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1972-73



NATIONAL CHEMICAL LABORATORY, POONA

**NATIONAL CHEMICAL LABORATORY
POONA
1972-1973**



COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH

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Director, NCL

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PREFACE

In last year's Annual Report, a reference was made to the formulation of a national plan for science and technology by the National Committee on Science and Technology (NCST). A draft of the plan for chemical industry in two volumes has already been submitted by the undersigned to the NCST and through the latter to the Planning Commission. Other NCL scientists have also played a very active role in the formulation of this plan.

NATIONAL PLAN FOR R & D FOR THE DEVELOPMENT OF CHEMICAL INDUSTRIES

The plan consists of more than 250 R & D proposals which are of immediate and long term relevance to the Indian chemical industry. The total R & D outlay envisaged for the implementation of these proposals amounts to Rs. 40 crores.

A large majority of the R & D projects recommended for implementation was proposed by the research laboratories under the CSIR, other government supported R & D laboratories in the country and a few public sector chemical undertakings. The proposals included in the plan take into account inter-group priorities in relation to the relative socio-economic needs, urgency and importance of the R & D projects and the relevant sectors of the chemical industry. Thus the sectors such as fertilizers and pesticides, which provide vital inputs for increasing agricultural production have claimed a maximum share amounting to 35% (Rs. 14.27 crores) of the total outlay. Petrochemicals which provide the foundation of the organic chemical industry service a large number of processing units in the small and medium scale sectors (viz. plastics, fibres, synthetic rubbers and bulk chemicals) have been allotted 28% (Rs. 12 crores) of the total inputs. Drugs and pharmaceuticals come next with an input of about Rs. 5 crores (12%). A significant feature of the science and technology plan for chemical industry is the special attention given for organizing and strengthening the R & D and design engineering infrastructure in the public sector chemical undertakings as well as in the national research laboratories of the CSIR. The R & D inputs in the public sector production units will be of the order of Rs. 26.7 crores (67% of the total outlay) and Rs. 12 crores in the CSIR (30% of the total inputs).

CSIR/NCL CONTRIBUTION TO THE NATIONAL EFFORT

The number of projects (from S&T plan) to be implemented in the research laboratories of the CSIR comes to 176 involving R & D inputs of Rs. 12 crores. Out of the above projects 65 projects have been allocated for

implementation in the NCL during the course of the 5th Plan. 27 projects from these envisage collaborative research with other laboratories in the country. In respect of the 38 projects to be exclusively implemented at the NCL, work was initiated, during the year under review, on 13 projects involving R & D inputs of the order of Rs. 165 lakhs over a period of 5-7 year. In addition, it is understood that 8 projects in the areas of electronics, solid state materials and instrumentation technology are likely to be assigned to the NCL for implementation in the coming years by the NCST.

R & D programmes of the NCL have been thoroughly scrutinized and reviewed over the last few years and especially during the year under review in the context of the anticipated assignment of projects identified by the NCST. This has naturally resulted in the re-examination of priorities, time targets and allocation of resources to the on-going projects so that work can be initiated on NCST projects in the coming years without much dislocation of the existing important activities.

The presentation of the present report is somewhat different from previous annual reports. The research activities of the NCL during 1972-73 have now been grouped under the following major areas of importance:— petrochemicals and bulk organic chemicals; pesticides; drugs, pharmaceuticals and fine chemicals; organic intermediates, dyes and industrial chemicals; utilization of plant and forest resources; the chemistry of biologically active compounds; studies in organic synthesis; radiation chemistry; studies in physical organic chemistry; industrial polymers, resins, elastomers and synthetic fibres; inorganic chemicals and catalysts; utilization of mineral resources; organo metallics; solid state materials; instrument technology; tissue culture studies; enzymes and fermentation technology and process development and chemical engineering studies. Under the broad areas mentioned above the projects are presented in the following order : sponsored, pilot plant, product/process oriented research and basic research projects. In presenting the work in progress attention is also drawn to the relevance of the work to the problems of industrial development and the country's socio-economic needs.

RESEARCH UTILIZATION

Details of the products manufactured on the basis of technology developed at the NCL are listed in Table I at the end of this report. Although the number of processes in production during 1972-73 has come down to 48 (51 in 1971-72), the value of production has gone up to Rs. 557 lakhs as against Rs. 471 lakhs in 1971-72. There is thus an 8 fold increase in the value of production during the last 5 years as can be seen from the data given below :

Year	1968—69	1969—70	1970—71	1971—72	1972—73
No. of processes in production	47	53	55	51	48
Value of production Rs. in lakhs	70	130	284	471	557

Information on 62 projects which are awaiting production is given in Table II. It will be seen that considerable progress has been registered in the commercial implementation of some of the high volume/value projects such as chloromethanes, chlorobenzenes, vitamin C and opium alkaloids. Table II also includes about 18 projects which have been recently licensed and which are expected to have a considerable economic impact. It is expected that the value of annual production based on NCL know-how during the 5th five year plan period will be of the order of Rs. 10-15 crores.

During the year under review production was reported for the first time on butyl titanate, clofibrate and dimethylaniline. M/s. Synthochem of Indore have set up a plant for the production of butyl titanate used in varnishes and enamels and the quantity produced during 1972-73 amounted to 16.6 tonnes valued at Rs. 5 lakhs. M/s. Biological Evans of Hyderabad have started trial production of clofibrate. M/s. Sahyadri Dyestuff and Chemicals of Poona have produced 25 tonnes of dimethylaniline valued at Rs. 2.5 lakhs during the year. M/s. Sonebon Laboratories, Kottayam also have established production of dihydroisojasmone and peach aldehyde during this year.

M/s. Hindustan Organic Chemicals (HOC), Rasayani, have completed the installation of 4000 TPA plant for chlorobenzenes based on NCL know-how. The plant will be commissioned in the next few months. The acetanilide plant of HOC attained near-rated capacity and the value of production achieved during 1972-73 amounted to over Rs. 138 lakhs. It was understood that the gross profits earned on this plant during the year under review nearly equalled 90% of the erected battery limit investment on the plant.

A 3000 TPA plant for chloromethanes based on NCL technology by M/s. Standard Alkali, Thana is under erection.

During the year under review, 17 processes were licensed to 21 parties through National Research & Development Corporation of India, New Delhi, in addition to 7 projects on which work was completed on sponsored basis. Details regarding these projects are given in appendices under sub sections 3 and 4.

Table II A reveals a review of research utilization of processes so far developed by the NCL on its own. It can be seen that by the end of 1972-73, in all 111 processes have been developed out of which 29 are in production,

31 released and not yet in production and 51 available for release. The value of production during 1972-73 of these 29 processes amounted to Rs. 401 lakhs. The NCL know-how on 60 processes so far released has been acquired by 75 parties.

SPONSORED PROJECTS

A similar review of contractual research undertaken at the NCL is given in Table II B. The total number of processes developed on sponsored basis up to 31st March 1973 amounted to 86 of which 15 projects are in production accounting for an annual turnover of Rs. 154.39 lakhs. On 36 projects, steps have been taken for establishing commercial production. In all 61 parties participated in the contractual research. The cumulative receipts on sponsored projects up to 31st March 1973 amounted to Rs. 62.51 lakhs and the cumulative value of production generated as a result of commercial implementation of the sponsored projects stood at Rs. 529.34 lakhs.

During the year under review sponsored work was initiated on 8 new projects while 7 sponsored projects were completed in the same period.

Table III highlights the technologies available from NCL for interested entrepreneurs, in which data on the field of utilization, imported components, scale of development and provisional terms of release in respect of each of the technologies are indicated.

PRODUCT ORIENTED RESEARCH

During the year under review, the total number of product-oriented research projects in operation amounted to 88 of which 5 were petrochemicals and bulk organic chemicals; 6 in pesticides; 7 in drugs, pharmaceuticals and fine chemicals; 6 in organic intermediates, dyes and industrial chemicals; 18 in the area of utilization of forest and plant resources; 3 in chemistry of biologically active compounds; 10 in industrial polymers, resins, elastomers; 7 in inorganic chemicals and catalysts; 2 in utilization of mineral resources; 4 in organo metallics, 5 in solid state materials; 4 in instrument technology; 1 in enzymes chemistry; 4 in fermentation technology and 6 in process development and chemical engineering studies.

BASIC RESEARCH

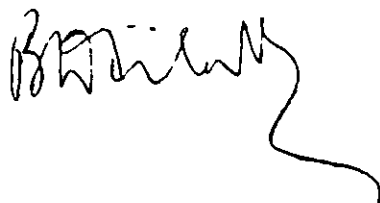
A survey of the basic research work pertaining to the period under review is also given under the relevant areas of R & D activities. Results of basic research have been recorded in 66 research papers published in scientific journals in India and abroad. In addition, about 22 research workers received post-graduate degrees (1 M.Sc. and 21 Ph.D.s) during 1972-73.

COST/BENEFIT ANALYSIS

The cost/benefit analysis of the work of the laboratory is presented

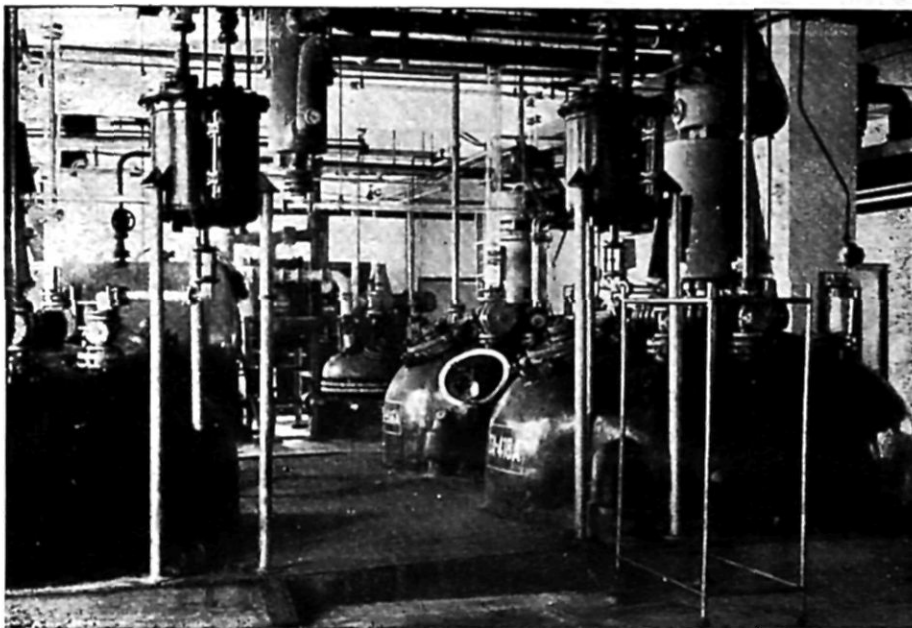
on page 153. Whereas figures for inputs represent actual expenditure, the benefits (output) accruing from NCL's work include statements of receipts on account of premia, royalties, sponsored projects, analytical testing charges, FCP sales, consultancy, etc. (direct benefits amounting to Rs. 14.65 lakhs), along with indirect benefits such as value of production, foreign exchange saved, generation of R & D capability (research degrees awarded), inquiries answered, research publications, advice to industry and Government departments, etc.

As against the NCL inputs (for 1972-73) of Rs. 79.73 lakhs (Rs. 69.52 lakhs recurring; Rs. 7.87 lakhs capital; Rs. 2.34 lakhs pilot plant), Rs. 4.53 lakhs were received as contributions from sponsors; Rs. 1.07 lakhs by way of royalties and premia, Rs. 2.40 lakhs as sale of FCP chemicals and other products, Rs. 0.30 lakhs as analytical fees, miscellaneous receipts Rs. 5.99 lakhs; institutional consultancy Rs. 0.36 lakhs. The total value of industrial production for the year under review was Rs. 557 lakhs of which Rs. 222 lakhs could be claimed as contribution to foreign exchange savings.



B. D. Tilak
Director

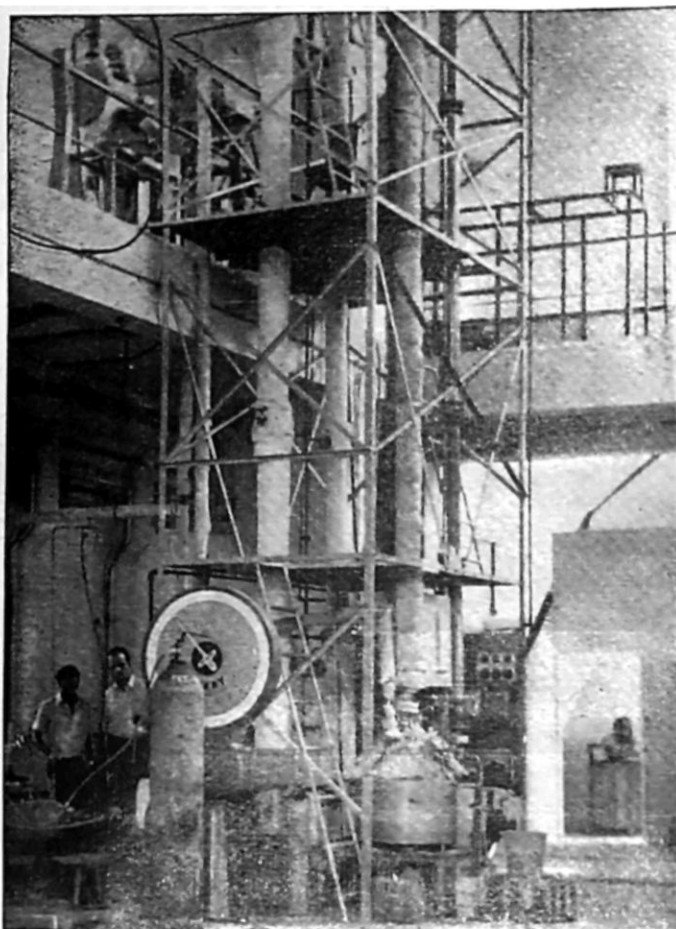
November, 1973



A View of the Vitamin C Plant at HAL, Pimpri

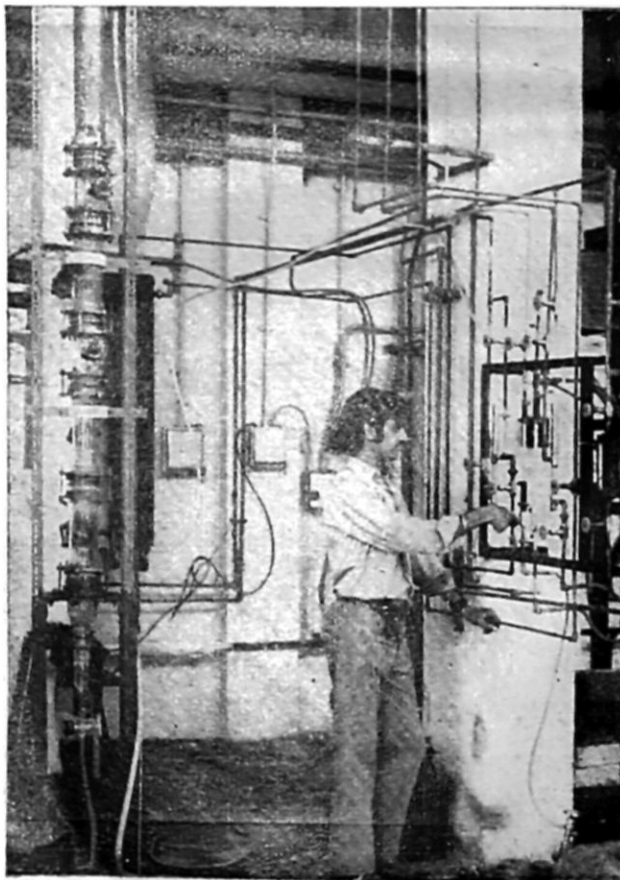


Mosaic Virus Free Co-740 Sugarcane



Pilot Plant at NCL
for the preparation
of Acrylates

Pilot Plant at NCL
for the preparation
of Propylene Oxide



RESEARCH AND DEVELOPMENT PROJECTS

1. PETROCHEMICALS AND BULK ORGANIC CHEMICALS

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

Propylene oxide is not at present manufactured in the country and the demand for a number of products based on propylene oxide is met by imports. This sponsored project has been undertaken to develop the know-how for its manufacture in order to make use of chemical grade propylene that would be available with the sponsor. The project is being worked out in close collaboration with a project engineering firm in order to facilitate its commercial implementation.

Process conditions for the formation of chlorohydrin and its subsequent epoxidation have been obtained on a small scale. Work is now in progress in a specially installed pilot plant.

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

Acrylic acid and its esters find use in several industries, including plastics, paper, paints, textile auxiliaries, etc. The estimated demand for these chemicals is 2-3 thousand TPA (valued at more than Rs. 2 crores) which is likely to double in the next few years.

This sponsored project has been undertaken to develop know-how for the manufacture of acrylic acid and acrylates from acrylonitrile, likely to be available in considerable quantities with the sponsor. Process parameters and engineering data for designing a commercial plant will be obtained. The work is being carried out in close collaboration with a project engineering firm.

Processes for the manufacture of glacial acrylic acid, methyl acrylate, butyl acrylate and 2-ethyl hexyl acrylate are being developed on laboratory and pilot plant scale.

1.3 *Catalytic vapour phase epoxidation of olefins* : (SP-85/72)

Oxides of olefins, especially of ethylene and propylene, are industrially important chemicals which are largely used in the manufacture of glycols, glycolethers, polyglycols, amines, detergents, surfactants, stabilizers and rocket propellants.

The sponsor is establishing this technology for the manufacture of ethylene oxide from ethylene using an imported catalyst. They have sponsored

the project to develop a suitable catalyst so that their present programme and future expansions in this field could be based on indigenous catalyst.

A number of catalysts have been prepared and their systematic screening is in progress.

1.4 1,3-Butylene glycol : (SP-86/72)

This glycol is an intermediate in the manufacture of polyesters and polyurethanes. It is used in surface active agents, plasticizers, humectants, etc.

1,3-Butylene glycol is normally manufactured by the hydrogenation of aldol obtained by the self-condensation of acetaldehyde. A sponsored scheme has been undertaken to develop this technology.

Encouraging results have been obtained in pilot plant runs for aldol formation. Work on standardization of the hydrogenation step is in progress.

1.5 Terephthalic acid (TPA) : (PP-9/69)

Terephthalic acid is used in the manufacture of polyester fibre which is increasingly used in the country. The entire demand is met by imports at present. The estimated demand by 1973-74 is around 58,000 TPA, valued at Rs. 5.8 crores.

Taking into consideration the availability of the raw materials in the country and economics of the alternate processes, the benzoic acid route is being investigated.

The reaction is being worked out in two types of reactors : (1) a stirred reactor with a reduction gear system, and (2) a tray reactor in which the reactants are spread in different thickness of layers and under pressure.

Simultaneously, reaction was carried out in a muffle furnace using different catalysts in varying concentration to find out the time of reaction at atmospheric pressure. NMR analysis has been standardized for determination of di-potassium terephthalate in presence of potassium benzoate.

2. PESTICIDES

2.1 Pentachloronitrobenzene (PCNB) : (ATT-167/70)

PCNB is a soil fungicide for cotton and for vegetables like cabbage, cauliflower, brussels sprouts, potatoes, peanuts, etc. The present import of this fungicide is around 45 TPA. However, the demand is estimated at about 100 TPA valued at Rs. 29 lakhs.

A process for its manufacture has been standardized on laboratory scale and pilot plant runs are being organized.

2.2 *Menazon* : (ATT-198/72)

Menazon is an excellent acaricide and is very effective for control of aphids on tobacco, mustard and other similar plants. This is imported and marketed in India. The demand for this product is about 10 TPA, and with some promotional efforts it could easily be raised to 50 TPA valued at Rs. 50 lakhs.

Laboratory scale synthesis of a key intermediate in the manufacture of menazon has been standardized. Large scale trials on this intermediate and its conversion to menazon will be undertaken.

2.3 *Phenthioate* : (ATT-199/73)

This organophosphorus pesticide is highly toxic to insects but has low toxicity to mammals. The estimated requirement of this pesticide by 1978-79 is nearly 50 TPA valued at Rs. 50 lakhs.

Preliminary laboratory scale work for the manufacture of this insecticide was carried out starting from benzyl cyanide. Further large scale work and optimisation of a manufacturing process is being undertaken under sponsorship from industry.

2.4 *Dichloropropionic acid* : (ATT-200/72)

2,2-Dichloropropionic acid is widely used as a selective weedicide. It is mainly used for the control of weeds in sugar cane, sugar beet, corn, potato and other similar crops. Presently the product is not manufactured in the country. Imports of dichloropropionic acid were 48 tonnes in 1969-70 and 30 tonnes in 1970-71. If the product is indigenously manufactured, the offtake may be expected to be much higher.

Several bench scale experiments using various reaction conditions and catalysts were carried out. The process is being standardized on 2 kg/ batch scale.

2.5 *O,O-Dimethylphosphorodithioate* : (ATT-203/72)

A process for the manufacture of the above common intermediate for organo-phosphorus pesticides such as malathion, menazon and phenthioate has been standardised on laboratory scale. Scale-up work is in progress.

2.6 *Nitrofen (TOK)* : (ATT-205/73)

Nitrofen is a useful herbicide, specially for sorghum, sugarcane and groundnut. The estimated demand by 1978-79 is 250 TPA valued at Rs. 75 lakhs. A laboratory process for the preparation of nitrofen has been optimized and larger runs are now being taken. The product has been found to be satisfactory in field trials.

2.7 *Insect hormones, pheromones and juvenile hormone analogues* : (AB-105/72)

Recent advances in the chemistry and biochemistry of insect hormones have been rapid and exciting, and it has become clear that compounds which mimic insect hormone and pheromone activity may play a great role in the selective control of harmful insect pests.

The work on this project has been undertaken with two objects :—

(i) Screening of plant materials to isolate compounds which may exhibit the desired activity, and (ii) Synthesis of juvenile hormone and insect pheromone mimics starting from intermediate compounds obtained from plant sources and juvenile hormone analogues from the basic raw materials. The attempts at screening of plants for juvenile hormone activity were discontinued for want of testing facilities.

Under the present scheme it has been planned to synthesize compounds having structural features similar to those present in the juvenile hormones and to test the activity of these compounds against several insect pests.

There is also a great potential for juvenile hormone analogues which will have specific activity against different families of insects.

The following known active analogues of insect juvenile hormones were synthesized on laboratory scale:

- (i) 10,11-Epoxy-N-ethyl-3,7,11-trimethyl-2,6-dodecadienamide
- (ii) 7,11-Dichloro-N-ethyl-3,7,11-trimethyl-2-dodecenamide
- (iii) Ethyl-10,11-epoxy-3,7,11-trimethyl-2,6-dodecadienoate
- (iv) Ethyl-7,11-dichloro-3,7,11-trimethyl-2-dodecenoate
- (v) 1-*p*-Nitrophenoxy-6,7-epoxy-3,7-dimethyl-2-octene
- (vi) 1-*p*-Chloroanilino-6,7-epoxy-3,7-dimethyl-2-octene

These compounds are being sent to agricultural research units for entomological screening.

3. DRUGS, PHARMACEUTICALS AND FINE CHEMICALS

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

This drug is known for its analgesic activity.

During the investigations, it was found that only the dextro-propoxyphene hydrochloride possesses analgesic activity. The leavo isomer as its 2-naphthalene sulphonate can be used as an antitissue agent.

Resolution of α -dl-1,2-diphenyl-2-hydroxy-3-methyl-4-dimethylamino butane into the *d* and *l* optical isomers was successfully achieved and the project concluded.

3.2 Vitamin B₆ : (ATT-16/66)

Vitamin B₆ is at present not manufactured in the country. The demand is estimated at 15 TPA valued at Rs. 64 lakhs. A four step synthesis has been optimized and the process has been offered to industry.

3.3 Clofibrate : (ATT-202/73)

Clofibrate is used in the treatment of arteriosclerosis and coronary artery diseases. The present demand is about 2 TPA which is quite likely to increase.

A two-step process for its manufacture was optimized and it has now been released to industry.

3.4 Fine Chemicals Project : (PP-7/64)

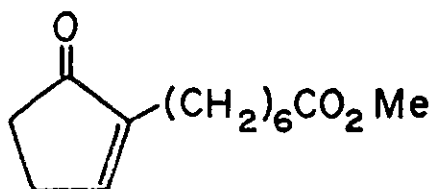
This unit has shown profit of Rs. 16,500 during the year. This pilot plant project is now merged with the normal developmental activity of the laboratory.

3.5 Prostaglandins : (AB-106/72)

Prostaglandins are a family of C₂₀-acids, exhibiting diverse pharmacological properties, the chief among them being muscle contracting activity, which has enabled their use as abortive agents for post-conceptive control in family planning. In addition, they are used to lower blood pressure, prevent blood clots, treat asthmatics and act as long lasting nasal decongestants.

The project has been undertaken with a view to (i) identify inexpensive and easily available raw materials for the preparation of prostaglandins, (ii) to synthesize prostaglandin antagonists and (iii) to develop an economically feasible process for the synthesis of prostaglandins.

Employing oleic acid as a model compound, a sequence of reactions for elaborating the cyclopentane ring (suitably functionalized) on the double bond was standardized. A new practical synthesis of an important prostaglandin synthon—the cyclopentenone, I—was attempted, starting from a readily available fatty acid. In this sequence three steps have been standardized so far.



I

For the synthesis of Corey's intermediates such as lactone of 5-formyl 2,4-dihydroxy-cyclopentane-1-acetic acid, the cis-lactone of 2 hydroxy-cyclohex-4-ethyl-1-acetic acid was successfully prepared. Ozonization followed by reductive cleavage of this lactone furnished an aromatized product which was identified. For the synthesis of the lactone having the required side-chain with appropriate functional group at C₁₆-position of prostaglandin 2-hydroxy-1-(3'-acetic acid, 4'-methoxyphenyl) heptane was synthesized. Further transformation of lactone corresponding to the one mentioned above is in progress.

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

This chemical is used in the manufacture of cosmetic products.

Work on the two-step preparation of the above compound is nearing completion; several batches of the final product have been prepared and sent to the sponsor for evaluation.

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

The objective of the project is to synthesize new compounds which may possess better pharmacological activity. During the period under report 8 new compounds were sent for biological evaluation. Further work is in progress.

4. ORGANIC INTERMEDIATES, DYES AND INDUSTRIAL CHEMICALS

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

Following successful work on the preparation of a blue cationic dye, a process for the preparation of a violet-red cationic dye was standardized. The dye was found to be equivalent to similar commercial cationic dyes in its dyeing and fastness properties. Because of the encouraging results, the sponsor has extended the project for a further period of one year.

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

The objective of this multipurpose pilot plant project was to scale up batch type processes developed in the laboratory. Under this project, large-scale batches on the following were carried out: clofibrate (2 kg/batch), vapour phase chlorination of butadiene, liquid phase chlorination of cyclopentadiene, chlorinated paraffin wax (2 kg/batch) and chlorination of propionic acid (0.5 kg/batch). This pilot plant project has now been merged with other developmental activities of the laboratory.

4.3 *Anthracene* : (ATT-83/68)

A crude mixture containing anthracene (18%), phenanthrene and

carbazole is available from the coal carbonization plants of Hindustan Steel Ltd., Bhilai. Anthracene is mainly required for the production of anthraquinone, a dye intermediate. The estimated requirement of anthraquinone is 3,000 TPA valued at Rs. 5.4 crores.

