chloride.

    J. Shivaji Univ., **1**, (1969).

51. Ghatge, N. D. and Vernekar, S. P.
Plasticizer extenders for PVC from cashewnut shell liquid.
European Polymer J., **6**, 1547 (1970).

52. Ghatge, N. D. and Patil, V. S.
Synthesis of isocyanates.
Die Angew Makromol Chemie., **19**, 83 (1971).

53. Ghatge, N. D. and Srinivasan, S. R.
Polyurethane textile coatings from shellac.
Textile Research Journal, **41**, 78, (1971).

54 Ghatge, N. D. and Kulkarni, D. M.
Epoxides from aromatic sulphides and sulphones.
Indian J. Tech., **9**, 323 (1971).

55. Ghatge, N. D. and Gokhale, R. G.
Rubber chemicals from cashewnut shell liquid.
Indian J. Tech., **9**, 391 (1971).

56. Ghatge, N. D. and Vernekar, S. P.
Organo-tin compounds in stabilization of polyvinyl chloride(PVC).
IOEC Product Res. and Dev., **10**, 214 (1971).

57. Ghatge, N. D. and Vernekar, S. P.
Syntheses of ultraviolet light absorbers for polyvinyl chloride (PVC) Part I.
Die Angew Makromol Chemie., **20**, 165 (1971).

58. Ghatge, N. D. and Vernekar, S. P.
Evaluation of ultraviolet light absorbers in polyvinyl chloride (PVC) Part II.
Die Angew Makromol Chemie., **20**, 175 (1971).

59. Ghatge, N. D. and Vernekar, S. P.
Soaps of aleuritic acid as stabilizers for PVC.
Indian J. Tech., **9**, 262 (1971).

Studies in polymer chemisry

60 Dixit, S. S., Deshpande, A. B. and Kapur, S. L.
Studies in the polymerization of methyl methacrylate with VCl_4-$Al(C.H_5)_3$ catalyst system in acetonitrile.
J. Polymer Sci., Part-C, **31**, 6 (1970).

61. Dixit, S. S., Deshpande, A. B. and Kapur, S. L.
Polymerization of acrylonitrile with VCl_4-$Al(C_2H_5)_3$ catalyst system in presence of acetonitrile.
J. Polymer Sci., *A*-1, **9**, 1167 (1971).

62. Dixit, S. S., Deshpande, A. B. and Kapur, S. L.
Alteration intacticity of polymethylmethacrylate obtained with $VOCl_3$-$AlEt_3$ in presence of an electron donor.
European Polymer Journal, **7**, 699 (1971).

63. Ghatge, N. D. and Gokhale, R. G.
The structure and configuration of dihydroxy diphenyl sulphide derived from tetrahydro anacardol by infrared spectra.
Rubber News, **10**, 21 (1971).

64. Ghatge, N. D. and Mahajan, S. S.
Evaluation of amines as antiradiation compounds in styrene butadiene rubber.
Die Angew Makromol Chemie., **18**, 49 (1971)

65. Ghatge, N. D. and Patil, S. B.
Studies in reclaims from styrene—butadiene rubber scrap
Kautschuk and Gummi. Kunststaffe 24, *Jahrgang Heff*, **6**, 299 (1971).

66. Ghatge, N. D. and Patil, V. S.
Studies in the reactivities of diisocyanates.
Die Angew Makromol. Chemie., **19**, 75 (1971).

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Studies in liquid polysulphide rubbers.
J. Indian Rocket Soc., **1**, 33 (1971).

68. Joshi, R. M.
Bond-energy/group contribution methods of calculating the standard heat of formation : Development of a new generalized bond-energy scheme for monomers and polymers : Part II- Hydrocarbon.
J. Macromol. Sci. Chem., **A5**(**4**) 687, (1971).

69. Joshi, R. M. and Joshi, S. G.
A new analytical solution of the binary copolymer composit on equation and suggested procedure for deriving the monomer reacitivity ratios.
J. Macromol. Sci. Chem. **A5**(**8**), 1329 (1971).