During the earlier efforts on purification of crude anthracene, preliminary work was conducted to enrich anthracene. Work for the isolation of 95% anthracene (technical) has now been standardized. Carbazole obtained in this process is an important starting material for the manufacture of dye pigments. The know-how will be offered to industry.

4.4 *New disperse dyes and pigments* : (ATT-157/70)

Several new dyes which could be used as disperse dyes for synthetic fibres and which have improved sublimation and other fastness properties were prepared.

Under this programme several new dyes containing reactive species such as arylazido, sulphonylazido and azoacetamido groups were prepared. These dyes could be used as reactive disperse dyes for cellulosic, protein, polyester, polyamide, polypropylene and cellulose acetate fibres and their blends. The work has been covered in a series of patent applications.

New sulphur heterocyclic compounds derived from anthraquinone were prepared. These are intense green compounds. Under this project some work has also been done for the preparation of fluorescent pigments. Greenish yellow, pink and violet hues were prepared and evaluated.

4.5 *Chlorinated paraffin wax* : (ATT-201/72)

Chlorinated paraffin wax is a cheap plasticizer extender mainly used for flexible PVC along with dioctyl phthalate. The product may also find use in fire, mould and water-proofing of canvas, rope and other textiles. The country's requirement in 1972-73 was estimated at 5,000 tonnes. Although the product is being manufactured in the country by several firms, the product available is reported to be unstable and unsatisfactory. A number of firms have shown interest in this project.

A laboratory method (2 kg. scale) was standardized for chlorinated paraffin containing 42% chlorine. The product was comparable to imported commercial samples. Work is being continued for obtaining a product containing a higher percentage of chlorine.

4.6 *Trioxane* : (ATT-204/73)

Trioxane is the key intermediate for the preparation of polyacetal resins.

Trioxane was prepared from concentrated aqueous solutions of

formaldehyde in the presence of an acid catalyst and subsequently purified. Samples of pure trioxane prepared are being tested to find out their suitability for the preparation of polyacetal resins.

4.7 *A new synthesis of chloropheniramine maleate* : (AB-116/72)

2-*p*-Chlorobenzylpyridine is one of the important intermediates for the synthesis of chloropheniramine maleate.

In continuation of our work on a new route for the synthesis of 2-benzylpyridine by the action of phenyl magnesium bromide on 2-chloromethyl pyridine, α -picolyl-2-benzyl pyridine was obtained and characterized. 2-Benzylpyridine, however, was formed only in small proportion. Therefore this route was not found to be economically viable for large-scale production. The work has been concluded.

4.8 *Studies in synthetic dyes* : (B-8.8/62)

(1) *Yellow disperse dyes for polyester fibres* : A number of yellow to orange disperse dyes derived from substituted anthraquinones were prepared. Preliminary studies indicated that they have very good fastness properties. A detailed evaluation is being carried out.

(2) *Benzanthrone* : The mechanism of benzanthrone formation in the standard technical process is under investigation. A new approach to benzanthrone synthesis, not involving the use of glycerol, is also being made.

(3) The preparation and properties of some yellow anthraquinonoid pigments are being studied.

(4) *Sulphur dyes* : Recent work on the NMR and mass spectra of the main constituent of Cibacron Orange R and its derivatives has shown that it is probably a dimer of anthraquinone-2-thioaldehyde.

In this connection, the properties of 2,4,6-triphenyl-s-trithiane were examined, and it was found that all the hydrogens of the heterocyclic ring could be completely exchanged by deuterium by refluxing a dioxane solution with NaOD in D₂O. Hydrolysis then gave α -deuterobenzaldehyde, and a very convenient general method for the preparation of aromatic -d-aldehydes has thus become available.

5. UTILIZATION OF PLANT AND FOREST RESOURCES

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

At the instance of Indian Council of Agricultural Research, work on the constitution of lac was undertaken at the NCL, partly on a sponsored basis. The purpose of this investigation was to elucidate the structure of

organic fragments constituting the lac resin. Over several years, detailed investigations on the constitution of lac resin were carried out and these led to the identification and structural elucidation of several new lac acids (jalaric acid, epishellolic acid, laksholic acids, laccijalaric acid), development of methods for their detection and quantitative estimation, isolation of pure lac resin and a detailed study of its structure. Thus a fairly clear knowledge of the structure of the polymer constituting lac resin has been obtained. The project is now concluded.

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

Longifolene is one of the important by-products from the fractionation of Indian turpentine oil. At the instance of the sponsor suitable commercial outlets for longifolene have been investigated. Thus a number of reactions of longifolene and isolongifolene were investigated and several new derivatives (mostly ketones) were prepared and evaluated as perfumery materials. Of these, one compound based on acylation of longifolene received market acceptability and this material is now being produced by the sponsor on a commercial scale. Another promising derivative from isolongifolene is an isomerisation product of longifolene. This ketone has outstanding perfumery characteristics and is likely to find extensive use in perfumery. Work in the NCL led to standardization of the procedure for the synthesis of this ketone. The work is now concluded.

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

Carene is the major constituent of Indian turpentine oil and the isolation of commercially valuable α -pinene from the turpentine oil, leads to accumulation of large quantities of Δ^3 -carene. Profitable outlets of Δ^3 -carene are under investigation.

Work carried out at the NCL was aimed at two directions Firstly, to develop new perfumery materials from Δ^3 -carene and secondly, to work out an economically viable procedure for the synthesis of menthol from carene. A number of derivatives of carene were synthesized and of these, one is now being produced by the sponsor on a commercial scale. Of the remaining, "Gardinone" appears to have interesting scope as a synthetic perfume.

Preliminary work carried out at the NCL led to the establishment of a method for the successful conversion of carene into (\pm) menthol.

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

The project has been sponsored to assess the commercial viability of solvent extraction process, for obtaining sandalwood oil, in comparison with the conventional steam distillation process.

Several factors were studied for the solvent extraction of sandalwood oil, viz. (a) selection of the solvent, (b) determination of a suitable particle size of the powder (c) purification of the crude oil either by steam distillation or by vacuum distillation. In each of the experiments physico-chemical properties were tested along with olfactory evaluation. Based on these studies, a process for the solvent extraction of sandalwood oil was standardized on pilot plant scale. 100 kg of the crude extracted oil and 7 kg of the standard oil, obtained during process development, have been sent to the sponsor for assessing consumer acceptance. The oil conformed to the specifications required. The project is now concluded.

5.5 *Flavonoids, tannins, stilbenes, lignans and quinones in some Indian forest trees : (SP-76/71)*

The project has been undertaken to isolate and study the flavonoids, tannins, stilbenes, lignans and quinones of some important Indian timber species and make an attempt to relate the natural durability of these timbers to their specific constituents.

A detailed study of the wood phenolics of some Indian timber species led to the isolation of various types of phenolic compounds and their structure determination. A number of these new compounds were screened for antibacterial and antifungal properties.

A number of coumarins and alkaloids were isolated from *Chloroxylon swietenia*, known as East Indian Santin wood. One of the coumarins contained a C-t-butyl ketone which is very unusual in natural products.

From the trunk bark of *Maclura pomiferum* seven xanthenes were isolated, of which three have been already reported from other sources. All the four new xanthenes have C-prenyl groups attached to xanthone nucleus and three of them have undergone oxidative cyclization. One of the xanthenes has a 4,4-dimethyl-chromene ring system not encountered earlier in natural products.

Some *Morus* and *Garceia* species have been worked out and a number of phenolic compounds which were reported previously have been characterised.

A number of phenolic compounds have been isolated and characterized from different species of *Albizzia*, *Picea* and *Tsuga*.

A beginning has been made on the chemistry of tannins occurring in Indian forest trees such as *Terminalia tomentosa*, *Cassia auriculata* and *Tsuga brunoniana*. Attempts were first made to separate monomers, dimers, trimers and higher polymers by gradient elution from polyamide chromatography columns.

From the acetone extract of peanut shells 5, 7-dihydroxy-chromone, a second example of a natural chromone, unsubstituted in the 2,3-positions has been isolated. The occurrence of the chromone, ereodictyol and luteolin indicated the likelihood of their common biogenetic origin.

5.6 Preparation of nitro-musk compounds : (SP-83/72)

There are three artificial musk compounds that are used in perfumery trade. These are musk xylol, musk ketone and musk ambrette. The method for the manufacture of musk xylol and musk ketone was standardised on 1 kg scale. Work on musk ambrette is in progress.

5.7 Synthesis of 1-menthol from citronella oil of Indian origin : (SP-84/72)

Work on the synthesis of 1-menthol from Java citronella oil was carried out earlier on laboratory scale. The sponsored project has been undertaken to (1) study the constituents of the Indian citronella oil and (2) synthesise 1-menthol from dicitronellal obtained from Indian citronella oil.

About 80 kg of Indian citronella oil was subjected to fractional distillation and citronellal rich fraction (95% pure citronellal) having the requisite physical properties, was obtained.

Towards synthesis of 1-menthol, experiments were carried out to cyclise d-citronellal to isopulegol. Further work is in progress.

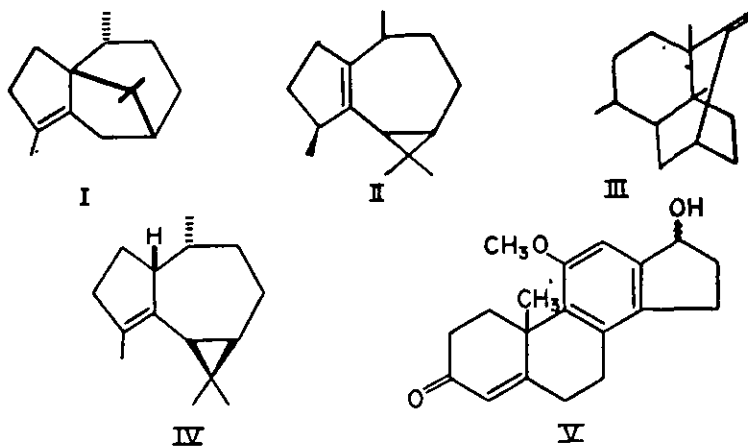
5.8 Chemical examination of essential oils, transformation products and synthesis of terpenoids : (B-7.4/60)

Cyperene (I) a sesquiterpene of well established structure is reported to give two saturated hydrocarbons on catalytic hydrogenation and two diketones on ozonolysis. It has now been shown that cyperene, $\alpha_D^{28} -9^\circ \cdot 29$, is a mixture of cyperene (I) and a new sesquiterpene (II) (See Fig. on page 12).

In connection with structural studies of rotundene and rotundenol, patchouli alcohol and pogostol were required. During processing of Singapore patchouli oil, the hydrocarbon fraction has been carefully examined and shown to contain scychellene (III) and α -gurjunene (IV). Occurrence of α -gurjunene in patchouli oil was not reported earlier.

The structure assigned to the condensation product of citral, β -naphthylamine and pyruvic acid was further confirmed. Additional new compounds have been prepared by using other amines.

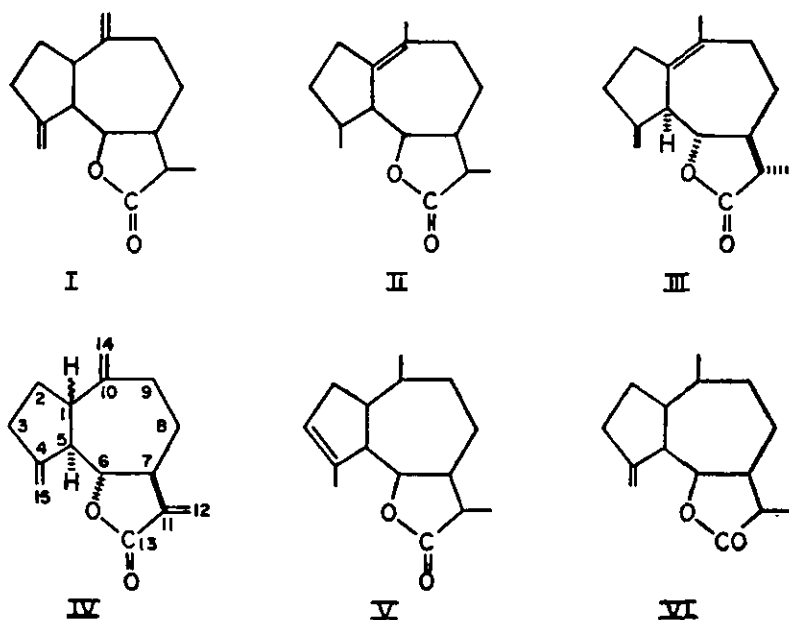
A new synthesis of the 18-Nor-steroid(V) has been completed.



5.9 Transformation products of costunolide and dehydrocostus lactone :
(B-8-15/66 and B-8-19/65)

Dihydrodehydrocostus lactone (I), on partial hydrogenation (Pd/c), yielded a mixture of two dihydroderivatives, of which one compound viz. (II) in which the cyclopentane exo-double bond was reduced and the cycloheptane-exo-double bond was migrated to the tetrasubstituted position, was isolated and characterised. It was found that compound (II) was identical with a compound (III) of known stereochemistry prepared from both arboresin and isophotosantonin lactone-acetate. On the basis of this identity the stereochemistry has been assigned to dehydrocostus lactone (IV) at three of the four asymmetric centres viz. C₅; C₈ and C₇.

When compound (I) was partially hydrogenated using platinum



catalyst, it gave a mixture of four dihydroderivatives of which two, viz. (V) and (VI) were isolated in pure state. Some transformations of (V) and (VI) are in progress.

5·10 *Compounds related to selinane, elemene and p-menthane (Synthesis of epoxides) : (B-8.16/67)*

In view of the presence of epoxide ring in a number of juvenile hormones and insecticides, a systematic study of preparing epoxides of well defined stereochemistry has been undertaken. This has led to the unambiguous preparation of a number of epoxy-p-menthanols.

It was shown that the NMR spectrum could be employed to assign the stereochemistry of oxide bridge of p-menthane epoxides. (\pm)-2 α -Methyl-6-benzylidene-cyclohexan-1- β -ol was prepared and epoxidised with perbenzoic acid; two epoxyalcohols were isolated from the reaction product and the stereochemistry of epoxide bridge was determined.

5·11 *Utilization of β -himachalene : (AB-17/67)*

β -Himachalene is the major sesquiterpene hydrocarbon of the essential oil of *Cedrus deodara* Loud. Though the essential oil itself has sufficient demand, this project was initiated to find out more sophisticated uses for the hydrocarbon portion of the essential oil.

β -Himachalene was subjected to a number of oxygenation/hydration reactions, but unfortunately the products proved to be very complex. Only one reaction (epoxidation) yielded a clean product in good yield and this compound (β -himachalene oxide) was found to have interesting olfactory characteristics.

A number of compounds prepared from β -himachalene or isolated from the essential oil of *Cedrus deodara* have been tested for insect hormonal activity. Though some of the compounds possess mimic juvenile hormone activity, none of these is outstanding. In view of this, further work is discontinued.

5·12 *Cotton textiles and blends with easycare and or fire retardant properties : (ATT-193/72)*

Cotton in the form of yarn or fabric can undergo chemical modifications such as, crosslinking reactions for dimensional stabilization and for easycare properties like soil-repellency, crease-resistance, quick drying and permanent press. Only the accessible hydroxyl groups of the D-glucopyranosyl units in the microfibril of cotton take part in the crosslinking reactions in conventional finishing operations. There is considerable scope for improving these crosslinking reactions, reducing the cost of the reagents and simplifying the finishing processes involved.

The project has been undertaken to study the sites and nature of the cross-links with the selected cross-linking reagents and finally modify the crosslinking reagents and conditions of reactions to have better performance at a lesser cost.

The accessibilities of the three hydroxyl groups at the 2-0, 3-0 and the 6-0 positions of the anhydrous glucose units of cotton cellulose were determined by reacting it with a nitrogen mustard in its active aziridinium form. The substituted glucoses were isolated from the reacted cellulose by hydrolysis, fermentation of the excess unsubstituted glucose followed by filtration through a millipore filter and freeze-drying. The derivatives are now being analysed by GLC after equilibration.

The relative proportions of the three substituted glucose considered together with the reactivity of three individual hydroxyl groups of glucose will give a quantitative measure of their accessibilities in cotton cellulose (print cloth) to the crosslinking reagents.

5.13 *Imparting dry and wet crease recovery to jute fabrics : (ATT-194/72)*

Bleached jute fabrics have good export potential. This will be further enhanced if the properties of dry and wet crease recovery could be incorporated in the fabric. Potential domestic consumption of such finished jute fabric is also likely to be high.

Accessibilities of the hydroxyl groups of the cellulosic part of the bleached jute fabric was studied by reacting it with a nitrogen mustard in its active aziridinium form. Isolated cellulosic and hemicellulosic fractions are also being similarly studied. These studies are likely to give a lead to the optimum conditions for the application of the selected crosslinking reagents and the curing procedure.

5.14 *Reagents for improving the wear life of cotton fabrics : (B-11.5/72)*

Under this project several novel chemical reagents containing reactive groups such as carbonylazido, sulphonyl azido and aminodichloro triazinyl groups were prepared. Such groups were incorporated in long-chain hydrocarbons and fatty acids. These new reagents covalently reacted with cellulosic hydroxyl groups and it is expected that they would provide internal lubrication and reduce the frictional wear and tear of the fabric. The preliminary results are encouraging. A provisional patent application has been filed and further work is in progress.

5.15 *Production of xylit : (SP-81/72)*

Xylit or xylitol is the sweetest of all alditols known so far. It can be obtained by hydrolysis of xylans present in the hemicellulosic fractions of agricultural wastes and subsequent reduction of the resulting xylose.

Xylit has a potential antiketone action and it corrects abnormal metabolism. It penetrates widely into body tissues and is utilized completely by participating directly in the metabolic system. It also promotes biosynthesis of glycogen in the liver and facilitates the utilization of amino acids. It is used clinically for correcting abnormal metabolism in diabetics and for replenishing fluid and sugar.

Xylans of several agricultural wastes were hydrolytically extracted. A process for the technical preparation of xylit starting from cocount shells has been optimized. The project has now been concluded.

5-16 *Dissolving pulps* : (AB-114/73) (Formerly PP-2/58)

A feasibility report for the production of rayon grade pulp from eucalyptus hydrids has been prepared by M/s Chemical and Metallurgical Design Co. Pvt., Ltd., (New Delhi) in consultation with the NCL. This report based on technology developed by the NCL clearly indicates that the project is technically and economically feasible. A turn-key project for the production of rayon and tyre cord grade pulps is now being offered by this firm.

Further work on tyre cord grade pulp from eucalyptus showed that pulps of super—super quality could be produced from eucalyptus.

A mixture of hard woods from Chanda forest of Maharashtra State was investigated for the preparation of premium grade High Wet Modules (HWM) fibre quality pulp. 50 Kg. of this pulp sample was then sent abroad for testing and the test report indicated its suitability for manufacture of HWM fibre. The filterability and consequently the processing of the pulp was found satisfactory. Further work on variation of percentages of different woodspecies in the mixture is being worked out.

5-17 *D-Galactose* : (AB-97/70)

D-Galactose is used in pharmaceutical industries and biochemical research. At present it is not manufactured indigenously. Although indigenous demand is not known, it has some export potentialities as a rare sugar.

Conditions were established to obtain pure-D-galactose from CNS-gum. Work was also conducted to obtain D-galactose in good yield from the aqueous extract of CN shells without isolating CNS-gum. This may reduce the cost of the final product.

5-18 *L-Rhamnose* : (AB-112/72)

L-Rhamnose is one of the important rare sugars required in biochemical research. It is at present imported in India. The requirement of L-Rhamnose in U.S.A. appears to be about 2 to 3 tonnes per annum.

The normal starting material for L-Rhamnose is quercilrin obtained from black oak. The above mentioned sugar is also present in some of the

indigenous gums and industrial agricultural wastes. Several indigenously available natural gums and industrial waste products are being tested as a source for L-Rhamnose.

5.19 *Dulcitol* : (AB-113/72)

Dulcitol can be used in several industrial products such as textile auxiliaries, pharmaceuticals, and surface active agents. The industrial or laboratory use of dulcitol will depend on its cost.

Preliminary work showed that dulcitol could be obtained in about 60% yield from *D*-galactose and also directly from CNS extract.

5.20 *Synthesis of D-fucose* : (AB-111/72)

D-Fucose is an important and costly rare sugar used in biological studies. Several fucosides derived from this sugar are important in studies in genetics. *D*-fucose has good export potentialities with limited internal consumption.

Work on the conversion of *D*-galactose into *D*-fucose in five steps has been undertaken and encouraging results obtained.

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

Some of the glycosides and thioglycosides are of current interest in studies in genetics. These sugar derivatives, some of which are very costly, have a good export potential.

A new method was earlier developed for the synthesis of aryl α -glycosides and thioglycosides from aryl alkyl alcohols. A number of these glycosides were prepared by this new method and exported to USA.

In continuation of these studies, it was observed that the mechanism of the new reaction is more complicated than observed earlier. New leads were discovered in these studies which led to the development of a new and elegant method for the synthesis of β -glycosides. Several β -anomeric glycosides and thioglycosides were prepared in good yields by this method.

Another new approach for a successful synthesis of α -glycosides has been developed and is being explored further. Several new maltosides have been prepared by the above procedure. Their mass spectral fragmentation patterns are being studied for structure elucidation.

5.22 *Utilisation of tamarind kernel powder (TKP)* : (ATT-137/69)

Work on TKP phosphate and borate was completed and is being offered to industry for commercial utilisation.

Work was undertaken on the preparation of thin boiling TKP as a

jute sizing material for putting in higher solid concentration with a relatively small increment of paste viscosity.

Work on evaluation of thin boiling TKP as a jute sizing material is continued.

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

Based on tapioca starch, a product which can be used as substitute for Gum arabic was developed.

The product has been approved by the Post & Telegraph Office Dept. for its use as an adhesive. Its use as a binder in pharmaceuticals and confectionary is also under investigation. The project is now offered to industry.

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

Recently preparation of coal by dry method has been practically discarded and wet washing of coal is being adopted as it can handle wider size ranges of coal with high efficiency. A normal washery circuit contains centrifuges and vacuum filters for dewatering small size coals. The particles below 200 mesh (B.S.) cannot be removed and are gradually built up in the system. If these fines are allowed to accumulate, the quality of the washed product deteriorates. Flocculants are now used as sedimentation aids for removing these coal fines. The estimated demand of coal washery flocculants in the country is about 200 TPA. The indigenous product now used needs improvement.

As a result of collaborative work with CFRI, Dhanbad, a flocculant composition was developed from indigenous polysaccharide raw materials. The product was evaluated by CFRI and found to possess higher flocculation efficiency than the indigenous product. The experimental work on the project is completed and the process will be shortly made available to industry.

6. CHEMISTRY OF BIOLOGICALLY ACTIVE COMPOUNDS

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

The Composite Drug Research Scheme initiated by the Health Ministry, Govt. of India, was formulated with a view to assess the medicinal usefulness of certain indigenous plants reputed to be clinically useful, and make a systematic study of their active principles.

Boerhavia diffusa Noital (Punarnava)

The ethyl acetate extract which showed anti-inflammatory activity was chromatographed to isolate the major component. This is being tested for physiological activity.

Asparagus raceosus Noital (Shatavari)

The major saponin, shatavarin I showed galactotropic and anti-oxytotic activity. The degradation studies and the synthesis of the permethyl derivative helped to arrive at a tentative structure, viz. Sarasapogenin-3-(D-glucopyranosyl (1-4) D-glucopyranesyl (1-4) (L-rhamnopyranosyl (1+2) D-glucopyranoside). To deduce the confirmatory evidence, shatavarin-I was partially hydrolysed to shatavarin-Ia with two sugar units. More of shatavarin-I was isolated to get sufficient quantity of shatavarin-Ia for further work.

Commiphora mukul (Hook ex Stocks) (Guggul)

A large number of steroids were isolated and their structure determined. The major steroid was shown to possess anti-cholesterol activity.

Besides these, diterpenes such as cembrene-A and mukulol and octadecan-1,2,3,4-tetrol and eicosan-1,2,3,4-tetrol were isolated.

Pueraria tuberosa (Vidharikand)

The petroleum ether sub-extract was found to possess oestrogenic activity. Chromatography of this sub-extract is being carried out to isolate the active principle.

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

Lanced poppy capsules contain some alkaloids. It is estimated that nearly 5000 tonnes of lanced poppy capsules are available per year.

A technically feasible method for the extraction of alkaloids from lanced capsules, on laboratory scale, was developed. A method was also worked out for the removal of opium alkaloids from the effluents most likely to be encountered in the New Alkaloid Works, Neemuch.

For the simultaneous estimation of morphine and other alkaloids, an analytical method was standardized using TLC densitometry.

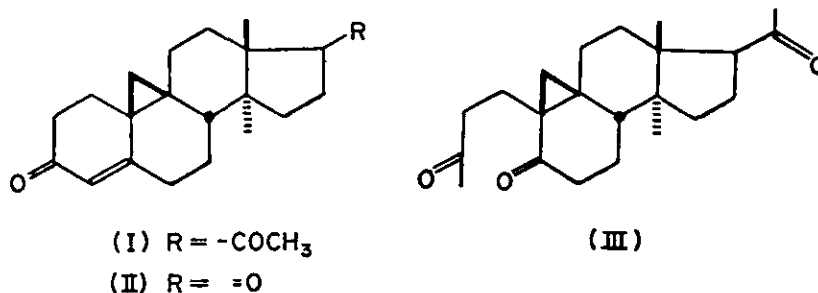
This scheme is now concluded.

6.3 *Utilization of the bye-products of opium alkaloid industry* (AB-21/67)

Opium marc—a waste product of Indian opium alkaloid industry is rich in two triterpenoids namely cyclolaudenol and cycloartenol. These triterpenoids were isolated from opium marc in good yields. The project was undertaken to transform these triterpenoids into hitherto unknown 9, 19-cyclosteroid hormone analogues (I) and (II). These hormone analogues are similar to progesterone structurally, except the 9, 19-cyclopropane ring and

the 14- α -methyl group. Later on, the physiological activity of these hormone analogues will be examined.

An 11-step sequence of reactions for the degradation of cycloartenol was finalized to afford an intermediate compound (III). Further transformation of (III) to (I) is in progress.



6.4 Colchicine : (ATT-68/68)

This is a valuable alkaloid used both in pharmacy and horticulture. It is in short supply throughout the world. Identification of a suitable indigenous source may open up export potentialities for this drug.

A method of extraction, isolation and purification of colchicine from *Iphegenia* species (which have shown appreciable amounts of colchicine content earlier) was standardized on a 3 Kg. batch scale. This was later on scaled up to 7 Kg. seeds per batch.

In collaboration with the Botanical Survey of India, some more seeds of *Iphegenia* (60 Kg), *Lilliaceae* and *Chlorophytum* species were collected. Investigations on the colchicine content of the last two varieties also showed promising results.

6.5 Chemistry of medicines in veterinary use : (ATT-192/72)

Considerable efforts are being expended in the country towards the introduction of new breeds of cattle. In coming years the management of veterinary diseases will acquire a new importance since it has been found that the foreign breeds are more susceptible to indigenous veterinary diseases.

Cedrus deodara oil which is available commercially in considerable quantities has been found to be useful as a complete cure against 'Sarcoptic mange' disease in cattle and sheep.

The essential oil of *Cedrus deodara* is a complex mixture of several components. With a view to identify the component(s) responsible for this activity, several fractions of these components were isolated. Some of these fractions were found ineffective. The testings at various other concentrations

of these component fractions upto 10% concentrations are being pursued. The activity of the more polar groups isolated from Cedrus deodara oil are being looked into.

6.6 Chemistry of plant growth regulators : (AB-104/72)

Plant growth regulators or plant hormones are believed to play an important role in the fields of agriculture, forestry and horticulture. Work on this project was started with a view to study the chemistry and physiological activity of different pollen extracts. Work on *Andropogon sorghum* (Jowar) and *Cucurbita maxima* (Red pumpkin) was undertaken. The ether extract of the pollen from Jowar was fractionated and the various fractions tested for physiological activity by standard assay methods. The plant tissue culture assay system showed that some of the fractions possess growth regulating activity. Further fractionation for the active component was carried out and testing of the individual compounds is in progress.

It is planned to work on the structure and synthesis of active compounds isolated from the above pollen extracts.

7. STUDIES IN ORGANIC SYNTHESIS

7.1 Functionalization of saturated hydrocarbons : (AB-103/72)

Functionalization of saturated hydrocarbons offers many interesting possibilities, specially for the synthesis of alcohols, ketones and acids.

This project is being investigated by two different routes viz. (i) photo-oxidation and (ii) catalytic oxidation.

(i) *Photooxidation* : It involves UV irradiation of saturated hydrocarbons in the presence of hydrogen peroxide or a peracid. Reactions were studied on model compounds like cyclohexane, methylcyclohexane and *p*-menthane. In the case of cyclohexane the reaction product was mainly cyclohexanol while a mixture of secondary and tertiary alcohols was obtained in the case of methylcyclohexane and *p*-menthane.

(ii) *Catalytic oxidation* : Work was initiated on the oxidation of *p*-menthane in the presence of a suitable catalyst. *p*-Menthane is usually obtained from the hydrogenation of dipentene. Conversion of *p*-menthane into oxygenated products was around 30%, as compared to the reported conversion of 10-12%. The reaction product was found to be a mixture of ketones, and secondary and tertiary alcohols.

7.2 Oxidation of olefins : (AB-102/72)

Oxidation of cyclic olefins by molecular oxygen in liquid phase, in the presence of transition metal salts is a valuable reaction of great significance, especially for the synthesis of α,β -unsaturated ketones and alcohols.

The work was initiated with a view to develop a practical method of making α , β -unsaturated ketones and alcohols by the oxidation of cyclic olefins.

Initially, cyclohexene was oxidized under different conditions, to evolve a suitable method. The effects of temperature, catalysts and solvents on the rate of oxidation as well as products formed from cyclohexene, were investigated. The products obtained were further treated to give essentially 3-cyclohexenol or 3-cyclohexenone, thereby making it a convenient process for their synthesis.

Oxidation of different cyclic olefins e.g. cyclopentene, cyclohexene, cycloheptene, and *cis*-cyclooctene was also carried out to determine the effect of ring size on the products formed. A number of compounds containing alkyl substituents on the ethylenic linkage of cyclohexene e.g. 1-methyl-1-cyclohexene, 1-ethyl-1-cyclohexene, 1-isopropyl-1-cyclohexene and 1-tert-butyl-1-cyclohexene were oxidized to determine the effects of substituents on the oxidation pattern.

7.3 Organic reactions in a solid matrix : (B-7.17/72)

Organic reactions in a solid matrix with reactive centres, are of special interest as a crude model for enzymetic reactions. It is planned to study some systems for olefin cyclizations and oxirane rearrangements.

Oxiranes, irrespective of their degree of substitution and other structural features, are quite sensitive to active Al_2O_3 . It is well known that Al_2O_3 contains intrinsic acidic and basic sites, which participate in the reaction. The acidic sites can be neutralized by sodium or potassium ions or by passing bases such as ammonia. Reactions of (+) 2 α , 3 α -epoxypinane with alumina impregnated with different alkalies or salts were studied. The study revealed that (+) 2 α , 3 α -epoxypinane was exclusively converted into *trans*-spino-carveol ($\sim 91\%$) with Al_2O_3 -NaOH, while NaCl doped alumina favoured typical carbonium rearrangements - a behaviour similar to the reaction of this epoxide with silica gel. The reactions of various other epoxides with these aluminas e.g. 2 α -10 epoxypinane and 2 β -10 epoxypinane were also studied.

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

The stereochemistry of the Ramberg and Backlund reaction products obtained by treatment of the vinylogous α -halosulfones with base was studied. Assignments were made on the basis of comparison with the products synthesized by Wittig reaction. Two stereoisomers of the conjugated dienes were isolated by preparative GLC on 30% SE-30 column (10 ft. in length and packed on 30-60 mesh chromosorb W). NMR, IR and UV spectral data confirmed that the compound with lower retention time was *cis* while the one with longer retention time was the *trans* isomer. Mass-

spectrum indicated a molecular weight of 158 in agreement with the proposed formula viz. 1-phenyl, 2-methyl, 1,3-pentadiene for the two isomeric dienes.

7.5 Studies in heterocyclic chemistry : (B-8-7/65)

7.5.1 Studies in hydride transfer

2,3-Cyclohepteno-cycloocteno-cyclohexeno- and cyclopenteno- N-methyl-1,4-dihydroquinolines were prepared and the acid catalyzed disproportionation of these compounds was studied. The examination of the tetrahydro component of the above disproportionation reaction revealed the predominant/exclusive formation of the *trans* isomers in the case of cyclohexeno, cyclohepteno and cycloocteno derivatives. In the case of the cyclopenteno derivative the *cis* and *trans* isomers were formed in 3:2 proportion. These results could be better explained on the basis of product development control phenomenon.

7.5.2 Synthesis of compounds with potential biological activity

The synthesis of phenothiaphosphinic acid was further extended for preparing its benzo and dibenzo derivatives. Attempts are also being made to prepare dithienothiaphosphinic acid derivatives.

7.5.3 Nitrogen heterocyclics

The structure and tautomeric equilibrium shown by the condensation product between arylamines and hydroxymethylene-cycloalkanones (like 3-hydroxymethylene-4, 5-dihydrofuran-2-one, 2-hydroxymethylene-1-tetralone, 3-hydroxymethylenethiochroman-4-one, 3-hydroxymethylene - N-methyl-1, 2,3,4-tetrahydroquinolin-4-ol) were studied in detail. The rearrangement reaction of these arylamino methylenecycloalkanone was further extended to prepare various mono- and diheteroatomic tetra and pentacyclic systems.

Pyrolytic and photolytic ring opening of 2,4-disubstituted N-arylazetidines were studied. They rearranged to give 2,4-disubstituted 1,2,3,4, tetrahydroquinolines. The steric requirement of these reactions are being looked into.

In the attempts for developing new synthesis for 1H-1-benzazepine systems, various quinoline N-oxides were treated with dimethylsulphoxonium methylide. The reaction resulted in dimeric products instead of the desired ring expansion reaction. However in case of 7-methoxy 4-phenyl quinolinium N-oxide gave 8-methoxy-5-phenyl-1H-1-benzazepine when benzene sulphonyl chloride was added to the reaction mixture prior to the addition of dimethylsulphoxonium methylide.

A slow acting herbicide was prepared from saw dust and 2,4-dichlorophenoxy acetic acid. The product is being evaluated for its weed control

effect on wheat. Rate of release of 2,4-D will be determined by mass spectral techniques.

8. PHYSICO-CHEMICAL PROPERTIES OF MATERIALS

8.1 *Photochemical oxidation of benzene to phenol* : (B-3 ·10/70)

Studies on UV photolysis of H_2O_2 in presence of benzene revealed that phenol was formed with no net decomposition of H_2O_2 .

Phenol is also formed in aqueous ferric salt solutions saturated with benzene when exposed to light. The effect of various anions on this reaction has been studied.

In both these cases hydroxymucondialdehyde was formed as a by-product. The reaction mechanism therefore involved OH radicals formed in the primary photolytic step.

8.2 *Radiolytic polymerization of trioxane* : (B-3 ·11/71)

In a situation when an extra amount of used atomic fuel would be available, it could be employed for several industrial uses. One such possible use is for the conversion of trioxane to an industrially important polymer-acetal resin. Radiation polymerization of trioxane has added advantages as compared to conventional chemical process in producing a purer product with simplicity in operations.

A detailed study on trioxane irradiation was undertaken with an objective of arriving at optimum conditions for polymerization, acetylation and stabiliation.

8.3 *Diffusion of cobalt in silver* : (B-2 ·1/67)

An investigation of the tracer diffusion of cobalt⁶⁰ in silver metal in the temperature range 795-925° was undertaken to clarify the existing contradictory results on the impurity diffusion of cobalt in solution. Diffusion coefficient of cobalt⁶⁰ tracer in silver was determined by the serial sectioning technique and was found to vary with temperature according to the equation :

$$D = 115 \cdot 9 \exp \left(\frac{-56829}{RT} \right) \text{ cm}^2/\text{sec.}$$

The observed activation energy of 56.8 Kcal/mole, close to the value for the self diffusion in silver, indicated that the impurity diffusion of cobalt in silver takes place by the formation and movement of the vacancies, in agreement with the results of Hirone et al. [*J Phys. Soc. Japan*, **16**, 455(1961)]. The conclusions of Hirano et al. *Acta Metall.*, **11** ·463 (1963), based on their experimental results of very low energy of activation and frequency factor

for the diffusion of cobalt in silver showing that the diffusion of cobalt in silver takes place through short circuiting paths such as dislocations, appear to be in error, which probably arises owing to their analysis of results by the indirect Gruzin's technique rather than the direct serial sectioning techniques. The direct serial sectioning methods were used to detect regions of the near surface diffusion effects and the diffusion through dislocations from the bulk diffusion.

8.4 Fractionation of turpentine oil : (SP-78/72)

Earlier it was found that turpentine oil obtained from Kashmir contained α -pinene ($\sim 31\%$), β -pinene ($\sim 11\%$), Δ^3 -carene ($\sim 49\%$), longifolene ($\sim 4\%$) and other minor components such as camphene, dipentene etc. ($\sim 5\%$). The sponsors are interested in setting up a plant for the fractional distillation of the turpentine oil available in the Kashmir region.

Highly pure samples of α -pinene, β -pinene and Δ^3 -carene were fractionated from turpentine oil. The conditions for estimation of α -pinene β -pinene and Δ^3 -carene in the binary mixtures were standardized. The measurements of boiling points of the pure components and their mixtures were made. With the data thus obtained, a plant design will be offered to the sponsor.

8.5 Thermodynamic excess properties of binary liquid mixtures : (B-2.13/70)

The heats of mixing at 40° of binary mixtures of n-butylamine with H_2O , methyl alcohol, ethyl alcohol, n-propyl alcohol and n-butyl alcohol, were determined. The results could be expressed by an equation of the form :

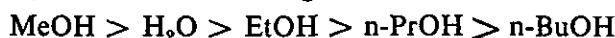
$$\Delta H^*/\text{Cal. mole}^{-1} = X_1 X_2 B + C (X_1 - X_2) + D (X_2 - X_3)^2$$

where X_2 is the mole fraction of n-butylamine and X_1 is the mole fraction of the second component of the binary mixture. B, C and D are constants which have the following values :

(I) n-BuNH ₂ ; C ₂ H ₅ O	B = - 3189.4 Cal. mole ⁻¹ C = - 624.10 mole ⁻¹ D = + 608.70 „
(II) n-BuNH ₂ ; + MeOH	B = - 3625 Cal. mole ⁻¹ C = - 853.29 „ D = + 644.9 „
(III) n-BuNH ₂ ; + E + OH	B = - 2742.4 Cal. mole ⁻¹ C = - 545.52 „ D = + 175.03 „

(IV) n-BuNH ₂ ; -n-PrOH	B = - 2708·2 Cal. Mole ⁻¹
	C = - 286·9 ,,
	D = + 336·2 ,,
(V) n-BuNH ₂ ;n-BuOH	B = - 2618·1 Cal. mole ⁻¹
	C = - 327·9 ,,
	D = + 223·6 ,,

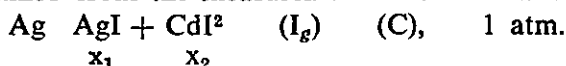
The heats of mixing of n-BuNH₂ with H₂O, MeOH, EtOH, n-PrOH and n-BuOH, follow the following order :



With the exception of the n-BuNH₂ -H₂O system, the decreasing order of the heats of mixing of n-BuNH₂ with methyl, ethyl, n-propyl and n-butyl alcohols might be attributed to the increase in the steric hindrance by the increasing alkyl chain length to the formation of n-BuNH₂ - alcohol complex in the binary mixtures.

8·6 Thermodynamic properties of binary salt mixtures : (B-2·14/72)

Activities of AgI in molten salt mixtures of AgI + CdI₂ were determined from the measurement of the E.M.F. of the formation cell :



The values of the partial molar excess free energy, $G_{\text{AgI}}^{-\text{E}}$, partial molar excess entropy $S_{\text{AgI}}^{-\text{E}}$ and the excess enthalpy $H_{\text{AgI}}^{-\text{E}}$ at mole fractions, 0·874, 0·582, 0·425 and 0·206 of AgI in the molten salt mixtures of AgI + CdI₂ at 823° K were determined. Studies at other temperatures are in progress.

8·7 Thermodynamic properties of complex ions of the transition metals : (B-2·15/70)

The thermodynamic parameters, ΔG , ΔH and ΔS associated with the interaction of transition metal ions Cu(II), Ni(II), Co(II) and Zn(II) with multidentate polyamines, N-(2-aminoethyl)-1,3-propanediamine (denoted as 2,3-tri) and N,N'-(2-aminoethyl)-1,3-propanediamine (denoted as 2,3,2-tet) were determined to find out how the commulative ring strain which developed in the linked 5-membered metal chelate ring system could be relieved by the substitution of one of the linked five-membered metal chelate rings by a six membered metal chelate ring.

It was observed that the enthalpies of formation of the corresponding metal complexes of N,N'-bis-(2-aminoethyl), 1,2-ethanediamine (denoted as trien) and N,N'-bis-(3-aminopropyl)-1,3-propanediamine (denoted as 3,3,3 tet) and the entropies of formation of Cu(II), Ni(II) and Co(II) complexes of 2,3,2-tet were in between the values for the corresponding metal complexes of trien and 3,3,3-tet. It was shown that, whereas, in an octahedral symmetry

of the metal-linear tetramine complexes, the substitution of a central 5-membered chelate ring by a 6-membered chelate ring, as occurs in the linked 5,6,5- [membered chelates of 2, 3, 2]—tet, not only removes the cumulative ring strain, but also compensates for the endothermic effect of the 6-membered chelate ring in relation to a 5-membered ring on the enthalpy of formation. Yet, a similar substitution in the tetrahedral symmetry could only partially remove the adverse effect of the linking of the three chelate rings on the enthalpy of formation.

A comparison of the thermodynamic data on the interaction of transition metal ions with N-(2-aminoethyl), 1,3-propanediamine (2,3-tri) with the existing thermodynamic data on the linked two five-membered and linked two six-membered metal chelate rings formed by N-(2-aminoethyl) 1,2-ethanediamine (2,2-tri) and N-(3-aminopropyl), 1,3-propanediamine (3,3-tri) was made respectively. It was shown that in the metal complexes of 2,3-tri, unlike the corresponding metal complexes of 2,2-tri and 3,3-tri, a chloride ion is coordinated to the central metal ion giving rise to comparatively lower enthalpies and larger entropies of formation as a result of the electrostatic interaction between the Cl ion and the central metal ion. It was also shown that the endothermic effect of the substitution of one of the 5 membered rings by a 6-membered ring has a greater magnitude than the exothermic effect arising from the relief in the strain as a result of the above substitution in the linked, 5,5-membered chelate rings of 2,2-tri, on the enthalpy of complex formation.

8·8 *Studies on adiabatic compressibility of macromolecules :*
(B-2·16/63)

The adiabatic compressibility of a cationic polymer, poly -4-vinyl/-N-n-butylpyridinium bromide was studied earlier along with some anionic polymers such as polyacrylic acid, polymethacrylic acid. This work was extended to study of synthetic polyelectrolytes to copolymer carrying both anionic and cationic groups in the polymer chain.

The two amphoteric polyelectrolytes namely acrylic acid-4-vinyl/-N-n-butylpyridinium bromide copolymer and methacrylic acid-4-vinyl/-N-n-butylpyridinium bromide copolymer were synthesized. The apparent molal volume, ϕV_2 and apparent molal compressibility ϕK_2 were found to be concentration independent for these amphoteric polyelectrolytes. The potassium salts showed the lowest ϕV_2 and ϕK_2 values as the electrostriction was highest here and a slight increase of those values was seen in presence of excess KBr solution (1·0 M) as the dissociation of the ions was somewhat reduced. However, in excess KBr solution, the acrylic acid containing copolymer having comparatively more carbonyl groups (21·7%) behaved more or less similar to poly (acrylic acid) whereas the methacrylic acid containing copolymer with only (15·1%) carbonyl groups behaved closely to that of poly-4-vinyl N-n butylpyridinium bromide. The limiting values of apparent molal volume

ϕV_5 for acrylic acid and methacrylic acid containing copolymers were 183.9 cc/mole and 184.6 cc/mole respectively. These experimentally obtained values fall short of 9.2 cc/mole and 6.2 cc/mole compared to the calculated values (if there was no interaction between acid and base groups which may be due to mutual interaction of acid and base groups in the polymer chain).

The adiabatic compressibility of another cationic polymer, poly-N-N dimethyl aminoethyl methacrylate is being studied. A few amphoteric polyelectrolytes with different amount of this cationic polymer and polyacrylic acid are being synthesized for further similar study.

8.9 Degradation of long chain molecules by ultrasonics : (13.3/63)

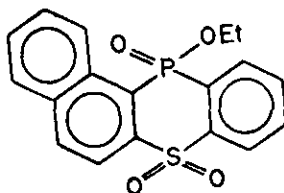
The study of ultrasonic degradation of macromolecules is of importance both in its fundamental and applied aspects. A number of industrially important polymers such as sol rubber, butyl rubber, Neoprene AD and others are being investigated.

The glass transition temperature and melting point of the fractionated as well as ultrasonically degraded polymers were studied by differential thermal analysis (DTA) technique. The calibration of the instrument fabricated in the laboratory is in progress.

8.10 Crystallography : (B-2.5/67)

A very precise determination of the structure of *m*-nitrobenzoic acid was made with the help of about 2000 observed structure factors; the bond lengths were accurate to within 0.01 Å and the bond angles to within about 0.5°. The crystals were monoclinic, space group $P2_1^2$ with $a = 13.22 \pm 0.01$, $b = 10.67 \pm 0.01$, $c = 10.37 \pm 0.01$ Å; $\beta = 91.2 \pm 0.1^\circ$, $\rho_c, 1.519 \text{ g.cm}^{-3}$ P_c for $Z = 8$, 1.517 g.cm^{-3} . The structure had two crystallographically independent molecules which formed dimers. A comparison of the molecular dimensions of *m*-nitrobenzoic acid with those of *p*-nitrobenzoic acid showed that the distribution of bond lengths in the aromatic ring with respect to the nitro group is similar in the two structures; these results implied that in both molecules, the nitro group interacts with the aromatic ring to a much greater extent than the carboxylic group.

A precise structure analysis of N, N-dimethylantranilic acid was



completed with the help of about 1000 observed structure factors. The crystals were monoclinic, space group $P\frac{2_1}{n}$ with $a = 7.66$, $b = 15.74$, $c = 7.58$ Å; $\beta = 100.0^\circ$ P_c for $Z = 4$, 1.218 g.cm⁻³. The molecules existed in the form of zwitterions; there was a strong hydrogen bond between the COO⁻ and NH(CH₃)₂⁺ groups in the molecule.

The preliminary structure of the following compound was solved at the suggestion of the synthetic organic chemistry group with the help of about 2000 structure factors. The structure had crystallographic disorder; the study of the disorder is in progress. The molecule is folded about the S-P direction, i.e., the central ring is in the boat form. The refinement of the structure is in progress.

8.11 *Spectrochemical studies* (B-5/63)

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 were investigated. Except the N-dimethyl anthranilic acid, all the compounds were found to be neutral molecules in the solid state. The N-dimethyl anthranilic acid was found to be zwitterionic in the solid state as has been reported earlier. The neutral structure for the N-methyl and N-dimethyl-*m*-aminobenzoic acid was found in sharp contrast to the zwitterionic form of the *m*-amino benzoic acid.

The NN-dimethylanthranilic acid which existed as a zwitterionic molecule in the solid state was found as a neutral molecule in carbon tetrachloride, chloroform, benzene, cyclohexane and dimethyl sulphoxide. Detailed IR and NMR investigations pointed to a strong intramolecular charge transfer hydrogen bond in the solid changing over to strong intramolecular hydrogen bond (without charge transfer) in the above solvents.

Infrared spectral analyses of 3(2'-arylamino methylene) tetrahydrofuran 2-ones in solutions of different solvents and at various concentrations showed that they exist as a mixture of two intramolecularly hydrogen bonded N-H...O and O-H...N tautomers. The equilibrium was found to be solvent dependent but was not appreciably shifted in the temperature range between 30 and 60° C. The close similarity of the solid state spectrum to that in solution suggested the existence of two tautomers in solid also.

8.12 *Dielectric properties of organic compounds* : (B-2.6/69)

From the dielectric constant, density and refractive index values of long chain organic compounds with extended polar groups, the dipole moments were evaluated by the standard Onsager-Kirkwood relations. The

values were compared with the vapour state and solution data in order to discuss their molecular structure.

8·13 *The nature of active sites on synthetic zeolites* : (B-2·8/72)

The high catalytic activity of synthetic zeolites is due to the presence of acid sites. In order to understand, quantitatively evaluate and correlate this phenomenon, a series of rare earth zeolites were synthesised. The DTA, TGA and X-ray analyses of a few zeolites were carried out. The adsorption properties and surface areas are being evaluated.

8·14 *Polyesters as stationary phases in GLC Analysis* : (AB-33/63)

This is a continuous project and development work is undertaken along with routine analysis of samples.

In continuation of the studies of polyester stationary phases, it was observed that the substitution of basic group in the aromatic nuclei of the polyester helps in the separation of halogenated benzene isomers.

These findings were utilised in actual qualitative and quantitative analyses of chlorinated products in continuous chlorination processes using different catalysts.

9. STUDIES IN PHYSICAL ORGANIC CHEMISTRY

9·1 *Conformational studies* : (B-8·45/69)

Conformational studies on the carbonyl group of tertiary amides were reported earlier. Study of the structure and conformations of some bridged triterpenes was undertaken.

A novel 13, 28-epoxide structure has been suggested for a number of triterpenes of the β -amyrin group. An unambiguous synthesis of at least one of these compounds was deemed desirable to confirm this structural feature and study its conformation. Preparation of the corresponding ether from oleanolic acid was taken as a model. The acid was converted to the known 13, 28- γ -lactone with dry HCl and was then reduced with BF_3 etherate and LAH to give the desired ether m.p. 229° , $(+)\text{D} + 2^\circ$. Echinocystic acid was then converted to the known diacetate bromolactone and was then reduced with BF_3 etherate and LAH. Extensive chromatography of the product gave a neutral compound $\text{C}_{30}\text{H}_{50}\text{O}_3$ which had the same R_f value, m.p. and rotation as the authentic natural sample.

9·2 *Studies on conjugated systems* : (B-5·2/62)

Proton chemical shifts of a series of 4-substituted phthalic anhydrides were determined in an effort to get supplementary evidence for the interpre-

tation advanced for the systematic variations in chemical shifts observed earlier in 4-groups of sterically hindered benzene derivatives. A consideration of the shifts in the anhydrides suggested that the larger part of the variations observed would be attributable to conformational changes.

Measurements of rotational barriers in some of the BF_3 -adducts of aromatic aldehydes prepared earlier are under way in the hindered groups.

9.3 *Studies of chemical reactivity* : (B-5.10/70)

(a) *Reactivity of o-hydroxyaryalmides* : In continuation of earlier work a detailed study of the properties of these compounds was undertaken. The anions of a number of these were investigated. In order to obtain necessary back-ground information, the difference between the behaviours of salicylic ester and 2-hydroxy-3-naphthoic ester in their reaction with DNCB in methanol was studied.

(b) *Reactions of acyl derivatives with tert-amines* : Some preliminary experiments were carried out using acetic anhydride and triethylamine. The main products of reaction were identified, and are being studied further.

(c) *Reactivity of malathion towards alkali and alkaline hydroxylamine*: In connection with some biochemical studies on malathion, Hestrin's ferric hydroxamate method was proposed for the rapid colorimetric assay of the substance. It was observed that change of solvent from water to 33% methanol nearly doubled the colour yield in the reaction. This remarkable fact attracted attention and the reasons for the observed behaviour were studied in detail.

An analysis of spectrophotometric and kinetic data along with a product study with the help of NMR spectra showed that in the methanolic medium the first stage of the reaction was elimination of dimethyl O, O-dithiophosphoric acid to give fumaric diester which leads to a dihydroxamate. In water, as solvent, the hydroxamation reaction was faster than elimination. Malathion thus behaved as a typical succinic ester and gave a monohydroxamate in this medium.

(d) *Oxidative transformations of aromatic and unsaturated compounds* Some preliminary work on hydroxylation of aromatic compounds using hydrogen peroxide and peracetic acid was carried out. An effort was made to understand the actual sequence of reactions involved and the roles of the catalysts used. More work is required to be done for a further understanding of the transformations.

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

Molecular orbital studies on isomeric forms of the acyl cation were carried out using the CNDO/2 approximation.

9.5 *Mass spectrometry* : (B-5.7/65)

9.5.1 *Electron impact and pyrolysis : similarities and differences.*
In continuation of the previous work in this area the behaviour of epimeric 3,5,6-triphenyl 4-amino Δ^1 -cyclohexenes was studied under electron impact and pyrolytic conditions. Unlike the nitro analogues which showed different electron impact and pyrolytic behaviour, the amino compounds showed similarities. This has been explained on the basis of Woodward-Hoffman orbital symmetry rules.

9.5.2 *Electron impact, chemical ionisation and field desorption spectral studies:* A comparative study of the processes operating in epimeric 3,5,6-triphenyl-4-nitro- Δ^1 -cyclohexenes was made under electron impact, chemical ionisation of field desorption conditions. Significant differences were observed in the fragmentation reactions. Many interesting novel observations were made on chemical ionisation, and field desorption behaviour.

9.5.3 *Studies on exo and endo 7-azabicyclo (2.2.1) heptanes:* The effect of various substituents at 2,3 and 7-positions on simple cleavage and double hydrogen transfer reactions was studied by high resolution and at low electron volts. Localization of charge on nitrogen led to α -cleavage reactions, which are dominant in the spectra. Very little differences were observed in the spectra of the *exo*- and *endo*- compounds.

9.5.4 *Substituent effects on mass spectral reactions:* The effect of substituent on retro-Diels Alder reaction was investigated at different electron voltages. The results were sent for publication.

9.6 *Mass spectral techniques* : (B-5.8/65)

9.6.1 *Ion kinetic energy (IKE) spectroscopy* : Necessary modifications of the mass spectrometer to record IKE spectra were made. This technique will be used to analyse isomeric compounds whose mass spectra are similar. The subtle differences in the fragmentations of isomeric compounds will be evident in the ion kinetic energy spectrum.

9.6.2 *Quantitative analysis by mass spectral techniques* : The ion current integration method for the quantitative analysis of organic compounds in complex mixtures was standardized. The sensitivity of the method is one nanogram. Efforts are being made to improve the sensitivity. The method has potential applications in product analysis connected with processes development work in pesticide residue analysis and in measuring the degree of pollution of air and water.