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A model for solid-gas reactions.
Chem. Engg. Sci., **26**, 1521 (1971)

71. Choudhary, V. R. and Doraiswamy, L. K.
Isomerization of *n*-butene to isobutene Part; I—Selection of catalyst by group screening.
J. of Catalysis, **23**, 54 (1971)

72. Choudhary, V. R. and Doraiswamy, L. K.
Application of gas chromatography in catalysis.
Ind. Engg. Chem. (Prod. Res. and Dev.), **10**, 218 (1971).

73 Sadana, A. and Doraiswamy, L. K.
Effect of catalyst fouling in fixed-, moving and fluid-bed reactors
J. of Catalysis, **23**, 147 (1971).

74 Parande, M. G. Balakrishnan S. and Srinivasan K. P.
Solubility of *p*-dichlorobenzene in benzene.
Indian J. Tech., **9**, 402 (1971).

75. Ramaswamy, V.
An enthalpy concentration diagram of the system acetone-carbontetrachloride.
Brit. Chem. Engg. & Proc. Tech., **16**, 1038 (1971).

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Mass transfer in rotating sieve disc contactor.
Chemical Process Engineering, 67 (1971).

77. Paul, R. N. and Johny, C. J.
Nitrobenzene as an extraction solvent for pyridine.
Brit. Chem. Engg. & Proc. Tech., **16**, 1135 (1971);

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A better solvent for pyridine extraction.
Brit. Chem. Engg. Proc. Tech., **17**, 69 (1972).

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Ethylene dichloride is unselective for lower members of saturated fatty acid series.
Brit. Chem. Engg. Proc. Tech., **17**, 251 (1972).

Analytical chemistry

80. Nambiar, O. G. B. and Subbaraman, P. R.
Preparation and polarographic behaviour of tris-(ethyloximinoaceto-acetato)-cobalt(III) and tris-(diacetylmonoximo)-cobalt(III).
Indian J. Chem., **9,** 1138 (1971).

81. Nambiar, O. G. B. and Subbaraman, P. R.
Polarography of coordinated oximes.
Australian J. Chem., **24,** 2089 (1971).

8.2 *Publications in collaboration with outside scientists*

1. Bose, J. L., Roberts, E. J. and Rowland, S. P.
Availability and state of order of hydroxyl groups on the surface of microstructural units of crystalline cotton cellulose.
J. Polymer Sci., **9,** 1431 (1971).

2. Bose, J. L., Roberts, E. J., Wade, C. P. and Rowland, S.P.
Assessment of the state of order of hydroxyl groups and of molecular segments in hydrocellulose.
J. Polymer Sci., **9,** 1623 (1971).

3. Bose, J. L., Roberts, E. J. and Rowland, S. P.
The availability of hydroxyl groups of native and mercerized cellulose.
J. Applied Polymer Sci., **15,** 2999 (1971).

8.3 *Chapters in the Books*

Bendale D. S.
Reactivity of pulp from cellulosic materials for rayon manufacture.
'Hand book of rayon,' Century Rayon Publication.

8.4 *Reviews and General science papers*

1. Rangachari, P. N.
Prostaglandins.
J. Sci. Industr. Res., **31,** 26 (1972).

2. Das, K. G.
Mass spectrometry.
Instruments India, *May*, 29 (1971).

8.5 *Papers sent/read at symposium, seminar, etc.*

1. Goswami, A.
   Studics on solid thin films.
   Symposium on ‘ Solid thin films,’ Bangalore, April, 1971.

2. Dhere, N. G. and Goswami, A.
   Vapour phase deposits of bismuth selenide.
   5th International Vacuum Congress, Boston, U.S.A., October 1971.

3. Goswami, A. and Kolhe, D. B.
   Electron diffraction study of vapour phase deposits of SnSb films.
   Symposium on ‘ Crystallography ’, BARC, Bombay, February 1972.

4. Goswami, A. P. and Goswami, A.
   Structure and dielectric properties of niobium oxide films.
   International Conference on ‘ Thin films, ’ to be held in May 1972 at Venice.

5. Goswami, A.
   A new instrument for measuring optical properties of thin films.
   UGC-symposium on ‘ Thin films technology ’ Andhra Univ., Waltair, March, 1972.

6. Goswami, A. and Ojha, S. M.
   A study of electrical properties of tellerium films.
   UGC-symposium on ‘ Thin films technology,’ Andhra Univ., Waltair, March, 1972.