9·6·3 *Pesticide residues in food, air and water* : Using gas chromatography and mass spectral techniques, analytical methods were standardized for the estimation of (1) malathion residues on wheat grain (2) lindane and carbaryl residues in straw, rice and bran and carbofuran residues in bhendi.

10. INDUSTRIAL POLYMERS, RESINS, ELASTOMERS AND SYNTHETIC FIBRES

10·1 *Expandable polystyrene* : (ATT-34/66)

Polystyrene foam is a versatile light weight plastic material which finds application in packaging, low temperature thermal insulation and as a structural material. Polystyrene beads are prepared by suspension polymerization technique. It is presently manufactured in the country based on imported know-how. The objective for undertaking this work was to develop indigenous know-how.

The process conditions were standardized to obtain 90% of the foamable beads having desired characteristics. Physical characteristics such as thermal conductivity, compressive strength and density of the sheets prepared from these beads were found to be comparable to similar product already in market. The above experiments were standardized on a pilot plant scale and the know-how will soon be offered to industry.

10·2 *Polymers for oil well drilling* : (ATT-115/68)

At the instance of Oil and Natural Gas Commission, work was undertaken to develop a suitable substitute for an imported product known as 'Halad'. The chemistry of 'Halad' has been elucidated and a process for its preparation is nearing optimization.

10·3 *Nylon 12* : (ATT-188/72)

Nylon-12 which possesses high dimensional stability at extreme temperatures, exceptional toughness, impact strength, chemical resistance, etc. is a very useful product in the manufacture of several kinds of extruded and moulded articles.

Methods for oligomerization of butadiene to 1,5,9-cyclo-dodecatriene using singular type catalysts such as diethyl-aluminium chloride and titanium tetrachloride were standardised giving 75-80% yields.

Experimental conditions for hydrogenating the cyclic triene to cyclo-dodecane in the presence of palladium-charcoal as catalyst were established yielding 85% of the final product.

It has been decided to discontinue further work on this project, as

butadiene which is the basic starting material is in short supply in the country and is not likely to be available for this purpose for several years to come.

10.4 *Polyacetals* : (ATT-196/72)

Polyacetals are linear thermoplastic polymers of formaldehyde or trioxane. They are good substitutes for non-ferrous metals and find applications in varied types of end uses e.g. gears, bushings, pulleys, speed meters etc. Formaldehyde, the basic raw material, is indigenously available, but these polymers are not yet manufactured in the country. According to one estimate, indigenous demand may be around 1000 TPA valued Rs. 2 crores.

Laboratory experiments on catalyst induced polymerization of trioxane were undertaken.

10.5 *Synthesis of resins for friction materials* : (SP-82/72)

Use of phenolic type resins is known for a long time as binder for friction materials. It has also been reported that cashew nut shell liquid (CNSL) can also be used to make binder resins. However the products when prepared using CNSL were found to have some undesirable properties. The project has been undertaken to study the chemistry of CNSL resins with a view to improve the properties of friction materials made therefrom.

Various aspects of CNSL reactions to form resins are under investigation.

10.6 *Ketone resin* : (ATT-187/72)

Ketone resin, a condensation product of cyclohexanone and para-formaldehyde, is used for imparting protective and glossy surface finish to paper, wood, metal and in printing inks. Its present imports are of the order of 250 TPA valued at Rs. 25 lakhs.

It was possible to prepare the resin having required softening range, but its solution viscosity was found lower as compared to similar imported product. Experiments on the preparation of resin of higher molecular weight are in progress.

10.7 *Polyurethane coating compositions for textiles and other substrata*: (ATT-46/64)

Polyurethane coatings which possess excellent flexibility at low temperatures are used to impart waterproofing or water repellancy properties to different substrata. The process know-how developed for coatings for nylon, wood, leather, metal, etc. has been offered to industry.

To improve the colour and odour of formulations developed earlier, it was decided to extend the work for the preparation of colourless and

odourless polyurethane formulations. These coatings would be useful for textiles, rain wears and umbrellas. A laboratory process was optimized and further work on larger runs is in progress.

10·8 *Stabilizers for PVC* : (ATT-161/70)

Earlier Ba-Cd type PVC stabilizer composition was developed. Organo tin compounds such as dibutyltin octate, dibutyltin maleate and dibutyltin laurate are also used for the stabilization of PVC. These stabilizers impart high heat stability and give transparent PVC products. According to one estimate the demand for various organo tin stabilizers will be around 200 TPA valued Rs. 120 lakhs.

Process conditions for the preparation of dibutyl tin oxide (DBTO) were standardized. Based on DBTO, processes for dibutyl tin laurate and dibutyl tin maleate (liquid) were also standardized. These products were comparable to imported products. The know-how for the manufacture of organo tin stabilizers is now being offered to industry.

10·9 *Nitrile rubber* : (ATT-52/67)

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

The present imports of nitrile rubber are of the order of 500 TPA valued at Rs. 50 lakhs. The demand is likely to be much higher if the product is indigenously manufactured.

Medium grade

Process conditions were standardised for the preparation of medium nitrile content rubber (34% bound acrylonitrile content).

High and low grades

Experimental conditions for the preparation of these rubbers are being established. A process for low grade nitrile rubber having $28 \pm 1\%$ acrylonitrile content was also standardised on a laboratory scale.

A firm has shown keen interest in acquiring the NCL know-how on nitrile rubbers.

10·10 *Sulphochlorinated polyolefin elastomers (SCPE)* : (ATT-90·1/68)

Sulphochlorinated polyolefin elastomers possess high outdoor weatherability, heat resistance and chemical resistance in addition to toughness and

abrasion resistance. These rubbers are used in a variety of ways to impart protective coating to different substrata. The estimated demand of SCPE in the country is around 200 TPA valued at Rs. 50 lakhs. Efforts are being made to assess the indigenous market for different grades of SCPE.

One step process for the production of general purpose SCPE was standardised on a small scale and efforts are being made to scale up the know-how.

10-11 Chlorinated polyethylene (CPE) : (ATT-90-2/71)

Chlorination of polyethylene improves the impact strength of rigid PVC. It is also used as a vulcanizable elastomer for cable covering, for ozone resistant rubber goods and as a flexible, tough thermoplastic. These products are not yet manufactured indigenously.

The chlorination of powdered polyethylene is under investigation.

10-12 Stereospecific polymerization : (B-13-1/60)

Stereospecific polymerization of methyl methacrylate with chromium acetylacetonate and aluminium alkyls (triethyl aluminium and triisobutyl aluminium) was reported earlier. The work was further extended to study the stereospecific polymerization of methyl methacrylate with homogeneous Ziegler-Natta catalyst systems such as cobalt acetylacetonate with the same aluminium alkyls in benzene medium. The kinetic studies showed the free radical behaviour of these systems in benzene medium, which was also confirmed by heteroblock structure of polymethyl methacrylate obtained.

The previous studies on polymerization of methyl methacrylate in polar solvents like acetonitrile proved the possibility of obtaining stereospecific polymethyl methacrylate. In continuation of this work, different solvents were used for polymerization of methyl methacrylate with Ziegler-Natta catalyst system. It was observed that methyl methacrylate could not be polymerized with Ziegler-Natta catalyst systems such as 1) VC_4-AlEt_3 , Br, 2) $VOCl_3-AlEt_2$ Br in CCl_4 medium. However it was observed to be vigorous in CCl_4 medium and black stable complexes were separated when poured in acidified methanol. The stable Ziegler-Natta catalyst complexes gave new complexes when allowed to react with methyl methacrylate. The kinetic studies of complex formation of methyl methacrylate with these catalyst systems are being carried out. The work on characterization of these complexes by IR, NMR and elemental analysis is in progress.

To compare the behaviour of these catalyst systems in CCl_4 medium the same catalyst systems were used to study the polymerization in hydrocarbon medium.

10·13 *Polymer characterization*

10·13·1 *Thermodynamic properties* : The new Bond Energy/Group Contribution method for calculating the standard heats of formation, (Pt III) in respect of alicyclic hydrocarbons has been published. Halocarbons are being correlated and bond-energy terms derived for Pt. IV of the general scheme.

10·13·2 *Copolymerization* : A critical review of methods of calculating monomer reactivity ratios, with discussion and evaluation of the new computation procedure developed earlier has been communicated for publication. Further work on formulation of new computer programmes for various methods of deriving copolymerization parameters is in progress.

10·13·3 *General methods* : Several thermograms of the standard polymeric material such as polyethylenes, chlorinated and sulfochlorinated polyethylenes, polyacetals, etc., were obtained from the general DTA/TGA apparatus.

10·13·4 *Solution properties of polymers* : An empirical method of estimating the second virial coefficient of polymers from solution viscosities has been suggested. The method gave satisfactory values as checked with available data for polystyrene in toluene and methyl ethyl ketone and polyvinyl acetate in acetone and methyl ethyl ketone.

11. INORGANIC CHEMICALS AND CATALYSTS

11·1 *Cadmium pigments* : (AB-81/70)

Cadmium sulphoselenide pigments are used in plastic, ceramic and printing ink industries because of their fastness and heat and alkali resistance properties. These pigments have been recently introduced in the Indian market. It is estimated that the total annual consumption of these pigments would be around 50 TPA valued nearly Rs. 30 lakhs. No reliable figures for import of these pigments are available.

Processes for preparation of orange, yellow and red shades of cadmium pigments were standardized on laboratory scale starting from cadmium salts available in the market. The pigments were found to be satisfactory in field use.

11·2 *Pearl pigments* : (AB-91/71)

Substances, which have a high refractive index and are in the form of thin plate-like crystals are commonly used to produce a nacreous mother-of-pearl effect, are called pearl pigments. The optical effect can be achieved on different substrata like plastic, leather, artificial pearls, etc., either by coating the surface of the object with a vehicle containing such crystals or by incorporating the crystals within the light transmitting substances.

The major synthetic inorganic pigments currently in use are lead carbonate, bismuth oxychloride, lead phosphate, lead arsenate, etc. The important requirements are particle size, smooth shape, insolubility in vehicles and plastics, and thermal stability during utilization.

The demand of pearl pigments is estimated around 100 TPA valued at Rs. 120 lakhs..

Laboratory preparations of bismuth oxychloride were optimised.

Several experiments were carried out to prepare lead phosphate and basic lead carbonate. Samples of different pigments have been sent to a firm for evaluation.

11.3 *Sodium hydrosulphite* : (ATT-187/72)

The Indian textile industry requires about Rs. 2 crores worth of sodium hydrosulphite annually. At present, it is manufactured using zinc dust which is imported. Development of an alternative route via sodium formate is being explored which may save import of zinc to the tune of Rs. 50 lakhs per year. This process was standardised on laboratory scale. Two firms have shown interest in purchasing this know-how.

11.4 *Fumed silica (White carbon)* : (ATT-197/72)

Fine fumed silica is employed for a number of applications ranging from adhesives and sealants, plastics, rubbers, printing inks, cosmetics, pharmaceuticals, textiles to thickening and thixotropic agents. Figures of import are not available, however, it is expected that there may be a good market if the product is indigenously available.

Investigations aimed at developing technology for fumed silica are progressing satisfactorily.

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

Silicon tetrachloride is used in the manufacture of ethyl silicate and fumed silica. The demand is expected to be around 2000 TPA valued at Rs. 20 lakhs. Starting from ferrosilicon a laboratory method to yield silicon tetrachloride was developed earlier. For scale up of this work a mechanically stirred reactor to chlorinate 2-3 kgs per hour of enriched ferrosilicon to yield silicon tetrachloride was designed, fabricated and erected. As the reactor could not be operated continuously due to mechanical difficulties, a fluidized bed reactor was installed and operated successfully at a product rate of 6 kg/hour. After collecting the necessary process and design parameters, the project will be offered to industry.

Starting with silicon tetrachloride, the preparation of ES-40 (Ethyl silicate with 40% silica by weight) was standardised. On completion of the

pilot plant work on silicon tetrachloride pilot plant runs on ethyl silicate will be undertaken.

Ethyl silicate is used as a binder in precision casting and as a bonding agent for commuted materials. The demand for ethyl silicate is estimated to be around 100 TPA valued at Rs. 8.5 lakhs.

11.6 *Iron oxide pigments* : (AB-79/70)

This project was referred to the laboratory by several firms having a disposal problem of by-product ferrous sulphate and by firms dealing in synthetic oxide pigments.

Iron oxide pigments are gradually gaining popularity on account of cheapness, range of possible shades and fastness of colours.

The preliminary investigations have shown promising results. Further work will be undertaken if sponsored.

11.7 *Molecular sieves* : (AB-107/72)

Molecular sieves are used for drying, adsorption and selective separation of gases and liquids. The synthetic zeolites (molecular sieves) of the type 3A and 4A have already replaced alumina and silica gel as laboratory/industrial dehydrants due to their superior properties. The 5A type synthetic zeolite will be in good demand for the purification and separation of petroleum gases with the growth of the petrochemical industry in the country. According to one estimate the projected demand of all types of molecular sieves is likely to be of the order of 300 TPA valued at Rs. 1.5 crores.

Synthesis of molecular sieves of type 3A, 4A and 5A was carried out in several batches and their physicochemical properties such as X-ray diffraction, surface area and adsorption examined. The adsorption properties indicate that these products are equivalent to or even better than imported products of parallel specifications. Attempts will be made to evaluate the samples made in the laboratory by users of the imported products.

12. MINERAL RESOURCES UTILIZATION

12.1 *Conversion of bauxite into anhydrous $AlCl_3$* : (SP-70/71)

The project was undertaken on sponsored basis with the objective of utilizing hitherto unutilized bauxite ore resources for the manufacture of anhydrous aluminium chloride. Anhydrous aluminium chloride has a sizable export potential.

After completing the laboratory trials in 3 inches diameter reactor, the work was scaled up to about 2 kg of aluminum chloride per hour. Based on the experiments conducted, the necessary technical data was supplied to the sponsor.

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

Under the sponsored scheme conditions were standardized on pilot plant scale (1 kg. TiCl_4 /hr.) for the chlorination of bauxite residue available from the ferric alum plant.

After completing the laboratory trials first in a 3" and later in a 9" dia chlorination reactor, followed by a demonstration run, the requisite technical data was supplied to the sponsor and the scheme is now concluded.

12.3 Recovery of chlorine from flue gases of vapour phase oxidation of TiCl_4 : (AB-108/72)

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 reuse.

Initial experiments were carried out using only chlorine gas and CCl_4 as absorption medium, later synthetic mixtures of chlorine, nitrogen, oxygen, etc., were used and their absorption in CCl_4 studied. Solvents like sulphur monochloride and trichloroethylene were also tried as absorption media.

Sulphur monochloride was found to absorb about 25-30% by weight of chlorine at room temperature. The absorption efficiency decreased with increase in temperature. By providing suitable cooling arrangements the absorption efficiency could be increased to about 45% by weight.

Trichloroethylene was found to be a very good solvent for the absorption of chlorine, but since formation of higher chloro derivatives of ethylene took place, desorption and recovery of chlorine did not appear practicable.

Desorption studies with sulphur monochloride showed that 95 per cent of the chlorine absorbed in this solvent could be easily recovered by thermal splitting of the chlorine rich solvent at 120° . During thermal splitting however, owing to high vapour pressure of S_2Cl_2 about 15% of the solvent loss was observed, which could be minimized by providing proper cooling arrangements.

12.4 Recovery of HCl from iron chloride liquors : (AB-109/72)

During the studies on enrichment of ilmenite by carbothermic reduction and acid leaching, it was observed that the process would be economically viable if the HCl available from iron chloride liquors could be recovered and recycled.

Initially the work was carried out with ferrous chloride prepared from iron scrap. Later leach liquors from beneficiation of ilmenite were used. Ferrous chloride crystals were dehydrated in a current of hydrogen using

either a vertical or a horizontal reactor. Optimum conditions for the dehydration and decomposition of ferrous chloride were worked out. HCl gas formed was recovered by scrubbing with water. The recovery of HCl and resultant iron powder was of the order of 90-92%. Samples of iron powder obtained were analyzed, and sent for testing to industry. It was found that the iron powder is acceptable to industry with certain modifications regarding flow characteristics and stability.

13. ORGANO-METALLIC COMPOUNDS

13.1 *Silicone and silicone intermediates* : (AB-93/71)

Silicone industry is estimated to have an approximate turnover of Rs. 100 lakhs per year. There are few firms who manufacture silicone consumer products from imported silicone intermediates. Silicone intermediates are not manufactured in the country.

A laboratory method for the synthesis of methyl chlorosilane mixture was standardized from upgraded ferrosilicon and methyl chloride. A new method for the separation of dimethyl and monomethyl components from a mixture of dimethyl dichlorosilane and methyl trichlorosilane was established and a dimethyl silicone low polymer was obtained.

During the pilot plant studies to convert about 20 kgs. per. batch of ferrosilicon to Rochow liquid (a mixture of chlorosilanes) several experiments were conducted in a suitably designed gas - solid reactor. It was found that the product composition considerably varied even though the reaction conditions were maintained identical. At the end of each experiment minor modifications were introduced in the pilot plant. The change in product composition was traced to a drawback in the formation of contact mass. The procedure adopted for preparing this mass is therefore being modified.

Some exploratory experiments on laboratory scale were tried for the synthesis of methyl bromosilanes so as to assess the comparative merits of the process with the chlorosilane process. The components of the mixture of methyl bromosilanes could be easily separated by simple distillation and dimethyl dibromosilane fraction could be hydrolyzed to the dimethyl silicone low polymer. The tentative cost calculations showed that this process would be economically viable if bromine could be efficiently recovered and used for making a commodity product like methyl bromide. Dimethyl dibromosilane obtained in the process was of high purity.

13.2 *Tin, titanium and lead organics* : (AB-99/72)

Synthesis of dibutyl tin oxide used in organo tin stabilizers for PVC was successfully developed. It was followed by successful preparation of PVC stabilizers such as dibutyl tin dilaurate, maleate and mercaptides.

Several other organo tin and titanium compounds are being studied for their potential uses in the fields of stabilizers, agrochemicals, etc.

A novel method for making di-organotin oxides was standardized.

Some colourless compounds containing both tin and titanium were prepared and tested for their stabilizing action on PVC. Results were encouraging. Compounds containing titanium and other metals such as copper, mercury, aluminium, are being prepared for studying their properties.

Phenyllead (IV) chelates and chelated lead (II) compounds were prepared. The chelating ligands used were salicylaldehyde, acetylacetone and 8-hydroxyquinoline. These compounds showed good stability and catalyst properties.

13.3 *Tin organics as agrochemicals* : (AB-100/72)

Organotin compounds have been widely used as fungicides. Triphenyltin hydroxide and acetate are used to protect tobacco, coffee and potato crops. Another tin organic, tributyl-tin oxide is known to be an effective wood preservative. Import figures for these fungicides are not readily available. According to NCST estimates, projected consumption of organo tin fungicides in 1973-74 is 30 T valued at Rs. 15 lakhs.

The project has been undertaken to develop technical preparation of the known tin fungicides and to develop new organotin compounds which could be used as agrochemicals. Preparation of triphenyltin hydroxide and acetate from triphenyltin chloride was standardized on laboratory scale. Other routes for these preparations are being explored. Benzyltin compounds which may be expected to show fungicidal properties have also been prepared.

Samples are being sent for user trials.

13.4 *Syntheses of aluminium alkyls* : (AB-101/72)

Aluminium alkyls are required as polymerization catalysts and for the synthesis of long chain alcohols. The technology is quite sophisticated because of the hazardous nature of the chemical.

Exploratory experiments on the preparations of dimethyl aluminium chloride, monomethyl aluminium dichloride, diethyl aluminium chloride, monoethyl aluminium dichloride, trimethyl aluminium and triethyl aluminium were carried out on laboratory scale with encouraging results.

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

In continuation of the previous work on the reactivity of chelated ligands, a number of compounds derived from substituted acetoacetanilides

such as, bis-(3-bromo-2', 5'- dimethoxyacetoacetanilidato)- beryllium(II), bis-(3-nitro-3'-chloroacetoacetanilidato)-beryllium (II), etc., were isolated and studied. The mass spectral data for the beryllium (II) chelates showed well defined break-down patterns and some ion rearrangement reactions. An interesting feature of the break-down pattern of the beryllium (II) chelates was the loss of the anilide group. The species formed after losing the anilide group again underwent cleavage yielding the fragment $(BeL)^+$ through the loss of $CH_3COCHCO$. These cleavages were supported by meta stable peaks in each case. The general mode of fragmentation of substituted (bromo-nitro-), chelates was essentially the same as that of the unsubstituted chelates.

New chromium (III) chelates, such as tris-(2', 5'- dimethoxy-4'-chloroacetoacetanilidato) chromium (III), tris-(acetoacetyl-diethylamidato) chromium (III), etc., were also isolated, which failed to undergo electrophilic substitution reactions, such as bromination and nitration. In the electronic spectra of the chromium (III) chelates the d-d transition band was located at $\sim 17500\text{ cm}^{-1}$. The observed magnetic moments for the chelates were in agreement with the other reported values for octahedral Cr(III) complexes.

New chelates of 2-hydroxy-1-naphthaldehyde with some group IV elements were synthesized. The chloro chelates, except the silicon compound were found to form adducts of the type $M_2Cl_2 \cdot X.NH_3$ ($M = Ti, Sn$ or Zr ; $X=4$) and $ML_2Cl_2 \cdot XCH_3NH_2$ ($M = Ti, Sn$ or Zr ; $X=2$). The infra-red spectra of these compounds showed two sharp bands in the $3300-3050\text{ cm}^{-1}$ region ascribable to the -NH stretching frequency. The adducts were found to lose the ammonia or methylamine molecules when heated above 80° .

The recent reports on the attractive technological potential of some cyclopentadienyl-metal complexes in areas such as, radiation protection, combustion catalysis, polymerization acceleration and rubber vulcanization have considerably enhanced interest in the syntheses of this class of compounds. In an attempt to synthesize new cyclopentadienyl complexes of titanium, particularly of mixed type, investigations were carried out on the reactions of bis-cyclopentadienyltitanium (IV) dichloride with a variety of organic ligands. Direct reactions with some β -diketones resulted in the substitution of one chlorine atom of the dichloride by the ligand. The dichloride was found to react with sulphur containing ligands, such as mercaptans, dimercaptans, mercapto acids, and mercaptoamines in presence of ammonia gas, both the chlorine atoms undergoing replacement by the ligand without causing any damage to the cyclopentadienyl groups. Of particular interest were the reactions between the dichloride and the sulphur containing ligands mercaptobenzoic acid and toluene 3,4-dithiol in presence of divalent metals zinc, magnesium and tin, which yielded some novel bimetallic complexes containing both the cyclopentadienyl and the sulphur containing ligand groups. Some titanoxane polymers containing the cyclopentadienyl

and other organo groups were also isolated. IR NMR and mass spectral studies were carried out in an attempt to characterize the newly synthesized compounds.

In an attempt to synthesize new complex forming macro-molecular compounds which could selectively concentrate a single metal from multifarious elements, the condensation reaction between *o*-aminothiophenol and terephthaldehyde was carried out. The reaction resulted in the formation, of an heterocyclic compound, involving ring closure. The isolated compound terephthaldehyde bis-(2-mercaptoanil) was characterized by elemental analysis, IR and mass spectral data.

In the IR spectrum of this compound, there was no band in the region 2100^{-1} corresponding to an -SH stretching frequency and spectral bands due to -NH group were present. The mass spectrum showed a well defined molecular ion m/e 348. The ligand was found to rearrange in alkaline medium and form bright coloured complexes with certain metal ions. The mercury (II) and cadmium (II) chelates were isolated in pure condition. However, the orange red zinc (II) complex could not be isolated in pure state. TGA and DTA data for the isolated chelates showed that these compounds decompose rapidly in the temperature region 275° - 300° .

13.6 *Surface adsorption of long chain paraffin metal sulphates :* (B-6.7/71)

The micellar properties of sodium, magnesium, calcium, manganese, cobalt, copper and lead dodecyl sulphates in aqueous solution were studied by light scattering and viscosity methods. Similar to light scattering data, the reduced viscosity of the salt solutions showed a distinct change above a certain level, the critical micelle concentration (CMC). The different metal salts were found to have nearly the same CMC values which are much lower than that of the sodium salt. The molecular weights were determined by light scattering measurements and due correction was made for the charge effect. The highest molecular weight was observed in lead salt. The aggregation number for different bivalent metal salts was more or less the same. The intrinsic viscosity for different salt solutions except that of the sodium was seen to have the same value in aqueous solution. The micellar volume and surface area, free energy change and thickness of ionic atmosphere was calculated and the values were analyzed to find out the effect of metal ions on the critical micelle concentration of monovalent and bivalent metal dodecyl sulphates.

14. SOLID STATE MATERIALS

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

Potentiometers of various types are indispensable components of all electronic instruments. The commonly used carbon track potentiometer has

a comparatively short life, and is prone to damage from humidity, dust and temperature. The use of tin oxide tracks as the resistive element has several advantages, such as : very low and consistent noise level; imperviousness to moisture; wide operating temperature range; negligible voltage coefficient of resistance; excellent high frequency performance and excellent tight-design tolerance.

In addition to the technical advantages mentioned above the tin oxide tracks are likely to have several economic advantages, in terms of size and lower cost of production when produced on mass scale.

The Bhabha Committee report had predicted a total demand of Rs. 3 crores worth potentiometers both of carbon film and wire-wound types by 1975. The demand for tin oxide potentiometers will depend on the cost of the material and its capability of replacing carbon track and wire-wound potentiometers in a vast number of applications.

Spraying techniques for the deposition of tin oxide glass (rod and flat) and ceramic (rod) substrates were standardized. A smooth transition from a metallic end contact to a semi-conducting tin oxide film was also achieved earlier.

The resistance to length (of the track) relation was found to be linear. Several batches of tracks, 10 at a time, were prepared on flat glass substrates with different doping compositions. The temperature coefficient of resistance (TCR) measurement of these tracks showed encouraging results (<100 ppm./ $^{\circ}\text{C}$) consistently. The voltage coefficient of resistance (VCR) and wattage of these tracks were found to be quite satisfactory. Thus all the requirements of a potentiometer have been achieved on a laboratory scale.

A rotating disc type conveyer has been designed to get about 100 tracks/batch. This is being fabricated and will be tried.

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

Recording tapes are now manufactured in the country using imported iron oxide compositions which are used for magnetic coating. According to the Electronics Commission the present estimated requirement of these compositions is around 10 TPA valued at Rs. 1 lakh. By 1975-76 the demand is likely to rise upto about 50 TPA valued at Rs. 5 lakhs.

A process to prepare the required grade of gamma ferric oxide was standardized. The magnetic properties of the product are found to be comparable with those of an imported sample.

The units producing magnetic recording tapes have been approached for evaluation of the NCL product.

14.3 *Synthetic gemstones* : (ATT-172/71)

Synthetic gemstones such as ruby and sapphire find a variety of uses such as, jewel bearings in watches, instruments, water and electrical meters and armaments. The gemstones are also used in jewellery for which a large gem-cutting cottage industry exists in the country. The requirements of this trade are estimated to be about 50 TPA.

Synthetic gemstones are prepared in a special oxyhydrogen burner (Verneuil furnace). A prototype of this unit was fabricated incorporating some modifications and innovations. Gemstones prepared in this unit were tested and found satisfactory by a commercial firm.

A new equipment suitable as a prototype for commercial production was fabricated and installed. Improvements in the high temperature refractories required for the unit are under way.

14.4 *Photoconducting materials* : (AB-5/62)

Know-how for CdS photoconducting cells based on single crystals has already been developed and released to industry. Layer type CdS photo cells which would have a larger area and hence higher power dissipation capacity are now being developed.

14.5 *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, technology for these products is being developed.

14.6 *High permeability ferrites* : (AB-90/72)

It is believed that pot core ferrites which are also known as high permeability ferrites are imported in large quantities at present. They are used in radio and television sets, telecommunication instruments, extra high tension transformers, low loss filters, etc. Attempts are being made to assess the demand for such ferrites in the country.

Preparation of very pure and active oxides, and their reaction with one another in an inert atmosphere to give the manganous-zinc ferrite, was reported earlier.

Several batches for the reacted powder required for these ferrites were prepared on laboratory scale. Following properties of the sintered toroids were found reproducible. They are comparable with those of imported ferrites.

(a) $Q \sim 90$ at 1-10 KHz.

(b) Initial permeability ~ 3000 at 1-10 KHz.

- (c) Resistivity ~ 100 ohm cm.
- (d) Density ~ 4.9 gms. per cc.

It is proposed to prepare pot cores from the above toroids which will then be sent for commercial evaluation.

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

A number of semiconducting glasses of the system $V_2O_5-P_2O_5-Tl_2O_3$ were studied. The phosphorus content was kept constant and thallium was introduced in increasing amounts at the expense of vanadium. Resistivity-temperature, thermo e.m.f.—temperature and piezoelectric measurements were carried out.

From the electrical properties and chemical analysis, it was established that conduction was due to hopping of carriers between vanadium ions in different valence states located at equivalent sites in the VO_6 octahedra. Apart from this, it has been seen that thallium is taking part in conduction just as vanadium does. EPR measurements showed the absence of any signal due to paramagnetic species of thallium showing the presence of the lower valence state of thallium Tl^+ .

A number of crystalline vanadium phosphates were also prepared to see how the proportion changed from the ordered to disordered structure. These also showed NTC of resistance. As was expected for hopping conduction, the conductivity showed a tendency to pass through a max. at the $V^{5+}/V^{4+} = 1$. It was found that lowering of conductivity was not so much due to the disorder in the glass, but was due to the V-V distances being increased because of phosphorus dilution. Good agreement was obtained between the values of V^{3+}/V^{5+} obtained from thermo e.m.f. and chemical analysis. The piezoelectric property was slightly more pronounced in the crystalline material. It was found that the presence of hydrogen bonds is responsible for the piezoelectrical properties.

14.8 *Amorphous films of transition metal oxides and sulphides* : (B-2.9/72)

An easy method of producing amorphous films of semiconducting materials directly from solutions was worked out. This method was standardized for making thin films of oxides (ZnO) and sulphides (CdS).

Thin ZnO films were prepared by heating at different temperatures. It was observed that ZnO was amorphous upto 400° and then a slow transition takes place to crystalline state and was completed at 850° . Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) studies revealed that two major weight losses at 125° and 850° could be associated with the endothermic reactions. Optical absorption in the UV range showed a shift in absorption edge towards a longer wave length as the film preparation

temperature increased. A major change in the log R vs. T characteristics was observed from the amorphous to crystalline state. It was found that the ambient conditions had no effect on the electrical properties of the amorphous films, whereas they did have effect in altering the electrical characteristics of the crystalline films.

The CdS films prepared by this method had cubic structure as established by electron diffraction technique. All conventional methods gave films having hexagonal or mixed (cubic+hexagonal) phases. The electrical conductivity in the dark from -100° to $+100^{\circ}$ was studied for these films revealing the presence of the traps at the depth of 0.03 to 0.05 eV, 0.9 eV matching the values reported in the literature. It was found that these films were photosensitive having max. response at 430 nm. Optical absorption edge at 470 nm was recorded. Photoconductivity relaxation curves at different light intensities showed the presence of shallow traps at a depth of 0.02 to 0.05 eV.

This study revealed that the new method of preparation of thin films will be a useful tool to study the transition from amorphous to crystalline states in oxide films. Further, it also revealed that the films had properties different from those prepared by other methods.

14.9 Ternary semiconducting compounds : (B-2.10/72)

In recent years, there has been increasing interest in new semiconducting compounds with diamond like structure. These substances have extensive potential applications as materials for semiconductor devices due to the special properties of the tetrahedral covalent bonds characteristics of the entire crystallo-chemical group of diamond. Bonds of this type create the best conditions for electron transfer, a phenomenon lying at the basis of the operation of semiconductor devices. By a proper choice of the elements, it is possible to prepare new semi-conducting compound. with predetermined properties. Materials with direct band gap are of special interest for optical studies. The present trend in research is to form solid solutions between binary compounds or to prepare ternary compounds.

Ternary semiconducting compounds such as CdSnAs_2 were successfully prepared. CdSnAs_2 is a member of II-IV-V₂ series in the polycrystalline state. Although the importance of the study of thin films of these ternary compounds had been realized, their preparation had not been possible owing to the disproportionation of the compound during evaporation. For the first time, films of stoichiometric composition were prepared and characterized by X-ray diffraction and electrical properties. These films when deposited on cold substrates are non-crystalline and crystallize only after baking at 400° . Thus this has opened up an avenue for correlation of electrical and transport properties of semiconductors between the amorphous and

crystalline states in a systematic manner. There is a lot of interest in understanding the band structure in the amorphous state.

A study of electrical and optical properties like electrical conductivity, thermo electricity, Hall effect, infra-red transmission spectra, etc., of both crystalline and non-crystalline thin films was carried out. The observed band gap of 0.26 eV measured optically and electrically was found to change rapidly with change in carrier concentration in the conduction band. This rare phenomenon is possible if the effective mass of the electron is very low. This indicates high mobility which may render these semiconductors useful for several applications.

14.10 *Amorphous magnetic materials* : (B-2.11/72)

It is proposed to study magnetic properties of the amorphous transition metal oxides and to investigate experimentally the consequences of breakdown of the Neel's model.

Studies on electrical conductivity, curie temperature, TGA, DTA, chemical analysis and X-ray patterns of amorphous gamma ferric oxide showed the absence of a regular crystalline structure; however, it showed quite interesting magnetic properties.

The oxide was prepared by the action of NH_3 and $\text{Fe}(\text{NH}_4\text{SO}_4)_2$ solution in thin films. The material collected was dried and this remained amorphous upto 300° . It became magnetic at 300° , still remaining amorphous and continued so upto 525° . On further heating to 540° it became crystalline and non-magnetic. X-ray pattern showed it to be $\gamma\text{-Fe}_2\text{O}_3$.

DTA, TGA results matched fairly with the above observations. Saturation magnetisation and electrical measurements were carried out. Chemical analysis and Mossbauer spectra proved that the material was $\text{Fe}(\text{OH})_3$ (heated below 300°), which further became $\gamma\text{-Fe}_2\text{O}_3$ and magnetic but amorphous.

Interesting results were obtained on cobalt and iron oxide mixture. This mixture, where cobalt varied from 1 to 25% remained magnetic and slowly became non-magnetic and at 35% cobalt it was completely non-magnetic.

14.11 *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.

A sample based on the composition (modified PZT-lead zirconate titanate) reported earlier was sent to CBERI, Pilani, for evaluation. The

bimorphs prepared with these ceramic strips have the desired high capacitances. Further they were tested by inserting them into commercial cartridges. The output from the strips was somewhat lower than that of the imported ceramics but the fidelity of the sound was quite high. Efforts are being made to improve the material and to standardize the sintering process to have the materials of reproducible properties. The relevant piezoelectric properties of the material are being investigated.

14.12 *Thin film sensors* : (AB-9/62)

Thin films are extensively used in microelectronic circuits as components such as resistors, capacitors, etc., in memory systems, as sensor elements for measuring temperature and also magnetic fields. During the last few years several organizations such as Indian Space Research Organization, Thumba, Institute of Tropical Meteorology, Poona, Physical Research Laboratory, Ahmedabad, and others suggested that the technology of thin film sensors be developed as these have specialized applications in various fields not covered by bulk thermistors.

Several thin film sensing elements were prepared. Some typical elements had the following resistance-temperature characteristics:—

S. No.	Resistance			Approximate TCR %/°C
	-80°	25°	+80°	
33	950 M Ω	18 M Ω	2.5 M Ω	-3.0
35	400	8 M Ω	1.5 M Ω	
84	—	75 K Ω	17 K Ω	
89	—	31 K Ω	6.7 K Ω	-2.4
904	—	4.4 K Ω	1.1 K Ω	

In response to enquiries, several organizations requested for the sensors for testing. These were sent to some organizations and the reports received from them are quite encouraging.

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

Studies on structures, crystal growth process, phase transition, etc., of vacuum-deposited films formed on various substrates at different temperatures were made by electron diffraction and microscopy methods. The majority of the compounds were of group III-V, chalcogenides of cobalt

and indium and oxides of bismuth and other metals, etc. It was shown that thin films of ZnS type structure of group III-V compounds could be obtained by thermal evaporation technique, provided substrate temperatures were appropriate instead of the usual triple temperature technique of Gunther. The conditions for epitaxial growths were standardised and the structural features and their orientations determined on various compounds.

14.14 *Physics of thin films* : (B-4.4/67)

In continuation of previous work of dielectric, optical and semiconducting properties of thin films, various oxides, sulphides and selenides were investigated. Niobium films formed by Vacuum evaporation of Nb_2O_5 were studied for their dielectric and structural properties. Dielectric constants of these films varying from 12-130 were found to be thickness dependent. Effects of frequency (10^2 — 10^5 Hz) and temperature (788 - 383°K) on capacitance and loss factor were also studied. $\tan \delta$ showed a minimum with the frequency and at about 1 KHz it was found to be oscillatory with temperature. The structural study showed that the deposits formed at high substrate temperature developed two phases viz. cubic ($a = 53.8 \text{ \AA}$) and tetragonal ($a = 5.38 \text{ \AA}$; $c = 4.22 \text{ \AA}$) growing epitaxially with appropriate orientations on the cubic face of NaCl. The refractive index and extinction coefficient of these films were also measured. A.C. properties of ZnS film capacitors were studied at different temperatures (78 - 380°K) and frequencies (10^2 - 10^5 Hz). Dielectric constant was found to be independent of frequency unlike the case of niobium oxide films. Capacitance, though dependent on temperature and frequency became constant at low temperature region for all frequencies. Loss factor showing a pronounced minimum with frequency increased with the rise of temperature and the loss factor minimum shifted to the higher frequency. Breakdown voltage was also measured. A proposed model of equivalent circuit predicts some characteristic features of the capacitors which were experimentally verified. Refractive indices of these films were measured for visible regions and the electric constants evaluated for optical frequencies. Effects of substrate temperatures on these films were also studied.

Studies were also made on semiconducting properties of tellurium, bismuth, antimony, bismuth oxide, InSb and chalcogenides of thalium at different temperatures ranging between 73 - 550°K . Electrical parameters viz. resistivity, activation energy, Hall coefficient, mobility, carrier concentration thermoelectric power, TCR, etc., were measured. In the case of tellurium it was found that the mobility followed $T^{3/2}$ law at lower temperature region instead of the usual $T^{-3/2}$ law valid for lattice scattering. These results were explained from the consideration of factors such as discontinuities, imperfections, grain growth, phase changes, etc.

Photoelectric properties of cadmium selenide were measured by the decay method and trap depth was calculated for various film thicknesses.

Optical parameters such as refractive index, extinction coefficient, absorption coefficient, etc., cuprous sulphide ($\text{Cu}_{1.8}\text{S}$) films were obtained from reflectance, transmittance, R_s and R_p data for various film thicknesses (350-3000 Å) and wavelengths of light (4500-7000 Å). Dielectric constant and optical energy band gap were also evaluated from these data.

Optical properties of bismuth suboxide, bismuth oxide and tin sulphide films were also determined using both normal as well as oblique incidence methods. In the case of bismuth suboxide, both n and k were found to be quite high and the former varied from 2.9 to 3.9 and the latter 1.5 to 2 with wavelength. Bismuth oxide films were found to be almost transparent and their refractive indices varied from 1.7 to 2.7, whereas k was very low with a minimum of about 0.1. In the case of tin sulphide films it was observed that n varied considerably with film thickness and wavelength say from 2.1 to 4 and k was found to vary from 0.24 to a very low value with increasing wavelength.

15. INSTRUMENTS TECHNOLOGY

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

These spectrometers which are useful in analytical and research work are presently imported. Cost of the imported instrument is around Rs. 2-3 lakhs. It is estimated that at least 100 such units per year will be required in the country.

Various subassemblies fabricated during the last two years were connected together and their relative performance was evolved. A few assemblies required minor modifications while magnet power supply had to be reconstructed.

Klystron power supply was modified to match with disposal 2K25 Klystron and the magnet pole gap was widened to accept newly built cavities. The widening of pole gap resulted in the drop in field strength. Necessary modifications in the power supply are being carried out. Modulator and sweep assemblies built during the year are undergoing final tests.

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

The programme of the work on designing and fabrication of the laboratory proto-type involved procurement of several optical components, many from indigenous sources and some from abroad. The basic optical layout has been worked out and the spectrometer is being assembled.

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

Further progress was made in the preparation of a prototype infra-red spectrometer.

15.4 *Solid state strip chart recorder* : (ATT-195/72)

Recorders are required for various purposes e.g. temperature recording, process monitoring, analytical machines, etc. Earlier, know-how for a 'Valve' recorder was developed and released to industry.

An improved solid state strip chart recorder has now been developed and is under evaluation.

A solid state chopper substitute for electromechanical chopper has also been successfully developed and will be incorporated in the improved recorder models.

16. TISSUE CULTURE STUDIES

16.1 *Plant tissue culture* : (AB-96.1/72)

The growth of whole plants from callus, pollen or endosperm of plant cells offers a new method of raising plantlets different from conventional sexual reproduction. Somatic hybridisation in the same species or between different species of plants opens up possibilities for the development of totally new types of plants.

The project includes fundamental work on the establishment, differentiation and hybridisation of plant cells of agricultural importance and the application of these studies to agriculture.

During the last few years rice, wheat and jowar were obtained as viable cultures and whole plants obtained from cultures of wheat and maize.

Sugarcane variety CO 740, which is extensively grown in Maharashtra is a high-yielding strain but it is almost invariably infected with mosaic virus. Methods were standardised in the laboratory for obtaining sugarcane plants by tissue culture methods and the plants were then transferred to soil in the green house. Over 90 per cent of the plants obtained by this method were free from the characteristic symptoms of the virus. 50 virus-free plants have so far been obtained and a second generation of 30 plants is now being grown in the NCL garden and at the Nimbkar Agricultural Research Institute, Phaltan. Field trials are in progress to determine whether the virus-free plants give significantly higher yield of cane sugar, tillers, etc.

Hybrid cabbage tissue cultures were established and two whole plants were obtained from these cultures and successfully grown in soil. More plants are being grown to obtain sufficient material for field trials.

Wheat plants, which were obtained from callus tissue, were also successfully grown in the farm and the seeds obtained from them were used

for raising a second generation of plants. Several of these were found to differ markedly from the parent plants from which the tissue cultures were obtained.

Protoplasts were obtained from several different plant cells and work on somatic hybridisation is in progress.

During the course of work on acylphosphatase several inhibitors of proteolytic enzymes were found to be present in the crude extracts of the seeds. Six of them were obtained in pure form and four are being purified. A proteolytic enzyme present in tissue cultures of *Vigna catjang* was also found to be inhibited by a protein inhibitor in the seed extract.

16.2 *Insect tissue culture* : (AB-96.2/72)

There are very few satisfactory antiviral and antitumour compounds available. The project on insect tissue culture is undertaken to study hormones and pheromones at the level of tissue culture with an object of studying possible hormone interactions leading to insect control.

Growth of vesicles as well as fibroblasts was observed *in vitro* from embryonic tissue of rice moth (*Corcyra cephalonica*).

17. ENZYME CHEMISTRY AND TECHNOLOGY

17.1 *Matrix-bound enzymes* : (AB-97/72)

Enzyme technology and its applications in industrial processes are still relatively undeveloped. The main limitations in the direct application of isolated enzymes are their instability and the difficulty in recovering the enzyme for reuse. A novel procedure for overcoming these difficulties is insolubilizing by covalently binding the enzyme to solid support materials. Such matrix-bound enzymes hold promise as elegant industrial catalysts since they can be manipulated easily, removed readily from the reaction solution and reused repeatedly. The development of stable insolubilized enzymes is likely to make an impact on several industrially useful chemical processes.

Although commercial use of matrix-bound enzymes is not yet reported it has been recognised as a new and promising area of research and development.

Several supports for the immobilization of industrial enzymes such as penicillin acylase were investigated and some of the active and promising systems are being studied in detail.

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

The bacterial phytase which was obtained in purified form was found specific for inositol polyphosphates and had no action on other phosphate

esters. The enzyme was ultracentrifugally homogeneous but showed two bands on acrylamide electrophoresis. Both bands had enzyme activity and are probably isoenzymes. They were present even in the crude extract. The purified enzyme contained no phosphorus or inositol. The conversion of inositol hexaphosphate to the triphosphate was rapid whereas the hydrolysis of the latter to the monophosphate was relatively slow. Inositol monophosphate could not be acted upon by the enzyme.

17.3 DPNase : (B-12.4/68)

The DPNase of ox brain, which was solubilized by lipase treatment, was obtained in purified form. Work on the kinetics and properties of the enzyme was completed. Several compounds were tested for their effect on enzyme activity or on the inhibition of the enzyme by nicotinamide but none of them had any effect. This project has been concluded.

17.4 Acylphosphatase : (B-12.7/70)

A specific acylphosphatase was obtained in ultracentrifugally homogeneous form from *Vigna catjang*. Its amino acid composition was determined. It was shown to have 3 half-cysteine groups and 1 buried -SH group which reacted only after prolonged incubation with DTND. The enzyme was sensitive to *p*-chloromercuribenzoate and oxidized glutathione, whereas reducing agents and H₂O₂ had no effect.

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

Microorganisms play an important role in maintaining the nitrogen cycle. The activity of denitrifying bacteria accounts for substantial losses of nitrate-containing fertilizers. With isolated enzyme systems biochemical studies have been undertaken to understand the basic mechanisms involved in these denitrifications.

The Stokes' radius of pure nitrite reductase was determined by different methods. A value of 35.5 Å for Stokes' radius was obtained from gel filtration experiments using Acker's equation. Similar values were obtained by the methods of Laurant and Killander, and Porath. A value of 6.05×10^{-7} cm² sec⁻¹ for the diffusion coefficient ($D_{20,w}$) was calculated from Stokes' radius using Stokes'—Einstein equation. The frictional ratio (f/f_0) was found to be 1.26 from gel filtration data.

The values for molecular weight obtained by different methods were: Archibald approach to equilibrium method, 80,000; using Stokes' radius and sedimentation coefficient, 81,000; from diffusion and sedimentation coefficients, 81,000; polyacrylamide disc gel electrophoresis, 80,000. However, a value of 68,000 for the molecular weight was obtained from gel filtration data from the plots of elution volume (V_e) versus log molecular weight. This strongly supported the suggestion of Siegel and Monty that the elution of

the protein in gel filtration depends on its Stokes' radius rather than the size of the protein. A value of 94,000 reported earlier for ultracentrifugally pure nitrite reductase could probably be due to the impurities present as the enzyme was found polydisperse on disc gel electrophoresis.

Disulfide content of nitrite reductase, determined by estimating total sulfhydryl content (-SH and -S-S-groups) before and after reduction of protein with sodium borohydride in 8 M urea, indicated the presence of one disulfide group per mole of enzyme. Similar results were obtained when total sulfhydryl content was determined as cysteic acid after performic acid oxidation. The enzyme was shown to contain 4 sulfhydryl groups. No loss in activity was observed after all four sulfhydryl groups were titrated with *p*-hydroxymercuribenzoate and 5,5'-dithio-bis-(2-nitro benzoic acid) suggesting that the -SH groups were not involved at the active site.

18. FERMENTATION PROCESSES

18.1 *National Collection of Industrial Microorganisms* : (AB-96/72)

The National Collection of Industrial Microorganisms now consists of 1600 non-pathogenic yeasts, bacteria and fungi which are supplied free of charge to research institutions and industries. 200 new cultures were added to the collection during the year and 731 cultures were sent to different institutions in the country. Isolation and identification of cultures for screening programmes for applied projects were carried out.

18.2 *Acid stable amylase* : (AB-67.1/68)

Among the many industrially useful enzymes which are at present being mostly imported, amylase is one which is much used in pharmaceutical compositions. The acid stable amylase is incorporated into digestive compositions to enhance the activity of the enzyme at the prevailing pH of the stomach.

Six strains of *Aspergilli* which showed stability and activity at pH 2.5 were once again confirmed to have positive saccharifying activity at pH 2.5. It was found that crude fermented liquors when subjected to concentration *in vacuo* at a temperature upto 45° did not show any appreciable loss of enzymatic activity; most of the activity could be precipitated with two volumes of alcohol from the concentrated fermented liquors. Solid enzyme preparations were made from different strains and their activities estimated and compared with commercially available imported enzyme samples. On the basis of available laboratory data, approximate cost calculations for the enzyme preparations were made and compared with the cost of the commercial samples. It showed that the yield of enzyme has to be considerably improved before the laboratory preparations could be taken up for possible industrial use. A few UV-mutation studies carried out

to enhance the enzyme yield in the strains under examination were not successful. Further studies to improve the yield of enzyme will be undertaken.

18·3 *Cellulase* : (AB-67·2/69)

The main objective of this project is to make use of the large amount of cellulosic material that is available in the country for conversion into food, particularly in the form of protein. The possibility of using cellulases of microbial origin for digestion of cellulose is being studied in several cultures.

Twenty-two new fungal cultures belonging to *Basidiomycetes*, *Pestalotiopsis*, *Fusarium* and *Aspergillus* were isolated from different soil samples and decaying native cellulose sources and tested for cellulase production. A few of these, when grown on screening medium produced activities in the range of 40-50 'CX' units, 1-14 filter paper units and 4-19 cotton units per ml.

With a view to study enzyme production under submerged fermentation conditions, two cultures which had shown promising shake-flask activities were tested in chain fermentors. Preliminary attempts resulted in low cellulase production and the growth was poor. With several modifications it was possible to enhance the enzyme yields to the levels obtained in shake-flask experiments.

In shake-flask experiments with the supplementation of a few compounds to Reese's medium, maximum cellulase activity in the culture filtrate was reached in 5-6 days instead of 12-14 days. Under submerged conditions, replacement of cellulose powder with other cheaper cellulose sources and inorganic salts gave, within 7 days, a definitely higher enzyme activity than reported so far with this culture. The activity ranged from 200 'CX' units per ml. on the 7th day to 300 'CX' units per ml. on the 15th day.

Surface fermentation investigations were also undertaken to study the yields of cellulase with cheaper raw materials. Preliminary studies showed that with mixed cultures maximum cellulase activities were reached on the 5th day, thus reducing the period required for attaining the maximum activity. The activities were found comparable to those obtained in shake-flask experiments.

18·4 *Vitamin C* : (AB-67·3/68)

Commercial production of sorbitol and sorbose as part of the manufacture of ascorbic acid (Vitamin C) was reported by Hindustan Antibiotics Ltd., Pimpri, on the basis of the NCL know-how. Active assistance was rendered by NCL staff during the commissioning of the above industrial production.

18·5 *Alkali stable protease* : (AB-67·4/70)

A new microbial strain which was found to produce alkali stable protease in submerged culture was isolated. The stability of the enzyme in

the presence of detergents tripolyphosphate and perborate was found to be similar to that of several commercial enzymes. It showed good stability after 48 hours at pH 8.5 to 10.5 while at pH 4.3 to 5.9, its activity was much lower and the enzyme was also rapidly inactivated. The stability of the enzyme to heat under alkaline conditions and in the presence of detergents etc., was found to be satisfactory but the yield of the enzyme was rather low. Mutation experiments to increase the yield of the enzyme are in progress.

18.6 *Retardation of loss of ammonia applied as fertilizer in soil :*
(AB-67.5/70) (Formerly described as Oxidation of ammonia in soil)

A considerable loss of nitrogenous fertilizers occurs in soil due to the action of nitrifying and denitrifying micro-organisms. In spite of their importance very few of these nitrifying cultures were obtained and maintained in pure cultures since they generally fail to survive on subculture. Work on the isolation of organisms which convert ammonia to nitrite and nitrite to nitrate was undertaken in order to study their metabolism and methods of inhibiting their action.

Thirty-two cultures were isolated from soil samples obtained from several sources. Thirty of the cultures showed rapid oxidation of ammonia to nitrite and several showed formation of nitrate. No diminution in the activity of the cultures was observed on subculturing. Work on the isolation of pure cultures is in progress.

18.7 *Milk curdling bacteria which produces vitamin B₁₂ :*
(AB-67.6/71)

Indian vegetarian diet is generally low in vitamin B₁₂ content. A convenient method for supply of vitamin B₁₂ to vegetarians would be through milk curdling bacteria capable of synthesising vitamin B₁₂.

A screening programme has therefore been initiated to obtain cultures which will curdle milk and at the same time provide vitamin B₁₂.

About 40 lacto bacilli were screened so far and a few are being investigated further.

18.8 *Plant protease :* (B-12.9/72)

Purification of the acid protease from germinated matki (*Cymopsis*) was attempted by different methods like ammonium sulphate fractionation, DEAE-cellulose chromatography followed by chromatography on Sephadex G-200 and affinity chromatography using haemoglobin coupled Sepharose 4B. It was found that the enzyme could be purified to a great extent but the ultimate yield was only about 10-15% of the starting total activity. Some modifications are in progress to get better yields of the enzyme in the final purified stage.

19. PROCESS DEVELOPMENT AND CHEMICAL ENGINEERING STUDIES

19.1 *Dimethylaniline* : (SP-87/72)

Dimethylaniline is an important intermediate used in the dyestuff industry, and also in the manufacture of tetryl. M/s. Sahyadri Dyestuffs, Poona, have recently set up a 300 TPA plant based on the liquid-phase high pressure batch process developed by the NCL. In view of the increasing demand for dimethylaniline, (estimated to be 1500 TPA, valued at Rs. 15 lakhs) work on developing a continuous process has been undertaken on sponsored basis.

19.2 *p-Toluidine* : (SP-88/72)

p-Toluidine is an important organic dye intermediate. The present demand for this intermediate is 300 TPA valued at Rs. 45 lakhs which is being met by indigeneous batch-wise production.

The present sponsored project was undertaken to develop a continuous process by the vapour phase catalytic hydrogenation of *p*-nitrotoluene. The suitability of two catalysts was established on a bench scale reactor. The catalyst life is being assessed in a single tube reactor operated continuously. The conversion of *p*-nitrotoluene was found to be complete and hence the product does not require further purification. The results obtained in the single tube reactor are satisfactory.

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

Ethanolamines are important intermediates used in several industries. They are not manufactured in the country at present and are imported to the tune of Rs. 26 lakhs.

Process conditions for triethanolamine rich mixture and monoethanolamine rich mixture were established earlier. Both the mixtures contained 60% of the respective rich component, and 40% of the remaining two amines. The overall yield in both the cases was 85%.

Further batch experiments were carried out to confirm the reproducibility. In addition, process conditions for getting a product mixture of 25% mono,- 35% di,- and 40% triethanolamines were established. The process is being offered to industry.

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

Both these products are not produced in the country and demand is estimated to be around 600 TPA of diphenyl and 500 TPA of chlorinated diphenyl.