7. Goswami, A. and Jog, R. S.
   Growth of SnSe films on single crystal substrates.
   UGC-symposium on ‘ Thin films technology ’, Andhra Univ., Waltair, March, 1972.

8. Menon (Miss), P. G. and Sinha, A. P. B.,
   Oxide magnetic materials.
   Winter School in ‘ Solid state chemistry, ’ IIT, Kanpur, December, 1971.

9. Narayanan, C. R.
   Change migration in some mass spectral fragmentations.
   8th International Symposium on ‘ Chemistry of natural products ’, New Delhi, February, 1972.

10. Narayanan, C. R.
Fragmentation pattern of steriodial alkyl ethers.
8th International Symposium on ' Chemistry of natural products', New Delhi, February, 1972.

11. Nair, P. M.
A nuclear magnetic resonance study of ascorbic acid and its derivatives.
Symposium on ' Applications of NMR spectroscopy in organic chemistry', Convention of Chemists, Bombay, October, 1971.

12. Das, K. G.
Air quality measurements by mass spectrometry.
Symposium on ' Air pollution,' College of Engg., Madras, December, 1971.

13. Das, K. G.
Mass spectrometry in pesticide rsidue analysis.
Symposium on 'Progress and problems in pesticide residue analysis', Punjab Agricultural Univ., Ludhiana, November, 1971.

14. Das, K. G.
Mass spectrometry in waste disposal and pollution studies.
Seminar on ' Waste disposal and air pollution ',
Institute of Technology, Coimbatore, October, 1971.

15. Das, K. G.
Air anality measurements by mass spectrometry.
Seminar on ' Meterological aspects of air pollution '
Poona, March, 1972.

16. Das, K. G. and Thayumanavan, B.
A mass spectral approach to characterize fructose units in oligosaccharides.
8th International Symposium on ' Chemistry of natural products ', New Delhi, February, 1972.

17. Rao, A. S.
Synthesis of (+)-(IR : 4S)-pulegone oxide; 1,6- epoxy-*p*-emphan-2-Ols and 1 m 3 (H), 4-*p*-menth-8-cu-3, 10-olide.
8th International symposium on ' Chemistry of natural products '
New Delhi, February, 1972.

18. Rama Rao, A. V.
Four novel flavones from the bark of *Morus rubra.*
8th International symposium on ' Chemistry of natural products ', New Delhi, February, 1972.

19. Nayak, U. R.
Some asects of longifolene chemistry.
Seminar on the 'Role of pine resin in the economic and industrial development of India', New Delhi, April, 1971.

20. Patil, V. D., Nayak, U. R. and Sukh Dev
Novel steroids from *Commiphora mukul* Hook, ex Stocks.
8th International Symposium on 'Chemistry of Natural Products', New Delhi, February, 1972.

21. Joshi, A. P., Nayak, U. R. and Sukh Dev
3-Hydroxylongifolaldehyde, the long-sought-after intermediate in the abnormal perbenzoic acid oxidation of longifolene.
8th International Symposium on the 'Chemistry of Natural Products,' New Delhi, February, 1972.

22. Ghatge, N. D. and Yadav, S. D.
Synthesis of polyisocyanates from pentadecenyl phenol (anacardol) and its use in modification of textiles and papers.
Symposium on 'Contribution to durable finishing of cotton textiles', Bombay, May, 1971.

23. Ghatge, N. D., Gujar, K. B. and Srinvasan, S. R.
Water proof fabrics using polyurethanes.
7th technological conference, SASMIRA, Bombay, February, 1971.

24. Mascarenhas, A. F.
Studies on tissue cultures of maize, wheat, rice and sorghum.
Convention of Biochemistry, Bangalore, December, 1971.

25. Mascarenhas, A. F.
Studies on tissue cultures of maize, rice and sorghum.
International symosium on, 'Morphogenesis in plant cell, tissue and organ cultures,' New Delhi, November, 1971.

26. Maskati (Miss), F. S.
Metabolism of laevorotatory citramalate.
Convention of Biochemistry, Bangalore, December, 1971.

27. Ranjekar (Miss), V. K.
Acyl phosphatase of V. catjang.
Convention of Biochemistry, Bangalore, December, 1971.

9. PATENTS IN FORCE

9.1 *Patents sealed*

1. **59497***
   Production of porous polymer suitable for preparing cation exchange resins.
   Govindan, K. P., Pandya, R N. and Krishnaswamy, N.

2. **59606***
   Preparation of cation exchange resin from porous cashewnut shell liquid polymer.
   Krishnaswamy, N., Pandya, R. N. and Govindan, K. P.

3. **59608***
   Rigid filters.
   Kapur, S. L. and Pandya, R. N.

4. **60555***
   Production of liquid rubber.
   Uma Shankar.

5. **62890***
   A new process for the production of 4-hydroxycoumarin and its derivatives.
   Shah, V. R., Bose, J. L. and Shah, R. C.

6. **63083***
   A new method for the preparation of 4-hydroxycoumarins.
   Shah, V. R., Bose, J. L. and Shah, R. C.

7. **64958***
   Improvements in or relating to polishing compositions.
   Shah, S. M., Hinge, V. K., Mhaskar, V. V. and Shah, R. C.

8. **65440***
   A process for the extraction of wax from sisal waste.
   Shah, S. M., Hinge, V. K., Mhaskar, V. V. and Shah, R. C.

9. **65976***
   Improvements in or relating to suspension polymerization of vinyl monomers.
   Joshi, R. M. and Kanpur, S. L.

10. **65977***
    Rubber base adhesive.
    Uma Shankar.

11. **66096***
A process for the production of bacterial diastase by submerged culture.
Babbar, I. J., Bekhi, R. M. and Srinivasan M. C.

12. **66194***
Improvements in or relating to can sealing composition.
Raghunath, D. and Kapur, S. L.

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Improvements in or relating to the manufacture of pressure sensitive adhesive tapes.
Kapur, S. L. and Rao, B. R. K.

14. **66966***
An improved process for the manufacture of porous rigid filters.
Kapur, S. L. and Pandya, R. N.

15 **67490**
Improvements in or relating to preparation of adhesive tapes.
Kapur, S. L. and Rao, B. R.

16. **71063**
Production of bacterial protease by submerged culture.
Babbar, I. J., Powar, V. K. and Jagannathan, V.

17. **71190***
Preparation of anion exchange resins.
Krishnaswamy, N., Govindan, K. P. and Dasare, B. D.

18. **73702**
A process for the preparation of cyclopentadecanolide (exaltolide).
Dhekne, V. V., Ghatge, B. B. and Bhattacharyya, S. C.

19. **77081***
Improvements in or relating to the preparation of polyamide compounds and their compositions as antipriming agents in steam generators.
Pathak, K. D. and Subba Rao, B. C.

20. **77225***
A process for the preparation of β-ionone from pseudoionone.
Joshi, B. N., Chakravarti, K. K., Shah, R. C. and Bhattacharyya, S. C.

21. **82822**
A process for the manufacture of high alpha cellulose dissolving grade pulps by alkaline pulping methods.
Vyas, G. M., Bendale, D. S. and Mahajan, M. B.

22. **86541**
A reactor for carrying out highly exothermic and explosive reactions particularly suited for chlorination of methane.
Mukherjee, S. P., Deshpande, A. D., Potnis, G.V. and Pai, M.U.

23. **86991***
Preparation of polyurethane printing rollers.
Ghatge, N. D. and Kapur, S. L.

24 **92977***
Improvements in or relating to the manufacture of hexachloroethane.
Mukherjee, S. P., Goswami, M., Soundararajan, S., Sadasivan, N., Sen, R. K. and Doraiswamy, L. K.

25. **98155***
An improved method for the hydration of sulphonated mixtures ot vinyl copolymer beads containing 90-95% sulphuric acid and the sulphonated vinyl copolymer beads.
Kapur, S. L. and Ramakrishnan, K.

26. **98156***
Preparation of solvent modified copolymers of vinyl monomers in bead form.
Kapur, S. L. and Ramakrishnan, K.

27. **108413**
A process for treatment of costus roots (*Saussurea lappa* Clarke) for isolation of inulin.
Kulkarni, G. H., Kelkar, G. R., Bose, J.L. and Bhattacharyya,S.C.