A process was developed on 1 kg/batch scale to obtain chlorinated diphenyl of desired specifications, with a chlorine content between 45 and 50% by weight. The product was a mixture of various chlorinated diphenyl compounds. As such, the composition of the final product is likely to vary depending upon the scale of operation, though the total chlorine content may remain the same. In view of this, pilot plant work is envisaged. However, the large scale work could not progress due to non-availability of diphenyl.

Efforts to prepare diphenyl from benzene in a molten lead reactor were unsuccessful. The project entails some fundamental studies on this critical process and will therefore to be undertaken with a basic orientation.

19.5 *Morpholine* : (ATT-160/70)

A process for morpholine starting from diethanolamine was developed last year. Raw materials are a major cost factor. In order to improve the economics of the process, certain modifications were worked out. By suitably altering the processing steps, it is now possible to recycle a few streams, resulting in reduction in alkali requirements and loss of morpholine.

Several runs were carried out with the modified process and yields of over 85% were consistently obtained.

19.6 *Sorbitol* : (ATT-55/66)

The original process for sorbitol from glucose was modified, employing higher pressures of the order of 200 atm. and a low catalyst concentration. A project report for a 1500 TPA plant (70 w/w solution) has been prepared. The modified process was successfully tried with inverted canesugar as starting material. The know-how is now released to industry.

19.7 *Alkylation of naphthalene* : (AB-92/71)

A project has been undertaken to develop a new route for the technical preparation of α and β naphthols through selective alkylation of naphthalene.

After standardising the procedure for the propylation of naphthalene using sulphuric acid as a catalyst, considerable laboratory scale experiments were conducted to study the effects of various variables on the reaction products.

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

Work on this project envisages development, testing and evaluation of different catalyst materials, based on synthetic zeolites (molecular sieve). This particular investigation relates to the use of molecular sieve catalysts in vapour phase alkylation reactions.

Based on the imported zeolites several substituted catalysts (REX-12 to REX-16) were prepared. Physico-chemical properties, such as adsorption,

surface area and the crystallinity of the fresh catalysts, as well as of spent catalysts, were determined in order to understand and correlate the catalytic properties. Zeolite type 13-X suitable for the catalyst preparation was synthesised and catalysts based on these are being prepared and examined for the vapour phase alkylation of benzene.

In continuation of the earlier work with ethanol and benzene as reactants, the performance of catalyst REX-12 was confirmed under the chosen optimum conditions. Under the same conditions REX-13 to REX-16 (belonging to the same series) were tested for their activity. Their performance was not satisfactory. Subsequently REX-12 (appearing better in the series) was tested for its life and it was seen that the activity was unsteady and rapidly fell with time. It was found that the reactivation of the catalyst did not help to regain its activity.

Subsequently, the performance of catalyst REX-12 was studied with ethylene substituted for ethanol. For the purpose, arrangements for accurate feeding of the reactants were made. The experiments of two hours duration were conducted at 180° having the benzene feed rate of 10 ml/hr. and the ratio of ethylene to benzene, 1:5. The performance of the catalyst was not satisfactory in respect of both per pass conversion and active life. The experiments were repeated but the poor performance was again observed.

The reasons for the overall poor catalytic characteristics of the sixteen REX and three REY molecular sieve catalysts, and the relatively better performance of REX-12 will be examined vis-a-vis the physical properties of the fresh and used (exhausted) catalysts when the required experimental data is available.

19·9 *Maleic anhydride* : (B-14·16/71)

Further work on the development of catalysts by using indigenously available low surface area material was continued. Supports such as porcelain, pumice, and ferrosilicon carbide were tried. A catalyst composition and operating parameters were fixed.

Work on the recovery of unreacted benzene by using activated carbon was continued. Recovery up to 75% of benzene could be obtained. Work on this project was temporarily discontinued from December 1972. As soon as the imported support is obtained further work will be taken up again.

19·10 *Amoxidation of hydrocarbons* : (B-14·15/71)

Work on the amoxidation of *o*-xylene to phthalonitrile was continued. Seven more catalysts were developed and assessed in a bench scale reactor. Conversion as high as 30% could be obtained. Further work on the development of catalysts is being continued.

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

19.11.1 *Gas-liquid reactions* : The reaction of ethylene and hydrogen chloride in nitrobenzene containing dissolved catalyst (to give ethyl chloride) was studied in detail and a kinetic equation has been formulated.

The absorption (accompanied by reaction) of phosphine in an aqueous solution of formaldehyde and hydrogen chloride was studied and a rate equation has been proposed.

Equations were developed for the analysis of several types of complex gas-liquid reactions.

19.11.2 *Gas-solid reactions*: A generalised equation for gas-solid reactions was developed which reduces to the various simple solutions available for particular situations.

The kinetics of the reaction between methyl chloride and ferrosilicon to give methylchlorosilanes is being studied.

19.11.3 *Catalyst fouling*: The model developed earlier for the effect of catalyst fouling in complex reactions was experimentally tested by using a consecutive reaction system (chlorination of tetrachloroethane to pentachloroethane and hexachloroethane). The model has been found to be satisfactory.

20. FOLLOW-UP ACTIONS

20.1 *Monochlorobenzene (MCB)*

A process for MCB was developed on sponsored basis for M/s. Hindustan Organic Chemicals Ltd., Rasayani. It is being engineered by a project engineering company. The detailed drawings prepared by the engineering firm were examined and necessary suggestions made. A complete process write-up for a 15 TPD plant was supplied to the firm. The erection of the plant is completed recently at HOC.

20.2 *Hexachloroethane (HCE)*

A continuous process, developed earlier, was released to M/s. Industrial Oxygen Ltd., Poona, for industrial implementation. After establishing the commercial feasibility and consumer acceptability of the product made in a pilot plant at the factory, a 300 TPA plant was designed. The plant is under erection and production is expected to commence soon.

20.3 *Chloromethanes*

The process for carbon tetrachloride and chloroform initially developed at the laboratory on a bench scale was further developed on a semi-commercial scale, jointly by the laboratory and M/s. Standard Alkali,

Chemicals Division, Thana. Intensive trials were carried out over a period of eight months. Several modifications were carried out on the reactor during this period to overcome the difficulties experienced. Finally the plant was operated successfully for a period of one month and then dismantled. Designs for a reactor of 4500 TPA chloromethanes plant have been supplied by the NCL to the firm. The plant is under erection and is scheduled to go on stream by the end of 1973.

20.4 Monoethylaniline (MEA)

A pilot plant of 15 kg of MEA per hour was installed and operated continuously. The product mixture was vacuum fractionated to obtain MEA conforming to specifications. This process is released and demonstrated to M/s. Atul Products, Bulsar.

20.5 Aniline

A process developed in the NCL for the continuous vapour phase hydrogenation of nitrobenzene to aniline was released to M/s. HOC, Rasayani. In order to assess the performance of the catalyst under the conditions desired at HOC, a single tube reactor assembly for continuous catalyst testing was designed and fabricated at the NCL. This was installed at the HOC factory site under the supervision of NCL scientists. A catalyst for this pilot plant was prepared and supplied for testing purposes. The plant was commissioned in January 1973 and the catalyst testing is in progress.

20.6 Ethylenediamine (EDA)

A continuous process for the production of ethylenediamine from ammonia and ethylene dichloride was developed. The process was demonstrated to the representatives of the project engineering firm, who prepared a detailed feasibility report on the technology for its client who has purchased this technology.

20.7 Phthalates

Work on diamyl phthalate (DAP) was undertaken at the instance of the Defence Department. A process for its manufacture was standardised. A 30 Kg sample of DAP was sent to the Defence establishment for evaluation.

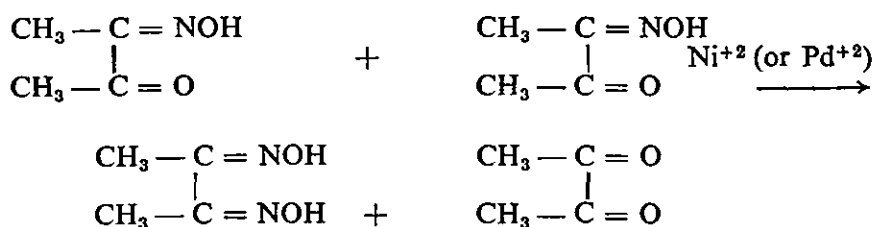
21. NEW ANALYTICAL METHODS

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

Basic research in this field is being pursued with the primary objective of developing new analytical methods for organic as well as inorganic compounds.

In non-polar solvents dimethylglyoxime, as well as diacetylmonoxime shows the absence of intramolecular hydrogen bonding as reflected in their IR spectra. Since the oxime groups (or the oxime and carbonyl groups in diacetylmonoxime) should revert to the *cis* position before chelation to a metal ion can occur, it is of interest to know if the polar solvent in which chelation takes place or the metal ion to be chelated is responsible for effecting the change from the *trans* to the *cis* position. The observation that the chelation takes place in an anhydrous non-polar solvent when the ligand and the metal ion are brought together suggests that metal ion turns over the co-ordinating groups from the *trans* to the *cis* position. Nickel and cobalt were chosen to prove this point.

The chelation reaction of diacetylmonoxime with transition metal ions such as Fe(II), Co(II), Cu(II), Ni(II), Pd(II) and V(V) was rather weak as indicated by the low formation constants of these chelates. In keeping with their low stabilities, they are also feebly coloured and hence are not likely to be analytically significant. Even so, Ni(II) and Pd(II) were found to react with diacetylmonoxime in a peculiar manner. By mixing the ligand and the metal ions in aqueous solution, a feeble colour of the monoxime chelate was developed. But soon the solution became turbid precipitating out gradually the insoluble nickel/palladium chelate of dimethylglyoxime. In other words, diacetylmonoxime was converted to dimethylglyoxime by these two metal ions. Though the mechanism of this reaction was not clear it appeared that diacetylmonoxime undergoes disproportionation under the influence of the metal ion as follows :—



The presence of diacetyl was confirmed in the end products. It must be emphasised that metal ions other than Ni(II) and Pd(II) were not effective in bringing about this “disproportionation”. Ni(IV) dimethylglyoxime chelate was also formed from Ni(II) diacetylmonoxime, under suitable oxidative conditions.

21.2 Analysis of amine mixture

The polarographic method developed earlier for the mixtures of ethylene diamine, diethylene triamine and triethylene tetramine based on the differential redox behaviour of their copper chelates was not entirely reliable when applied to on-stream reaction products. A more reliable method has now been developed.

21.3 *Estimation of ethanolamine in morpholine*

For the determination of diethanolamine in morpholine, the characteristic reactions of the secondary amine functions could not be exploited because both carried the secondary amine groups. Attention was therefore focussed on the reactivity of the alcoholic function. It was established that under controlled conditions dichromate quantitatively and preferentially oxidised diethanolamine. This observation was taken advantage of in developing a titrimetric procedure for estimating diethanolamine in morpholine.

21.4 *Microanalytical methods*

21.4.1. *Simultaneous microdetermination of sulphur and phosphorus in organic compounds* : The interference caused by the presence of phosphorus in the estimation of sulphur, adopting the titrimetric finish, was studied in some phenothiophosphine trioxides. As a result of this study, a method has been evolved for the simultaneous determination of sulphur and phosphorus in compounds of the above types containing a known ratio of these atoms.

21.4.2 *Simultaneous microdetermination of chlorine and bromine in organic compounds* : After combustion of the compound by the Schoniger method, the alkali halide was titrated argentometrically. The silver halide formed was weighed and the ratio of halogen : silver determined, which helped in determining the percentages of chlorine and bromine in the compound.

22. INFRASTRUCTURE ACTIVITIES

22.1 *Analytical groups*

22.1.1 Physico-chemical analyses

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.

Analytical methods were developed and standardised for the following during the period under review:

Ethylenediamine : The polarographic method previously reported for differentiating and determining ethylenediamine, diethylene triamine and triethylenetetramine was replaced by an improved titrimetric method based on the differential behaviour of their copper chelates.

Morpholine : Unreacted diethanolamine in morpholine was determined by preferentially oxidizing diethanolamine with a known excess of dichromate and back titrating this excess oxidant.

Acrylic monomers : Methods were standardised for the determination of acrylonitrile, acrylamide and methyl acrylate.

22.1.2 *Microanalysis*

The major activity of this group includes micro-analysis of organic and organo metallic compounds for various elements, 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 research organizations, universities, etc., on payment depending upon the internal workload.

This group is also concerned with the development of new analytical methods for the applied projects in the NCL, analysis of organic compositions of industrial importance and research in microanalytical chemistry and analytical organic chemistry.

During the period under review, 2380 samples were analysed for various elements (2316) and functional groups (64).

22.1.3 *Mass spectrometry*

During the period under review 1231 samples were analysed.

22.1.4 *Spectrochemical and other analysis*

With the help of various physico-organic techniques such as UV, IR, NMR, Visible Spectra, VPC and GLC, analytical and structure elucidation work was carried out in the laboratory in support of the laboratory's research programmes.

Number of samples studied

NMR	3909
UV	7
IR	4049
VPC/GLC	3156
X-ray powder patterns	100
Determination of surface area by BET technique	20
Thermogravimetric analysis (DTA, TGA, TG, etc.)	88

22.1.5 *Ultracentrifugal analysis*

During the year 20 samples were analysed.

22.2 *Instrumentation section*

The section is primarily engaged in the maintenance and upkeep of special type of analytical instruments, like NMR spectrometer, spectrophotometers for IR, UV, visible gas chromatographs, X-ray machines, etc., besides normal servicing bridges, potentiometers, microbalances, furnaces colorimeters, etc.

Total number of jobs and test reports completed were 397.

22.3 *Engineering Section*

22.3.1 *Mechanical Engineering*

Apart from maintenance of the laboratory's installations, utility services and equipment, several pilot plants were designed in collaboration with the scientists concerned, and fabricated.

The total job orders executed were 2931.

The following pilot plant fabrications were carried out for the Chemical Engineering and Process Development Division during the year :

- i. Horizontal reactor for Silicone project
- ii. High speed rotating disc reactor for catalytic reactions.

The following special fabrications of equipment and components were carried out during the year :

- i. Brass galvanometer
- ii. Gas burner for Vapour Phase burning of SiCl_4
- iii. Stirring assembly made of brass for liquid diffusion cells
- iv. Ultrasonic interferometer cell
- v. 'Desaga' type T.L.C. applicator with base plate, for Central Fuel Research Institute, Dhanbad.

22.3.2 *Civil Engineering*

During the period under review the following constructions were carried out :

- i. Construction of a shed for the pump-house at NCL
- ii. Construction of a shed for Organic Synthesis group
- iii. Construction of Glass House for plant tissue culture experiments

22.4 Glass Blowing Section

This section primarily undertakes repairs of glass apparatus for various research groups in the Laboratory. During the period under report, 4002 jobs were completed.

Glass assemblies of various types were fabricated as per specific drawings furnished by scientists. About 2017 fabrications of glass assemblies were carried out. Of these 636 fabrication jobs were related to the sponsored projects. About 5292 standard ground glass joints were fabricated for internal use.

22.5 Library

The library houses about 60,341 accessioned items consisting of books, periodicals, publications, patents, standards, technical reports, etc., in its stock. 4,341 volumes comprising (i) bound volumes of journals and (ii) books, newly added (1,269) during the period under review. 817 periodicals, including annual reports from various institutions, were received.

Library facilities are extended to readers from industry, Govt. departments, universities, colleges and other research institutes.

22.6 Division of Technical Services

Commercial intelligence

As a follow-up of Advisory Panel recommendations, market data was collected for nitrofurazone and calcium mandelate.

Compilation and documentation of techno-economic, market and scientific data relevant to Indian chemical industry in general and NCL research programme in particular was carried out.

Industrial liaison and co-ordination

Over 3800 enquiries pertaining to NCL projects and general technical enquiries from different individuals, industries, Govt. organisations and starred questions from the Parliament were attended to. 154 applications in chemical and allied industries for industrial licences with foreign collaboration were scrutinized.

A large number of entrepreneurs interested in NCL projects were attended to and relevant information supplied. 2038 visitors from educational institutions, industries and other organisations were shown round the laboratory.

Non-technical notes on processes (approved by PRC) along with tentative cost estimates were prepared. The total number of non-technical notes which are at present available for circulation to interested parties is 88.

A note on "technological support to industry by NCL" was prepared for an inquiry from CSIR. This is being given to entrepreneurs interested to know how NCL can support their activities.

Contractual research proposals were prepared for the following projects—Organo phosphorus pesticides, chlorohexidine, vanillin from lignin sulphonate, resin-flux for resin-cored solder wire, chemical examination of the effluent coming out of pulping units, atomic absorption spectrophotometer, catalyst for exhaust gas purifier, liquid bleach, adhesive for filters, SO₂ from low grade pyrites, nitromusk compounds, 1-menthol from citronellol, vapour phase reduction of p-nitrotoluene to p-toludine, resin for friction materials, phosphorus pesticides, and dimethyl aniline by continuous process.

Liaison with CSIR

Two NCL processes were referred to CSIR/NRDC for licensing to 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. Information for inclusion in the CSIR Annual Report Jan-Dec. 1972, was sent.

Research schemes submitted to CSIR for financial assistance were scrutinized taking into consideration their feasibility, relevance to CSIR's and the country's research and industrial programmes. Comments on 123 such schemes were sent to CSIR.

The division continued to render photographic and draftsman services to other divisions of the laboratory.

Publicity

A radio talk "Vidnyanachi Tirthakshetre" was prepared. This was broadcast by Poona Station of All India Radio.

23. RESEARCH ANALYSIS AND RESEARCH MANAGEMENT

Research analysis

Preliminary techno-economic feasibility reports on Bisphenol-A and Synthetic gem stones were submitted for the consideration of the Process Release Committee.

Cost estimates were prepared for methyl chlorosilanes.

Research planning and management

Work connected with the formulation of Science & Technology Plan

for Chemical Industry was continued. The following notes were prepared: (i) Guidelines for foreign collaboration, policies and procedures, (ii) Utilization of inhouse R & D results by large scale industries, and (iii) Interlink between NCST/DST/CSIR and other R & D Agencies.

Transfer of technology

Transfer of technology studies were continued to identify and analyse the problems (internal & external) connected with successful transfer of know-how to industry. The analysis of information received from entrepreneurs who have acquired know-how from CSIR laboratories in response to the NCL questionnaire has been carried out. Efforts will be made to suggest a system for accelerating the transfer of research results to industry. Part of this study under the title "Factors affecting utilization of indigenous research results-some observations on the Indian Chemical Industry", is under publication.

Patents

Work was undertaken to analyse the role of patents in improving domestic R & D capability. Patent laws of different countries were studied and a statistical survey of patents filed in India during the past 20 years was undertaken with a view to propose a model for a patent system for developing countries. This study will soon be published.

Planning for R & D projects

Various decision making steps in planning for R & D projects were stated.

Proper follow up of the steps suggested may avoid delays and wastages of resources and help successful transfer of R & D.

The procedure provides for interdisciplinary approach, cross fertilization of ideas and self screening of the proposal which may generate team spirit and motivation amongst R & D workers.

Reports

(i) Perspective plan for NCL

As directed by the NCL Executive Council, a perspective plan for the NCL was prepared, which indicates the main thrust of NCL activities in the coming 10-15 years. The major effort of the NCL will relate to R & D work on-(i) projects identified by NCST, (ii) projects sponsored by industry, (iii) projects involving improvement, adaptation and development of existing and imported technology, (iv) development of new technology relevant to the country's social, economic and industrial needs and (v) basic scientific research necessary from the point of view of future technological needs.

The R & D work will be carried out in the following areas :

Natural and synthetic organic chemicals of importance to industry and agriculture, solid state materials, industrial inorganic chemicals, organo-metallic compounds, industrial microbiology, plant and animal tissue culture, industrial polymers and elastomers, process development related to petrochemicals and bulk organic chemicals, scale-up of processes and studies in chemical engineering, physico-chemical studies and instrumentation.

Within the above broad framework several research projects with a large socio-economic impact have been proposed for investigation.

(ii) Vth Five Year Plan for NCL

The guidelines laid down for the "Perspective plan for NCL" were followed while preparing the Vth Five Year Plan. During this period work on 20 research areas will be undertaken, out of which several projects have already been initiated.

(iii) Status report was prepared on the "pulping of mixed hard woods available in the forest of Chanda district of Maharashtra".

In addition to the above the following reports/programmes were compiled:

- (i) Report on NCL for inclusion in CSIR Annual Report 1972
- (ii) NCL Annual Report 1971-72 & Achievements 1971-72
- (iii) NCL Research Programme 1973-74

APPENDICES

1. SPECIAL EQUIPMENTS/FACILITIES AVAILABLE*

<i>Name of the equipment/instrument</i>	<i>Function</i>
1. Amino acid analyser (Beckman model 120 C)	Automatic analysis of amino acids and related compounds
2. Double focussing mass spectrometer CEC 21-110 B	Qualitative and quantitative analysis of compounds
3. Mixing mill	PVC mixing
4. Vapour phase chromatographs (VPC) Gas liquid chromatographs (GLC)	Qualitative and quantitative analysis of organic compounds

* These are in addition to those reported last year.

2. SERVICES TO INDUSTRY, RESEARCH INSTITUTES, UNIVERSITIES, ETC.

2.1 *Supply of cultures*

During the year under report, 731 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 carried out on payment for universities, research institutes, Govt. departments, private parties, etc.

Microanalysis	117
NMR	13
IR	120
Mass spectra	143
Thermogravimetric analyses (viz. DTA, TGA, TG, etc.)	22

Technical aid was rendered to about 25 agencies including universities, Govt. departments and industry in the form of experimental work, instrumental repairs, fabrication of special glass apparatus and analyses of special nature.

Training in the areas of glass blowing techniques, plant tissue culture, maintenance of instruments and chemical and spectroscopic analytical methods was given to 19 representatives of I.I.Ts., colleges and universities.

3. SPONSORED PROJECTS

3.1 *Sponsored projects concluded during 1972-73*

- | | |
|---|---|
| 1. Conversion of bauxite into anhydrous aluminium chloride | M/s. Tata Chemicals Ltd., Bombay |
| 2. Preparation of chlorohydroxyquinoline | M/s. Biological Evans Ltd., Hyderabad. |
| 3. Extraction of morphine and other alkaloids from lanced poppy-straw | Ministry of Finance, Govt. of India, New Delhi. |
| 4. Production of rutile titania from Indian ilmenite | M/s. Asian Paints, Bombay. |
| 5. Solvent extraction of sandalwood oil | Govt. Sandalwood Oil Factory, Bangalore. |
| 6. Thermosetting resins for industrial laminates | M/s. Formica India Ltd., Poona-19. |
| 7. Production of xylit | M/s. Unichem Laboratories Ltd., Bombay. |

Besides, work on two schemes viz. Perfumery products based on Δ^3 -Carene and perfumery products based on longifolene and isolongifolene which formed part of the earlier schemes was concluded.

3.2 *Sponsored projects undertaken during 1972-73*

- | | |
|--|--|
| 1. 1,3-Butylene glycol | Indian Petrochemicals Corporation Ltd., Baroda. |
| 2. Manufacture of dimethylaniline by continuous process | M/s. Sahyadri Dyestuffs and Chemicals (P) Ltd., Poona. |
| 3. Preparation of 1-menthol from citronella oil of Indian origin | M/s. Bhavana Chemicals Ltd., Bombay. |

- | | |
|---|---|
| 4. Preparation of nitromusk compounds | M/s. Opal Fine Chemicals, Bombay. |
| 5. Catalytic vapour phase epoxidation of olefins | Indian Petrochemicals Corporation Ltd., Baroda. |
| 6. Synthesis of resins for friction materials | M/s. Hindustan Ferodo Ltd., Bombay. |
| 7. Vapour phase reduction of <i>p</i> -nitrotoluene to <i>p</i> -toludine | M/s. Sudarshan Chemical Industries (P.) Ltd., Poona. |
| 8. Fractionation of turpentine oil | M/s. J & K Industries Ltd., Srinagar. |
| 3.3 <i>Sponsored projects continued from 1971-72</i> | |
| 1. Acrylic acid/acrylates from acrylonitrile | Indian Petrochemical Corporation Ltd., Baroda. |
| 2. Cationic dyes for acrylic fibres | M/s Sahyadri Dyestuffs and 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-amino-benzoate | M/s. Indian Schering Ltd., Bombay. |
| 5. Flavonoids, tannins, stilbenes lignans and quinones in some Indian forest trees (PL-480) | U.S. Dept. of Agriculture, Washington. |
| 6. Propylene oxide | Indian Petrochemical Corporation Ltd., Baroda. |
| 7. Synthesis of potential pharmacologically active substances | M/s. Sarabhai Research Centre, Baroda. |

4. TECHNOLOGY TRANSFER

4.1 *Demonstrations*

Process know-how for the following products was demonstrated :

<i>Process</i>	<i>Name of the party</i>
1. Carbazole Dioxazine Violet pigment	M/s. Square Chemical Works, Bombay-9.
2. Clofibrate	M/s. Biological Evans Ltd., Hyderabad.
3. Phenylacetic acid	M/s. Lakshmi Chemical Industries (P.) Ltd., Hyderabad-1.
4. D.C. Recording polarograph	i) M/s. Laxsons Engineering & Electronics (P.) Ltd., Bombay-29. ii) M/s. Chromatography & Instruments Co., Baroda.
5. Direct reading spectrophotometer/ colorimeter	i) M/s. The Scientific Instrument Manufacturing Co. Ltd., Allahabad. ii) M/s. Neotronics Corporation, Bombay-80.
6. Terpeneol and terpin hydrate	M/s. Dujodwala Industries, Faridabad.

4.2 *Processes leased out during 1972-73**

<i>Process</i>	<i>Party</i>
1. Aniline	M/s. Hindustan Organic Chemicals Ltd., Rasayani.
2. Can lining composition	M/s. Arya Chemical Works, Calcutta.-13.
3. Carbazole Dioxazine Violet pigment	M/s. Square Chemicals, Bombay-9

<i>Process</i>	<i>Party</i>
4. Clofibrate	M/s. Biological Evans Ltd., Hyderabad.
5. D.C. Recording polarograph	i) M/s. Elico (P.) Ltd., Hyderabad-18 ii) M/s. Laxsons Engineering & Electronics (P.) Ltd., Bombay-29. iii) M/s. Chromatography and Instruments Co., Baroda.
6. Direct reading spectrophotometer colorimeter.	i) M/s. The Scientific Instrumen, Manufacturing Co., Ltd., Allahabad. ii) M/s. Neotronics Corporation Bombay-80.
7. Ethylene from alcohol	M/s. Industrial Oxygen Co., (P.) Ltd., Poona-1.
8. Flexible magnets	M/s. Ajanta Enterprises, Bombay-13
9. Monoethylaniline	M/s. The Atul Products Ltd., Atul, Dist., Bulsar.
10. Nitrile rubber (low, medium and high grade)	M/s. Synthetics & Chemicals Ltd. Bombay-20.
11. Radiosonde thermistors	M/s. Bhagyanagar Laboratories, Hyderabad.
12. 70% Sorbitol	M/s. Maize Products, Ahmedabad.
13. Potentiometric strip chart recorder	i) M/s. Bagga Electronics, Bombay-4. ii) M/s. Indo-Burma Petroleum Co. Ltd., (Electronics Mfg. Divi- sion) Calcutta-1.
14. Nicotine sulphate	Shri. P. Jaipuria, Calcutta-14.

<i>Procees</i>	<i>Party</i>
15. Sachets-Hot and Cold	M/s. Biswanath Fatesaria, Calcutta-7.
16. Phenyl acetic acid	M/s. Lakshmi Chemical Industries (P.) Ltd., Hyderabad-1.
17. Foundary core binder	M/s. Card-Chem Industries, Hyderabad.

* All have been released through NRDC on non-exclusive basis

4.3 *Processes assigned to NRDC during 1972-73*

1. Bisphenol-A
2. Cashew nut shell gum (CNS gum)
3. Cellulose powder
4. Clofibrate
5. N, N-Dimethylbiguanide HCl and phenethylbiguanide HCl
6. Flexible magnets
7. Foundry core binder (Sinol core binder)
8. Gemstones (synthetic)
9. Gum arabic substitute
10. Microfilters
11. Monoethylaniline
12. Morpholine
13. Radiosonde thermistors
14. Tamarind kernel powder (TKP) phosphate and TKP borate
15. 2, 2, 4-Trimethyl-6-ethoxy-1,2-dihydroquinoline

4.4 *Premia and royalty received by NRDC through NCL processes during the year 1972-73*

Premia

<i>S.No.</i>	<i>Name of the process</i>	<i>Name of the firm</i>	<i>Premia received (Rs.)</i>
1.	Aniline	M/s. Hindustan Organic Chemicals Ltd.	10,000.00
2.	Butyl titanate	M/s. Synthochem	3,000.00
3.	Cadmium sulphide photo cells	M/s. Chinoy Electronics	1,500.00
4.	Carbazole Dioxazine Violet pigment	M/s. Kondivita (P) Ltd.	25,000.00

<i>S.No.</i>	<i>Name of the process</i>	<i>Name of the firm</i>	<i>Premic received (Rs.)</i>
5.	Carbazole Dioxazine pigment	M/s. Square Chemicals	25,000 ·00
6.	Carbazole Dioxazine pigment	M/s. Vapson products	25,000 ·00
7.	Clofibrate	M/s. Biological Evans Ltd.	10,000 ·00
8.	Dimethylaniline	M/s. Sahyadri Dyestuffs & Chemicals (P) Ltd.	10,000 ·00
9.	Dioctyl phthalate	M/s. R. L. Dalal & Company	37,499 ·00
10.	Foundry core binder	M/s. Card Chem Industries	2,500 ·00
11.	<i>p</i> -Menthane hydroperoxide	M/s. Camphor & Allied Products	5,000 ·00
12.	Microfilters	M/s. Sona Micro Filters	3,000 ·00
13.	Monochloroacetic acid	M/s. Hico Products (P) Ltd.	30,000 ·00
14.	Monoethylaniline	M/s. Atul Products Ltd.	25,000 ·00
15.	Nicotine sulphate	M/s. Pradeep Jaipuria	2,000 ·00
16.	Nitrile rubber	M/s. Synthetic & Chemicals Ltd.	30,000 ·00
17.	Phenyl acetic acid	M/s. Laxmi-Chemical Industries	10,000 ·00
18.	D. C. Recording polarograph	M/s. Chromatography & Instr. Co.	15,000 ·00
19.	D. C. Recording polarograph	M/s. Elico (P) Ltd.	15,000 ·00
20.	D.C. Recording polarograph	M/s. Laxsons Engg. & Electronics (P) Ltd.	15,000 ·00
21.	Potentiometric strip chart recorder	M/s. Bagga Electronics	10,000 ·00
22.	Potentiometric strip chart recorder	M/s. Indo-Burma Petroleum Co. Ltd.	10,000 ·00

<i>S.No.</i>	<i>Name of the process</i>	<i>Name of the firm</i>	<i>Premia received (Rs.)</i>
23.	Rubber reclaiming agent	M/s. Swastik Rubber Products	7,000.00
24.	Rubber reclaiming agent	M/s. Swastik Rubber Products	7,000.00
25.	Hot & Cold sachets	M/s. Bishwanath Fatesaria	2,000.00
26.	70% sorbitol	M/s. Maize Products	18,750.00
27.	Direct reading spectrophotometer/Colorimeter	M/s. Neotronics Corporation	10,000.00
28.	Direct reading spectrophotometer/colorimer	M/s. Scientific Instruments Co.	10,000.00
29.	Terpineol	M/s. Dujodwala Industries	5,000.00
Total Premia			3,79,249.00

Royalties

<i>S.No.</i>	<i>Name of the process</i>	<i>Name of the firm</i>	<i>Royalty received (Rs)</i>
1	Bacterial diastase for desizing	M/s. Chemaux (P) Ltd.	5,180.98
2	Tert-butyl catechol	M/s. Percynic Chemicals	1,433.50
3	Cadmium sulphide photo cells	M/s. Chinoy Electronics	164.70
4	Can sealing composition	M/s. Arya Chemicals Works	6,320.68
5	Catechol	M/s. Percynic Chemicals	1,928.10
6	Cation exchange resin from CNSL base	M/s Tulsi Industries	1,483.70

<i>S.No.</i>	<i>Name of the process</i>	<i>Name of the firm</i>	<i>Royalty received (Rs.)</i>
7	Vapour phase chromatograph	M/s. Associated Instruments Mfg. Co. (I) Pvt. Ltd.	25,650.00
8	Hard ferrites	M/s. Semi Conductors Ltd.	2,444.98
9	Hexylresorcinol	M/s. Unichem Lab. Ltd.	10.63
10	β - Ionone	M/s. Industrial Perfumes Ltd.	41,039.64
11	Nicotine sulphate	M/s. Urvkunj Nicotine Industries	9,039.34
12	Polyurethane printing rollers	M/s. Sree Saraswati Printing Press Ltd.	2,247.04
13	Sachets-Hot and cold	M/s. Thermo Chem. Lab.	287.45
14	Sachets-Hot and cold	M/s. Vasant Industrial Corporation	39.55
Total Royalty			97,290.29
Total Premia and Royalty			4,76,539.29

5 SEMINARS AND LECTURES

5.1 Lectures

The following lectures were delivered by visiting scientists:—

1. Dr. C. L. Soffpaur
Southern Utilization Research and Development Division,
New Orleans. Overview of Textile Research at SRRL
2. Dr. M. J. Thirumalachar,
Hindustan Antibiotics Ltd.,
Pimpri, Poona-18. Antifungal antibiotics in the treatment of fungal diseases of humans
3. Prof. Santi R. Palit, Indian Association for the Cultivation of Science, Jadavpur, Calcutta-32. Non-Faradaic electrolysis

- | | | |
|-----|---|---|
| 4. | Lord. J. M. Tedder, Purdi Prof. of Chemistry, The University, St. Andrews, U. K. | <p>Heterocyclic diazo-compounds (possible dye-line intermediates.)</p> <p>Some cyclic addition reactions of orthoquinones</p> <p>Directive effects in free radical substitution reaction.</p> <p>A new look at the Electronic Theory of Organic Chemistry.</p> <p>An Organic Chemist's view on the role of phosphate in biological reactions.</p> |
| 5. | Dr. V. R. Srinivasan, Prof. of Microbiology, Louisiana State University, Baton Rouge, USA | <p>Fermentative and enzymatic studies on cellulose.</p> |
| 6. | Prof. M.V.C. Sastri Dept. of Chemistry IIT, Madras. | <p>Some aspects of research in heterogeneous catalysis</p> |
| 7. | Dr. Kashinath S. Sardesai, I. P. C. L., Baroda | <p>Monomers, polymers and recent advances in polyester technology.</p> |
| 8. | Dr. R. V. Smythe, Project Leader, Biology of Wood-Products, Insect Lab., USA. | <p>Termite and powder-post beetle-research at Wood Products Insect Laboratory, Mississippi.</p> |
| 9. | Prof. T. Urbanski, Warsaw, Poland. | <p>Penicillin (4 lectuses)
Charge transfer phenomenon (Part I & II).</p> <p>Free radicals through mechanical action.</p> <p>Non-aromatic heterocyclic systems.</p> <p>Organic anions-alkylation in aqueous medium</p> |
| 10. | Prof. M. P. Cava, University of Pennsylvania, U.S.A. | <p>Chemistry of non-classical sulphur heterocyclics.</p> |

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| 11. | Dr. David Patterson, Dept. of Colour Chemistry, University of Leeds, U.K. | Continuous coupling methods for making azo pigments. |
| 12. | Dr. Arthur Finch, Dept. of Chemistry, Royal Holloway College, University of London, U.K. | Spectroscopic properties and structures of some phosphorus compounds.

Thermodynamic properties of some Boron compounds. |
| 13. | Dr. S. P. Polli, Central Drug Research Institute, Lucknow. | Biogenesis theory and its role in the discovery of new natural products. |
| 14. | Dr. V. P. Kane, Department of Chemistry, Princeton University, U.S.A. | Novel synthetic reactions. |
| 15. | Prof. R. Kumar, Indian Institute of Science, Bangalore. | Several lectures on fluidization techniques. |

5.2 Following NCL scientists delivered lectures at various institutes, universities, colleges, etc.

<i>Name of the scientist</i>	<i>Subject</i>	<i>Venue</i>
1. Dr. M.N.S. Murthy	Quantum chemistry (27 lectures)	N. Wadia College, Poona.
2. Dr. L. M. Pant	Crystallographic work at NCL	Banaras Hindu University, Varanasi.
3. Dr. K. G. Das	Organic mass spectrometry (3 lectures)	Osmania University, Hyderabad.
4. Dr.P.R. SubbaRaman	The chemistry of complex compounds in relation to complexometric titrations.	Cement Research Institute, of India New Delhi.
	The methodology of complexometric titrations as applied to the constituents in cements and related materials.	Cement Research Institute, New Delhi

<i>Name of the scientist</i>	<i>Subject</i>	<i>Venue</i>
5. Dr. P. N. Rangachari	Industrial Fermentations.	Abbasaheb Garware College, Poona.
6. Dr. C. SivaRaman	Chemistry of immunoglobulins	Virus Research Centre, Poona.
	Biophysical and biochemical methods used in virology (2 lectures)	—do—
	Ultracentrifugation and electrophoresis in Biochemistry	University of Poona, Poona.
	Allosteric proteins	Ahmednagar College, Ahmednagar.
	Immunoglobulins	—do—
7. Dr. V. Jagannathan	Work in the Biochemistry Divn. of NCL	Indian Institute of Science, Bangalore.
8. Dr. M. C. Srinivasan	Seed transmission of bacterial pathogens	Indian Agricultural Research Institute, New Delhi.
9. Dr. C. R. Narayanan	Mass spectrometry and structure determination of terpenoids	Yeshwant Mahavidyalaya, Nanded.
	Mass spectrometry of some steroidal alkyl ethers	—do—
	Conformation of the carbonyl group by NMR & IR spectroscopy	—do—
10. Dr. V. N. Gogte	Tautomerisam in arylamino methylene-cycloalkanones	Shivaji University, Kolhapur.

<i>Name of the scientist</i>	<i>Subject</i>	<i>Venue</i>
Dr. V. N. Gogte	Synthesis of nitrogen heterocyclics by rearrangement reaction	Shivaji University Kolhapur

6. STAFF STRENGTH AS ON 31-3-1973.

(1) Scientific

(i) Director	1
(ii) Scientist F	6
(iii) Scientist E	17
(iv) Scientist C	53
(v) Scientist B	35
(vi) Scientist A	49
(vii) S. S. A.	81
(viii) J. S. A.	38
(ix) S.L.A.	52
Total	332

(2) Technical	173
(3) Non-technical	100
(4) Class IV technical	131
(5) Class IV non-technical	77
Total	813

(6) Research fellows, Pool Officers and Guest Workers

(a) JRF & SRF	53
(b) CSIR Pool Officers	14
(c) Guest Workers	25
Total	92

(7) Scientific staff working on sponsored projects

(a) NCL staff	5
(b) Staff deputed by sponsors	13
Total	18

7. STAFF NEWS

7.1 *Deputations/training etc.*

1. Dr. Sukh Dev at the invitation of the Royal Australian Chemical Institute, Adelaide, visited Australia and delivered lectures as a guest speaker at the Division of Organic Chemistry of the Institute of Adelaide, South Australia (May 1972).
2. Mr. A. M. Lele attended a course on 'Management by Systems Process' at the Administrative Staff College of India, Hyderabad (June 1972).
3. Mr. A. A. Gunari returned from Czechoslovakia, after completing a post-graduate course on 'Macromolecular Chemistry' (September 1972 to April 1973).
4. Dr. B. D. Tilak visited USSR as a member of the Indian Delegation in connection with the proposed agreement for collaboration in the field of Science and Technology ((October 1972).
5. Mr. P. P. Lohokare, attended a course on 'Value Engineering' at the National Institute for Training in Industrial Engineering (NITIE), Bombay (October 1972).
6. Dr. B. D. Tilak visited U. K. as a leader of the Indian Team for Sub-Group on Research and Development in pursuance of the deliberations of the third meeting of the Indo-British Technological Group held in New Delhi in March, 1972 (November 1972).
7. Mr. Karam Chand attended a course on 'Personnel Administration' at NITIE, Bombay (November 1972).
8. Mr. D. Raghunath attended a seminar on 'Processing of High Polymer Products' at the Indian Institute of Technology, Madras (January 1973).
9. Dr. S. L. Kapur, Dr. C. R. Narayanan and Dr. S. Gundiah participated in the deliberations of the 60th (Diamond jubilee) session of the Indian Science Congress at Punjab University, Chandigarh (January 1973).
10. Mr. N. D. Seth, Dr. N. R. Ayyangar and Dr. S.H. Abhyankar attended a seminar on 'Ancillary Industries' at Hindustan Organic Chemicals, Rasayani (February 1973).
11. Dr. V. J. Rao attended a symposium on 'Solid State Materials at National Physical Laboratory, New Delhi (February 1973).

12. Mr. J. V. Rajan participated in the All-India Conference—NITIE Alumni Association, at NITIE, Bombay (February 1973).
13. Dr. A. P. B. Sinha left for U.S.A. to undertake collaborative research work in the field of semiconductors with Prof. J. M. Honig at the Purdue University under India-US Exchange of Scientists Programme (March 1973).

7.2 Participation of NCL scientists in Seminars and Symposia

- | | |
|---|---|
| 1. Seventh Annual Conference of Electron Microscope Society of India, New Delhi. | Dr. A. Goswami |
| 2. Crystallography symposium, Banaras Hindu University, Varanasi. | Dr. L. M. Pant,
Dr. S. S. Tavale |
| 3. Seminar on Environmental pollution, Bombay. | Dr. K. G. Das |
| 4. Seminar on Pollution, IMA Poona. | Dr. K. G. Das |
| 5. Seminar on Bacterial leaching of ores, Jamshedpur. | Dr. P. N. Rangachari |
| 6. All India Starch Round Table Conference, Mahabaleshwar | Dr. J. L. Bose,
Dr. T. R. Ingle,
Dr. J. P. Varma |
| 7. Guha Research Conference Dharmasala (MP) | Dr. V. Jagannathan |
| 8. Symposium on 'Theory and Practice on Application of Reactive Dyes' Bombay | Dr. B. D. Tilak,
Dr. N. R. Ayyangar,
Mr. N. V. Badami,
Mr. A. G. Lugade |
| 9. Seminar on Development of Small Scale and Ancillary Industries at HOC, Rasayani. | Dr. B. D. Tilak,
Dr. R. B. Mitra,
Dr. N. R. Ayyangar,
Dr. S. M. Abhyankar,
Mr. N. D. Seth |
| 10. UGC Symposium on Mechanistic Organic Chemistry at Madras. | Dr. V. N. Gogte |
| 11. Diamond Jubilee Session of the Indian Science Congress, Chandigarh. | Dr. S. L. Kapur,
Dr. S. Gundiah |

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|---|---|
| 12. Seminar on processing of high polymer products at the Indian Institute of Technology, Madras. | Mr. D. Raghunath |
| 13. IRMRA VIth Tech. Seminar, Madras. | Dr. N. D. Ghatge |
| 14. Summer School on 'Scale-up concepts in chemical engineering' organized by IIT, Delhi. | Dr. G. R. Venkitakrishna,
Mr. S. P. Mukerjee |

7.3 *Awards and Honours*

- (1) Dr. P. Roy Chowdhury was awarded a gold medal for the paper entitled 'Adiabatic compressibility of polyelectrolytes' published in the *Vijana Anusandhan Patrika*.
- (2) A certificate of merit was awarded by the Invention Promotion Board, New Delhi, in August 1972, for the process of 'Recovery of Pyridine bases from their aqueous solution'. The process is covered by Indian Patent No. 111311 in the names of M. U. Pai, G. V. Potnis, N. K. Buzruk, and R. N. Paul.

7.4 Post-graduate degrees received by NCL staff members and research fellows/guest workers

<i>S.No.</i>	<i>Name</i>	<i>Degree</i>	<i>University</i>	<i>Subject of thesis</i>	<i>Name of the guide</i>
*1.	Bhide, R. D.	Ph.D.	Poona	Some studies on photoconducting and other properties of thin films	Dr. A. Goswami
2.	Brahme, P. H.	Ph.D.	Bombay	Chemical engineering studies in a slurry reactor, hydrogenation of glucose on raney nickel	Dr. L. K. Doraiswamy
3.	Choudhary, V. R.	Ph.D.	Poona	Kinetics and catalysis-A kinetic study of catalytic isomerisation of n-butene to isobutene	Dr. L. K. Doraiswamy
*4.	Gogte (Mrs), V. V.	Ph.D.	Poona	Studies in terpeneoids (Chemical examination of the constituents of resin from <i>Alantbus malabarica</i> DC)	Dr. Sukh Dev
5.	Gokarn, A. N.	Ph.D.	Bombay	Kinetics studies in fluid-solid reactions	Dr. L. K. Doraiswamy
6.	Gopinathan (Mrs), S.	Ph.D.	Poona	Synthetic studies in organotin and substituted titanoxane compounds	Dr. J. Gupta

<i>S.No.</i>	<i>Name</i>	<i>Degree</i>	<i>University</i>	<i>Subject of thesis</i>	<i>Name of the guide</i>
7.	Iyer (Mrs.), P. K.	Ph.D.	Poona	Nucleotide metabolism (Nicotinamide-adenine dinucleotide phosphate) glycohydrolase from ox brain	Dr. V. Jagannathan
8.	Kale, K. M.	Ph.D.	Marath wada	Adiabatic compressibility of polyelectrolytes and other allied macromolecules	Dr. P. Roy Chowdhury
9.	Kamat, S. K.	Ph.D.	Poona	Synthesis of nitrogen heterocyclics	Dr. B. D. Tilak
10.	Kantak, U. N.	Ph.D.	Poona	Studies on some cyclopentadienyl titanium complexes	Dr. D. N. Sen
11.	Maskati (Miss), F. S.	Ph. D.	Poona	Studies on microbial enzymes	Dr. M. R. Raghavendra Rao
*12.	Nair, K. R. R.	Ph. D.	Poona	Spectroscopic and polarographic studies on molecular structure	Dr. P. M. Nair
13.	Om Prakash	Ph. D.	Poona	Studies on microbial enzymes (Nitrate reductase <i>A-fischeri</i>)	Dr J. C. Sadana
*14.	Pande, B. S.	Ph. D.	Nagpur	Studies in terpenes	Dr. Sukh Dev

<i>S.No.</i>	<i>Name</i>	<i>Degree</i>	<i>University</i>	<i>Subject of thesis</i>	<i>Name of the guide</i>
15.	Patil, B. G.	M. Sc.	Shivaji	Properties and functions of esterases	Dr. B. V. Ramachandran
*16.	Patil, S. D.	Ph. D.	Shivaji	Spectroscopic and kinetic studies on molecular structure and reactivity	Dr. P. M. Nair
17.	Patil, V. B.	Ph. D.	Poona	Natural anthraquinone pigments	Dr. K. Venkataraman
*18.	Pol, P. G.	Ph. D.	Poona	Synthesis and physical properties of some new phosphors	Dr. A. P. B. Sinha
19.	Ravikumar, P. R.	Ph. D.	Rajasthan	Studies in some Indian medicinal plants	Dr. Sukh Dev
20.	Sathe, V. M.	Ph. D.	Poona	Synthesis of oxygenated terpenoids	Dr. A. S. Rao
21.	Singh, A. M.	Ph. D.	Poona	Chemistry of lac and lac acids	Dr. Sukh Dev
*22.	Tadwalkar, V. R.	Ph. D.	Poona	Synthesis of terpenoids and steroids	Dr. A. S. Rao

* These are research fellows/guest workers

7.5 NCL scientists recognized by different universities as research guides.

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|--------------------------------|---|
| 1. Dr. Ayyangar, N. R. | Poona |
| 2. Dr. Bose, J. L. | Bombay, Poona, Nagpur, Shivaji |
| 3. Dr. Chakravati, K. K. | Poona, Shivaji, Karnatak |
| 4. Dr. Damodaran, V. | Shri Venkateswara University,
Tirupathi |
| 5. Dr. Das, K. G. | Poona, Bombay, Kerala
Marathawada, Kalyani |
| 6. Dr. Doraiswamy, L. K. | Poona, Bombay, Nagpur, Calcutta,
Jadavpur |
| 7. Dr. Ghatge, B. B. | Poona |
| 8. Dr. Ghatge, N. D. | Poona, Bombay, Shivaji - |
| 9. Dr. Gogte, V. N. | Shivaji |
| 10. Dr. Goswami, A | Poona, Shivaji |
| *11. Dr. Gupta J. | |
| 12. Dr. Ingle, T. R. | Poona |
| 13. Dr. Jagannathan, V | Bombay, Poona, Baroda |
| 14. Dr. Joshi, R. M. | Poona, Bombay |
| 15. Dr. Kapur, S. L. | Bombay, Poona, Punjab- |
| 16. Dr. Katti, S. S. | Bombay |
| *17. Dr. Kelkar, G. R. | Poona |
| 18. Dr. Kulkarni, G. H. | Nagpur |
| 19. Dr. Kulkarni (Miss), S. B. | Poona |
| 20. Dr. Kulkarni, S. N. | Poona, Bombay, Karnatak, Shivaji |
| 21. Dr. Mathur, H. B. | Poona, Agra, Madras, Bombay,
I.I.T. Bombay |
| 22. Dr. Mitra, R. B. | Poona |
| 23. Dr. Nair, P. M. | Poona, Shivaji, Andhra |
| 24. Dr. Narayanan, C. R. | Poona, Bombay, A. B. Univ
Zaria, Nigeria |
| 25. Dr. Pai, M. U. | Bombay |
| 26. Dr. Pant, L. M. | Poona |
| *27. Dr. Ramachandran, B. V. | Shivaji, Poona |
| 28. Dr. Rama Rao, A. V. | Shivaji, Poona |
| 29. Dr. Rangachari, P. N. | Poona, Shivaji |
| 30. Dr. Rao, A. S. | Poona, Shivaji |
| 31. Dr. Roy Chowdury, P. | Marathwada, Poona, Shivaji |
| 32. Dr. Sadana, J. C. | Poona, Lucknow |

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| 33. Dr. Sen, D. N. | Poona, Bombay |
| 34. Dr. Sethi, S. C. | Poona |
| 35. Dr. Sinha, A.P.B. | Poona, Banaras, Vikram, Bombay,
Karnatak, |
| 36. Dr. Siva Raman, C. | Poona |
| 37. Dr. Subbaraman, P. R. | Poona, Bombay, Kerala |
| 38. Dr. Sukh Dev | Poona, Bombay, Agra, Punjab,
I.I. Science-Bangalore, Rajasthan |
| 39. Dr. Tilak, B. D. | Bombay, Poona |
| *40. Dr. Venkataraman, K | Poona, Bombay, Madras, Banaras,
Karnatak |

* *Retired/Emeritus scientists*

7.6 Consultancy

During the year consultancy was offered to the following firms either through individual scientists or groups of scientists, wherein 15 scientists were involved.

1. M/s. Hindustan Organic Chemicals Ltd., Rasayani.
2. M/s. Hindustan Antibiotics Ltd., Pimpri, Poona-18.
3. M/s. Indian Petrochemicals Ltd., Baroada.
4. M/s. Vidarbha Organic Chemical Industries Pvt. Ltd., Nagpur.
5. M/s. Industrial Oxygen Co. (P) Ltd., Poona.
6. M/s. Hindustan Ferrodo Ltd., Bombay
7. M/s. National Rifles Ltd., Ahmedabad
8. M/s. Camphor and Allied Products Ltd., Bareilly.
9. M/s. Poona Synthetic Co., Poona
10. M/s, Aniline Dyestuffs & Pharmaceuticals Ltd., Bombay
11. M/s. Indian Dyestuffs Industries, Bombay

7.7 Membership of committees

<i>S. No.</i>	<i>Name of the scientist</i>	<i>Position</i>	<i>Name of the committee</i>
1.	Dr. Ayyangar, N. R.	Member	Planning Group V: Dyes, Intermediates for dyes, Pharmaceuticals and other fine organic/inorganic chemicals of National Committee on Science and Technology (NCST) Panel V.

<i>S. No.</i>	<i>Name of the scientist</i>	<i>Position</i>	<i>Name of the committee</i>
1.	Dr. Ayyangar, N. R.	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 materials Sectional Committee CDC 11/A2.21
4.	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/petro-chemicals/ polymers of NCST Panel V
		Member	Planning Group IX: Chemical engineering research and design capability development of NCST Panel V
5.	Dr. Goswami, A.	Member	National Committee on Science & Technology of Thin Films, Bangalore
6.	Dr. Ghatge, N. D.	Member	Board of Governors, Indian plywood Industries Research Institute, Bangalore.
7.	Dr. Jagannathan, V.	Member	Planning Group IV : Organic fine chemicals including pharmaceuticals drugs, perfumery chemicals (essential oils), physicochemicals, carbohydrates of NCST Panel V
		Member	Planning Group on the utilization of agricultural and animal wastes

<i>S. No.</i>	<i>Name of the scientist</i>	<i>Position</i>	<i>Name of the committee</i>
7.	Dr. Jagannathan, V.	Member	NCST Panel on consumer industries-groups on food and sugar
8.	Dr. Mathur, H. B.	Member	Planning Group II : Fertilizers and other plant nutrients of NCST Panel V
		Member	Variable Energy Cyclotron (VEC) Committee, Dept. of Atomic Energy, B.A.R.C., Bombay
9.	Dr. Mitra, R. B.	Member	Planning Group VII : Pesticides of NCST Panel V
		Member	ISI-Dye intermediate Sectional Committee-CDC 46
		Member	Electronic Commission of India, Govt. of Maharashtra, Bombay
10.	Dr. Pant, L. M.	Member	Indian National Committee for International Union of Crystallography
11.	Dr. Sinha, A.P.B.	Member	Planning Group VIII: Marine chemicals—forward technologies relating to use of marine chemicals of NCST Panel V
		Member	National Committee on Science & Technology, Planning group on special materials
12.	Dr. SivaRaman, C.	Member	Expert Committee on Biochemistry of Indian Council of Medical Research
13.	Dr. SubbRaman, P. R.	Member	ISI—Structural and Metal Divisional Council-SMDC-2 and Chemical standards Section Committee-CDC-I
14.	Dr. Sukh Dev	Member	Planning Group IV:Organic fine chemicals including pharmaceuticals, drugs, perfumery che-

<i>S. No.</i>	<i>Name of the scientist</i>	<i>Position</i>	<i>Name of the committee</i>
14.	Dr. Sukh Dev	Member	micals (essential oils) physico-chemicals, biochemicals, carbohydrates of NCST Panel V. Planning Group VIII: Marine chemicals-forward technologies relating to use of marine chemicals of NSCT Panel V
15.	Dr. Tilak, B.D.	Member Convener Director Chairman Director Member Member Chairman	NCST, Govt. of India NCST, Panel V for Chemical Industry Indian Petrochemicals Corporation Ltd., Baroda Board of Directors, Hindustan Organic Chemicals Ltd., Rasayani Hindustan Antibiotics Ltd., Pimpri, Poona. Defence R & D Council, Min. of Defence, New Delhi Court and Academic Council, Poona University Standing Committee for Dyestuff Industries
16.	Dr. Unni, M. K.	Member	ISI—Industrial Organic Chemicals Sub-Committee-CDC 4 : 14

8. PUBLICATIONS

8.1 *Research Papers*

Solid state materials

- Goswami, A. and Dhere, N. G.
Vapour phase deposits of bismuth selenide.
J. Vac. Sci. Technol., **9**, 523, (1972).
- Goswami, A. and Nikam, P. S.
A study of vacuum deposited films of CrTe and CrSe on single crystals.
Thin Solid Films, **11**, 353 (1972).