28. **108414**
A process for treatment of costus roots (*Saussurea lappa*) or inulin isolated therefrom for the production of fructose.
Kulkarni, G.H., Kelkar, G. R., Bose, J.L. and Bhattacharyya S.C.

29. **109489**
Production of aryl-glycosides.
Ingle, T. R. and Bose, J. L.

30. **111311**
A process for the recovery of anhydrous pyridine bases from their aqueous solution.
Buzruk, N. K., Paul, R. N., Potnis, G. V. and Pai, M.U.

31 **113406**

A new thermistor composition exhibiting high positive temperatuer coefficient of resistivity (posistor).
Brahmecha, B. G. and Sinha, K.P.

32. **116453**

Disperse dyes for polyesters with good affinity and sublimation fastness.
Raman, S. K. and Tilak, B. D.

33. **116675**

Improvements in or relating to polymers possessing polyamide linkages for plastics, fibres and like industrial applications.
Ghatge, N. D., Patil, S. B. and Patil, V. S.

34. **117210**

A direct process for the manufacture of dibutylin di-iodide and of the oxide therefrom.
Gupta, J., Gopinathan, C., Gopinathan (Mrs.), S. and Awasarkar, P. A.

35. **117403**

Preparation of water thinnable emulsion paints from linseed oil.
Kapur, S. L. and Bakshi, S. H.

36. **118476**

Methods of preparing isocyanates, mono and polyureas from pentadecenyl phenol (anacardol).
Ghatge, N. D. and Yadav, S. D.

*These patents (20) are being commercially exploited.

9.2 *Patents filed*

1 **67513**

Improvements in or relating to the separation of niobium and tantalum from each other by liquid-liquid extraction.
Sarma, B. and Gupta, J.

2 **81072**

Improvements in or relating to the preparation and production of catalysts for the hydrogenation of organic substances with particular reference to fatty oils.
Murthy , M. N. S. and Biswas, A. B.

3 **94766**
Improvements in or relating to the preparation of jatamansi root and isolation of a coumarin constitutent therefrom.
Unni, I. R., Maheshwari, M. L., Paknikar, S. K. and Bhattacharyya, S. C.

4. **113703**
Improvements in or relating to magnesium zinc ferrites.
Krishna Rao, V. V., Kanade (Miss), S. B. and Sinha, A. P. B

5. **113825**
11 ORT nickel zinc ferrites.
Krishna Rao, V. V. Kanade (Miss), S. B. and Sinha A.P.B.

6. **121538**
A process for the shellac-polyester urethane compositons for use in textiles, water proofing adhesives, paints and varnishes and in electrical insulating varnishes.
Ghatge, N. D., and Srinivasan, S. R.

7. **123638**
Improvements in or relating to the manufacture of cashewnut shell gum (CNS-gum).
Ingle, T. R., Vaidya, S. H. and Pai, M. U.

8. **125138**
Method of preparing polyisocyanates and polyureas from pentadecenyl phenol (anacardol).
Ghatge, N. D. and Mahajan, S. S.

9. **126354**
A process for obtaining useful steriods from a new plant source.
Sukh Dev, Patil, V. D. and Nayak, U. R.

10. **126393**
An improved method for the manufacture of calcium hypophosphite.
Goswami, M., Lobo, J. and Brahme, P.H.

11. **127743**
A process for obtaining colchicine from a new plant source.
Kapadia, V. H., Sukh Dev and Rao, R. S.

12. **127750**
A process for production of pyridoxine hydrochloride.
Joshi, C. G. and Sukh Dev.

13. **130254**
A process for the manufacture of valcanizable graft copolymer of chlorosulfonyl polyethylene with maleic anhydride (and its substituted analogues) for use as a synthetic rubber and as a base in coating compositions.

Joshi, R. M

14. **130551**
A new process for separation of the dimethyl and monomethyl components from a mixture of dimethyl dichlorosilane and methyl trichlorosilane.
Gupta, J., Gopinathan, C., Gopinathan (Mrs.) S., Eapen, M. J. and Awasarkar, P. A.

15. **131606**
Improvements in or related to a process for the preparation of gamma ferric oxide.
Lakhbir Singh, Chavan, A. M. and Kotasthane, A. N.

16. **134641**
A device for warming and cooling.
Lakhbir Singh and Kotasthane, A. N.

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