3. Goswami, A. and Koli, S. S.
A study on vapour phase deposits of PbSe films.
Indian J. of Pure and Applied Physics, **10**, 629 (1972).
4. Murthy, M.N.S. and Sinha, A.P.B.
Computational errors and consequent small alterations in a few term-energies of nickel and vanadous trigonal bipyramidal complexes.
Ind. J. of Pure and Appl. Physics, **10**, 179 (1972).

Physico-chemical studies

5. Roy Chowdhury, P. and Kale K. M.
Adiabatic compressibility of some amphoteric polyelectrolytes.
J. Appl. Polymer Sci., **16**, 2057 (1972).
6. Roy Chowdhury, P.
Adiabatic compressibility of polyelectrolytes.
Vijnana Parishad Anusandan Patrika, **15**, 21 (1972).
7. Badrinarayanan, S. and Mathur, H. B.
The correlation factor and the mass dependence for the diffusion of cobalt in copper.
Nuclear Physics and Solid State Physics (India), **14C**, 197 (1972).
8. Srivastava, S. K., Raju, E. V. and Mathur, H. B.
Thermodynamics of the interactions of Ni(II) with cysteine.
J. Inorg. and Nucl. Chem., **35**, 253 (1973).
9. Badrinarayanan, S. and Mathur, H. B.
Isotope effect for the diffusion of cobalt in copper.
Indian J. Pure and Appl. Phys., **10**, 512 (1972).

Physical organic chemistry

10. Das, K. G. and Madhusudanan, K. P.
Substituent effects on molecular ion abundances.
Ind. J. Chem., **10**, 277 (1972).
11. Das, K. G., Gogate, V. N., Seetha (Miss), M. and Tilak, B. D.
Stereochemistry of hydride transfer; Mass spectral evidence for the relative stabilities of the carbonium ions.
Ind. J. Chem., **10**, 924 (1972).
12. Das, K. G. and Thayumanavan, B.
Mass spectral studies of some oligosaccharide per methyl ethers.
Org. Mass Spectrum **6**, 1063 (1972).

13. Mishra, R. and Sukh Dev.
Formylation of PMR-shift for the detection of methyls on carbons linked to hydroxyl.
Tetrahedron Letters, **48**, 4865 (1972).
14. Rao, V. S. and Nair, P. M.
Proton chemical shifts in BF_3 -adducts of *p*-substituted benzaldehyde.
Indian J. Chem., **11**, 82, (1973).

Natural organic products

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Inorganic chemistry

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8.2 Publications in collaboration with outside scientists

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Evidence of two types of accessible surfaces in fibrous cotton.
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Distribution and structure of reagent residues resulting from wet-cure and boke-cure crosslinking of cotton with N-ethylbis (2-chloroethyl) amine.
Textiles Res. J. **42** 321 (1972).
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Terpenoids of Lodgepde Pine Bark.
Phytochemistry, **11**, 365 (1972).
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Some new tetranortriterpenoids from the wood of *Cedrela toona*.
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8.3 Chapters in the Books

1. Tilak, B. D. and Ayyangar, N. R.
Acridine dyes in acridines.
The chemistry of heterocyclic compounds, Vol. 9 (2nd Edition),
R. M. Acheson Ed., Interscience, New York (1973) pp. 579-613.
2. Sukh Dev.
Biogenetic concepts in terpene structure elucidation.
Some recent developments in the chemistry of natural products,
Prentice-Hall, New Delhi (1972).
3. Rama Rao, A. V. and Venkataraman, K.
Insect pigments derived from hydroxyanthraquinones.
Some recent developments in the chemistry of natural products,
Prentice-Hall, New Delhi (1972).

8.4 Review articles and general science papers

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Pesticides need stronger indigenous base.
"The Hindu", *Survey of Indian Industry 1972, Section IV, p. 115*.
2. Narayanan, C. R.
A popular article on Antimatter.
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NCL in the service of the nation.
Chemical Industrial News, (1972).

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1. Goswami, A.
Electron diffraction studies of thin films.
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New Delhi, November, 1972.
2. Goswami, A, Rao, B. V. and Prabhat Singh.
Structural and optical properties of niobium arsenide films.
Symposium on 'Solid state materials'
National Physical Laboratory, New Delhi, February, 1973.

3. Murthy, M. N. S., Deshpande, C. E. and Kanade (Miss), S. B.
Chemical techniques in the preparation of electroceramics.
Symposium on 'Solid state materials' sponsored by National Physical Laboratory and Solid State Physics Laboratory, New Delhi, February, 1973.
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Organic pollutants in air and water.
Seminar on 'pollution', IMA, Poona, February, 1973.
5. Das, K. G., Fales, H. M., Milne, G.W.A. and Pedder, D. J.
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6. Nayak, U. R.
Turpentine as a raw material for perfumery.
Seminar on 'Advances in perfumery,' Bombay, December, 1972.
7. Sen, D. N.
Metal complexes and reactivity of the chelated organic ligands.
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8. Rangachari, P. N. and Krishnamachar, V. S.
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Seminar on 'Bacterial leaching,' Jamshedpur, December, 1972.
9. Ingle, T. R.
Oxidation of starch.
All India starch round table conference, Mahabaleshwar, March, 1973.
10. Varma, J. P.
Dialdehyde starch.
All India starch round conference Mahabaleshwar, March, 1973.
11. Nair, P. M. and Rao, V. S.
Buttressing effects in some trisubstituted benzenes.
Kanekar memorial symposium, Chandigarh, January, 1973.
12. Tilak, B. D.
Azides, new reactive disperse dyes for cellulosic protein and synthetic fibres and their blends.
Symposium on 'Theory and practice on the application of reactive dyes' Bombay, February, 1973.

13. Ayyangar, N. R. and Abhyankar, S. M.
Some industrial chemicals from HOC products.
Seminar on 'Development of small scale and ancillary industries at HOC,' Rasayani, February, 1973.
14. Gogte, V. N.
Synthesis of heterocyclic compounds-approach to the synthesis of 11-azasteroids.
UGC symposium on 'Mechanistic organic chemistry,' Madras, February, 1973.
15. Kotasthane, A.N., Chavan, A.M., Lakhbir Singh and Biswas, A. B.
Hydrogenation of ethyl-oleate with copper-nickel catalyst.
Symposium on 'The science of catalysis and its application', Banaras Hindu University, Varanasi, March, 1973.
16. Gogte, V. N.
PMR investigation on the tautomeric and isomeric equilibria in 2-arylaminoethylene cycloalkanones.
Prof. C. R. Kanekar memorial symposium, Chandigarh, January, 1973.
17. Kapur, S. L.
Research and Development in man-made fibre industry.
All India Manufacturers Organization Seminar, Bombay, August, 1972.
18. Bose (Miss), B., Irani, R. K. and Bose, J. L.
Mechanism of stannic chloride catalyzed glycosidation of phenols.
6th International symposium on carbohydrate chemistry (IUPAC), Madison, USA, August, 1972.
19. Bose, J. L.
Cationic starch.
All India 1st starch round table conference, Mahabaleshwar, March, 1973.
20. Ramaswamy, V.
Thermodynamic properties of dichloro-tetrafluoroethane.
13th International Congress of Refrigeration, Washington, October, 1972.

8.6 *Book reviews*

"Chemical Engineering" by J. M. Conlson & J. F. Richardson, Vcl.3, Edited by J. F. Richardson and D. G. Peacock, Pergamon Press, 1971. Reviewed by Dr. M. U. Pai, in Journal of Scientific & Industrial Research, P. 343, 31(6), June, 1972.

9. PATENTS IN FORCE

9.1 *Indian 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 the production of 4-hydroxycoumarin and its derivatives.
Shah, V. R., Bose, J. L. and Shah, R. C.
6. **63083***
A new method for 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 Kapur, 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.
13. **66803**
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.K.
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 of 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. **109489**
Production of aryl-glycosides.
Ingle, T. R. and Bose, J. L.
28. **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.
29. **113406**
A new thermistor composition exhibiting high positive temperature coefficient of resistivity (posistor).
Brahmecha, B. G. and Sinha, K. P.

30. **116453**
Disperse dyes for polyesters with good affinity and sublimation fastness.
Raman, S. K. and Tilak, B. D.
31. **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.
32. **117210**
A direct process for the manufacture of dibutyltin di-iodide and of the oxide therefrom.
Gupta, J., Gopinathan, C., Gopinathan (Mrs.), S. and Awasarkar, P. A.
33. **117403**
Preparation of water thinnable emulsion paints from linseed oil.
Kapur, S. L. and Bakshi, S. H.
34. **118476**
Methods of preparing isocyanates, mono and polyureas from pentadecenyl phenol (anacardol).
Ghatge, N. D. and Yadav, S. D.
35. **121538**
A process for the shellac-polyester urethane compositions for use in textiles, water proofing adhesives, paints and varnishes and in electrical insulating varnishes.
Ghatge, N. D. and Srinivasan, S. R.
36. **123638**
Improvements in or relating to the manufacture of cashewnut shell gum (CNS-gum).
Ingle, T. R., Vaidya, S. H. and Pai, M. U.

* These patents (20) are being commercially exploited.

9.2 *Indian 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 constituent 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. **125138**
Method of preparing polyisocyanates and polyureas from pentadecenyl phenol (anacardol).
Ghatge, N.D. and Mahajan, S. S.
6. **126354**
A process for obtaining useful steroids from a new plant source.
Sukh Dev, Patil, V. D. and Nayak, U. R.
7. **126393**
An improved method for the manufacture of calcium hypophosphite.
Goswami, M., Lobo, J. and Brahme, P. H.
8. **127743**
A process for obtaining colchicine from a new plant source.
Kapadia, V. H., Sukh Dev and Rao, R. S.
9. **127750**
A process for production of pyridoxine hydrochloride.
Joshi, C. G. and Sukh Dev.
10. **130254**
A process for the manufacture of vulcanizable graft copolymer for use as a synthetic rubber and as a base in coating compositions.
Joshi, R. M.
11. **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.

12. **131606**
Improvements in or related to a process for the preparation of gamma ferric oxide.
Lakshbir Singh, Chavan, A. M. and Kotasthane, A. N.
13. **134641**
A device for warming and cooling.
Lakshbir Singh and Kotasthane, A. N.
14. **328/72****
A new process for the halogenation of copper phthalocyanine.
Ayyangar, N. R., Moghe, P. P. and Tilak, B. D.
15. **2229/72****
New dyes containing azido groups for cellulosic protein and synthetic fibres.
Ayyangar, N. R., Badami, N. V., Tilak, B. D. and Daruwalla, E. H.
16. **2231/72****
Improvement in or relating to the preparation of new reactive dyes.
Ayyangar, N. R., Badami, N. V., Tilak, B. D. and Daruwalla, E. H.
17. **2232/72****
Process for the preparation of D-Galactose from cashewnut shells.
Ingle, T. R., Vaidya, S. H. and Pai, M. U.
18. **2235/72****
New dyes containing azido groups for cellulosic protein and synthetic fibres.
Ayyangar, N. R., Badami, N. V., Tilak, B. D. and Daruwalla E. H.
19. **2258/72****
Improvements in or relating to the preparation of new reactive dyes.
Ayyangar, N. R., Badami, N. V., Tilak, B. D. and Daruwalla, E. H.
20. **2259/72****
Improvements in or relating to the preparation of new reactive dyes.
Ayyangar, N. R., Badami, N. V., Tilak, B. D. and Daruwalla, E. H.

21. 324/Cal/73**

Improvements in or relating to the preparation of new reactive dyes derived from copper phthalocyanine.

Ayyangar, N. R., Badami, N. V., Tilak, B. D. and Daruwalla, E. H.

22. 325/Cal/73**

Improvements in or relating to the preparation of new reactive finishing agents for textile fibres.

Ayyangar, N. R., Lugade, A. G., Tilak, B. D., and Daruwalla, E. H.

** These patents have been newly filed during the year.

9.3 Foreign patents in force

1. Belgium Patent No. 75806.

A process for obtaining useful steroids from a new plant source. (corr. to Indian patent No. 126354).

Sukh Dev, Patil, V. D. and Nayak, U. R.

2. U.S.A. Patent No. 3070625.

A process for the preparation of azelaic acid semi ester suitable for making civetone dicarboxylic acid. (corr. to Indian patent No. 58868).

Nayak, U. G., Chakravarti, K. K. and Bhattacharyya, S. C.

3. U.S.A. Patent No. 740088.

Preparation of exaltone. (corr. to Indian patent No. 59419).

Ghatge, B. B., Nayak, U. G., Chakravarti, K. K. and Bhattacharyya, S. C.

4. U.S.A. Patent No. 2946783.

Preparation of costus root oil and the production thereof. (corr. to Indian patent No. 59853).

Kelkar, G. R. and Bhattacharyya, S. C.

5. Cuba Patent No. 149082.

Manufacture of nicotine sulphate from tobacco or tobacco waste. (corr. to Indian patent No. 45666).

Gedeon, J. and Goswami, M.

6. U.S.A. Patent No. 2749247.
Air drying wrinkle finish coating composition. (corr. to Indian patent No. 44737).
Aggarwal, J. S. and Sharma, P. G.
7. U.S.A. Patent No. 2685593.
Manufacture of useful compounds and products from the seed oil of *mallotus philippinensis* Muell, Arg. (Kamala). (corr. to Indian patent No. 44736).
Aggarwal, J. S., Sharma, V. N. and Gupta, S. C.

RESEARCH UTILIZATION

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

S. No.	Name of the process/product & Indian Patent No.	Field of utilization	Name of the manufacturer (year of commencement of production)	Production		Terms of release
				1972-73	Upto Mar.1972	
				Qty/Value Rs. in lakhs.	Qty/Value Rs. in lakhs.	
1	2	3	4	5	6	7
1.	Acetanilide	Intermediates	M/s. Hindustan Organic Chemicals Ltd., 1820 T Rasayani, through project engineers M/s. R.L. Dalal & Co., Bombay-18 (1969).	2785.75 T 196.07		Non-exclusive
2.	Acriflavine	Pharmaceuticals	M/s. Western India Fine Chemicals, 83, Lalbahadur Shastri Marg, Bombay-80 (1969).	950 Kg. 3.96	2679 Kg. 11.82	Sponsored
3.	Adhesives for decorative laminates	Laminates	M/s. Swastik Rubber Products, Ltd., Poona-3 (1969).	12 T 1.27	52.75 5.26	Sponsored

1	2	3	4	5	6	7
4.	Anion exchange resin from melamine (71190)	Demineralization of liquids	M/s. Tulsi Industries, Bhosari Indl. Area, Poona-26. (1963).	280 Cu. ft. 0·30	565 Cu. ft. 1·75	Non-exclusive
5.	Antipriming compositions (77081)	Antipriming in locomotives	Research, Design and Standard Organisation, M & C Wing, Chittaranjan (1964).	9·6 T 0·30	98·70 T 2·60	Non-exclusive
6.	Bacterial diastase (66096)	Textile desizing	M/s. Chemaux (P) Ltd., Sitaladevi Temple Rd., Mahim, Bombay-16 (1967).	1·75 (Approx. upto 30-9-72).	341·58 17·25	Exclusive
7.	Berberine hydrochloride	Pharmaceuticals	M/s. Nitin Pharmaceuticals, 180/82, Samuel Street, Bombay-9 (1965).	4·0 T 8·00	16·30 T 32·36	Sponsored
8.	<i>tert</i> -Butylcatechol	Synthetic rubber	M/s. Percynic Chemicals, United Bank of India Bldg., Sir P.M. Road, Bombay-1 (1972).	6·3 T 2·90	1·5 T 0·77	Non-exclusive
*9.	Butyl titanate	Varnishes, enamels	M/s. Synthochem, 33-A, Laxmi Nagar, Indore-1 (1973).	16·6 T 5·00	—	Non-exclusive

1	2	3	4	5	6	7
10.	Cadmium sulphide photoconductive cells	Electronics	M/s. Chinoy Electronics, 64, Koregaon Park, Poona-1 (1971).	440 Nos. 0·05	631 Nos. 0·08	Non-exclusive
11.	Calcium hypophosphite (126393)	Pharmaceuticals	M/s. Hypophosphite & Co., 79-F, Princess Street, Bombay-2 (1967).	15 T 9·00	35·25 T 19·30	Sponsored
12.	Calcium silicate	Low density insulators	M/s. Newkem Products Corpn. Harganga Mahal, Bombay-14 (1968).	255 T 9·00	767·21 T 27·00	Sponsored
13.	Can sealing composition (66194)	Metal can industry	M/s. Arya Chemical Works, 114/2A, Dharmatolla Street, Calcutta-13 (1962).	31·8 T 1·80	183·40 T 10·14	Exclusive
14.	Carbimazole	Pharmaceuticals	M/s. Indian Schering Ltd., Sion-Trombay Road, Chembur, Bombay-71 (1970).	11·4 Kg. 0·94	38 Kg. 3·15	Sponsored
15.	Catechol	Pharmaceuticals	M/s. Percynic Chemicals, Bombay-1 (1972).	7·20 T 1·80	1·95 T 0·34	Non-exclusive

1	2	3	4	5	6	7
16.	Cation exchange resin from CNSL (59497, 59606)	Demineralization of liquids.	M/s. Tulsi Industries, Poona-26 (1960).	— 0.30 (approx.)	7310 cu. ft. 6.14	Non-exclusive
17.	Chloral hydrate	Industrial chemicals	M/s. Hindustan Insecticides Ltd., Industrial Area, New Delhi-15 (1963).	0.05 T 0.007	20.73 T 2.13	Sponsored
*18.	Clofibrate	Pharmaceuticals	M/s. Biological Evans, 18/1 & 3, Azamabad, Hyderabad-20 (1973).	100 Kg. 0.17 lakhs (approx.)	—	Non-exclusive
19.	Diethyl-m-amino phenol	Dye intermediate	M/s. Sahyadri Dyestuffs and Chemicals (P) Ltd., 117, Vithalwadi Road, Poona-9 (1973).	37.8 T 24.57	13.64 T 6.26	Sponsored
20.	Dihydroisogajasmone and peach aldehyde	Perfumery	(i) M/s. S. H. Kelkar & Co. (P) Ltd., Bombay-Agra Road, Bombay-80 (1965). (ii) M/s. Sonebon Laboratories, Kottayam-5 (1973).**	1.8 T 3.50 0.07 T 0.18	5169 Kg. 10.47 —	Non-exclusive Non-exclusive
*21.	Dimethylaniline	Industrial chemicals	M/s. Sahyadri Dyestuffs and Chemicals P. Ltd., Poona (1972).	25 T 2.50	—	Non-exclusive

1	2	3	4	5	6	7
22.	Ethylene oxide condensates	Surface active agents.	M/s. Hico Products (P) Ltd., Mogal Lane, Bombay-16 (1965).	514 T 70.00	1859 T 217.88	Sponsored
23.	Ferrites (Hard)	Electronics	M/s. Semiconductors Ltd., Nagar Road, Poona-14 (1968).	5.2 T 1.30	2.6 lakh Nos. 2.80	Non-exclusive
24.	Geraniol, citronellal and citronellol	Perfumery	M/s. Opal Fine Chemicals, Veer Savarkar Marg, Bombay-25 DD. (1970).	4 T 4.40	5 T 6.10	Sponsored
25.	4-Hydroxycoumarin (62890, 63083)	Pharmaceuticals	M/s. Unichem Laboratories Ltd., Jogeshwari, S. V. Road, Bombay-60 (1964).	—	489.70 Kg. 1.296	Exclusive
26.	β -Ionone (77225)	Perfumery, Pharmaceuticals	M/s. Industrial Perfumes Ltd., Army & Navy Bldg. M. G. Road, Bombay-1 (1968).	25.23 T 40.37	51.33 T 48.96	Non-exclusive
27.	Nicotine sulphate	Insecticides	M/s. Urvakunj Nicotine Industries Petlad-Cambay Road, Dharmaj, (Dist. Kaira) (1963).	48 T 8.80	39.81 T 6.64	Non-exclusive
28.	Orthotolyl biguanide	Soap	M/s. Industrial Perfumes Ltd., Bombay-1 (1970).	1.52 T 0.41	4.95 T 1.34	Exclusive

1	2	3	4	5	6	7
29.	Perfumery products based on longifolene (Capinone)	Perfumery	M/s. Camphor and Allied Products, Bareilly (1968).	6.4 T 5.26	22.153 T 18.28	Sponsored
30.	Perfumery products based on Δ^3 -carene (Meracene)	—do—	—do— (1968).	4.5 T 2.10	4.502 T 1.59	Sponsored
31.	β -Phenethyl alcohol	Perfumery	M/s. Sunanda Aromatic Industries, K.R.S. Road, Mettagalli, Mysore (1970).	35 T 12.25	34 T 11.30	Sponsored
32.	Phthalates-diethyl and dibutyl	Plasticizers	(i) M/s. Alta Laboratories Ltd., Giri Vihar, Khopoli, (Dist. Kolaba) (1969). (ii) M/s. India Carbon Ltd., Dr. A. B. Road, Worli, Bombay-18 (1971).	115.6 T 7.17 2655 T 141.00	1718.5 T 116.26 2178 T 121.17	Non-exclusive Non-exclusive
33.	Phthalates-diethyl and dimethyl	Plasticizers	M/s. Mysore Acetate and Chemicals Co. Ltd., Mysugar Buildings, Sir J. W. Road, Bangalore-2 (1970).	146 T 10.00	55.02 T 4.20	Non-exclusive

1	2	3	4	5	6	7
34.	Polyurethane printing rollers (86991)	Printing	M/s. Sree Saraswathy Printing Press Ltd., 32, Acharya P. C. Road, Calcutta-9 (1965).	303 pieces of rollers 0.50	—	Non-exclusive
35.	Rigid filters (59608, 66966)	Tube wells	M/s. Ashim Filters, 196, Defence Colony, New Delhi (1965).	2800 Mtrs. 5.86	6420 Mtrs. 13.16	Non-exclusive
36.	Rubber blowing agent	Rubber chemicals	M/s. Swastik Rubber Products Ltd., Poona-3 (1968).	6.23 T 0.85	169 T 23.71	Non-exclusive
37.	Rubberized cork sheet	Gaskets	M/s. Bharat Casements (P) Ltd., P. B. No. 89, Baroda (1966).	25 lakh pieces 9.43	46.23 lakh Nos. 15.88	Non-exclusive
38.	Rubber reclaiming agent	Rubber chemicals	M/s. Swastik Rubber Products Ltd., Poona-3 (1968).	7.68 T 1.52	18.38 2.46	Non-exclusive
39.	Sachets—Hot and Cold	Substitute for hot water bag and ice bag	(i) M/s. Thermo Chem Laboratories, A-39, H-Block, MIDC, Pimpri, Poona-18 (1972). (ii) M/s. Vasant Industrial Corpn., 356, Great Nag Road, Nagpur-2. (1971).	6000 pads 1.00 800 pads 0.08 (approx.)	— —	Non-exclusive Non-exclusive

1	2	3	4	5	6	7
40.	Silica gel	Humidity control	M/s. Minco Products, 301/27, T. H. Road, Madras-21 (1963).	12 T 0.96	83 T 5.71	Sponsored
41.	Sorbide nitrate	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay-71 (1969).	175 Kg. 2.42	328 Kg. 6.63	Sponsored
42.	Thermistors	Electronics	(i) M/s. Semicconductors Ltd., Poona-14 (1963).	10.48 lakh Nos. 7.80	36.67 lakh Nos. 31.68	Non-exclusive
			(ii) M/s. Tempo Semiconductors, Subhash Road, Vile Parle (E) Bombay-57 (1963).	19,600 Nos. 0.98	45,000 Nos. 1.81	Non-exclusive
43.	Vapour phase chromatograph	Instruments	M/s. Associate Instruments Manufacturers (India) P. Ltd., 26-27, Asaf Ali Road, New Delhi (1969).	27 units 5.40	65 units 12.95	Exclusive
44.	Warfarin	Rodenticides	M/s. Unichem Laboratories Ltd., Bombay-60 (1968).	—	179.60 Kg 1.04	Released along-with item No. 25.

* During the period under review, production has been newly reported on these items.

** Production reported for the first time by the party.

Table 1 : (Contd.)

Value of some major chemicals produced in Fine Chemicals Project

	Value in Rs.
45. Alumina (Column & TLC grade)	28,740
46. Silica gel (Column & TLC grade)	1,00,195
47. Sodium metaperiodate	31,680
48. Phenyl isothiocyanate	4,950
Total	Rs. 1,65,565

VALUE OF PRODUCTION BASED ON NCL KNOW-HOW

Year	No. of items manufactured (including major FCP items)	Value of production (Rs. in lakhs)
1950—65	15	14·82
1965—70	53	278·15
1970—71	55	283·84
1971—72	51	471·20
1972—73	48	557·11
	Total	1605·12

Note : (1) The following processes which were included in Table I of the Annual Report 1971-72 have now been dropped :—

- (a) Hexylresorcinol
- (b) Liquid rubber
- (c) Radio opaque dyes
- (d) Rubber based contact adhesive
- (e) Sisal wax

Two of them (Radio opaque dyes and Sisal wax) on which production is likely to be resumed by one licensee or the other appear in Table II. Other processes will not be reported in future reports unless possibility of their commercial utilization is envisaged.

(2) Peach aldehyde was shown as a separate process in last year's Annual Report. Since the technology is a part of the know-how for dihydro-isojasmone, both are shown together.

TABLE II : PROCESSES RELEASED AND AWAITING PRODUCTION

S. No.	Name of the process and Ind. Pat. No.	Field of utilization	Name of the party and year of release	Terms of release	Present status of implementation *
1	2	3	4	5	6
1.	Aniline	Organic intermediate	M/s. Hindustan Org. Chemicals, Rasayani (1973).	Non-exclusive	B
2.	Benzoic acid from toluene	Pharmaceuticals	M/s. Aniline Dyestuffs & Pharmaceuticals, Pvt. Ltd., 28, Apollo St., Bombay-1. (1969).	Sponsored	C
3.	Benzoic acid (IP) from crude methyl benzoate	—do—	M/s. Indian Petrochemicals Corpn. Ltd., P.O. Jawaharnagar, Baroda. (1972).	Non-exclusive	A
4.	Substitutes for bostik sealants	Adhesives	M/s. Swastik Rubber Products Ltd., Poona-3 (1972).	Non-exclusive	B
5.	Butylated hydroxy anisole	Antioxidant	M/s. Mhatre Arts Studio, Mahant Road, Vile Parle, Bombay-57 (1970).	Sponsored	C

* For explanation of codes please see note on page 133

1	2	3	4	5	5
6.	Calcium silicate from wollastonite	Industrial inorganic chemicals	M/s. Chemicals India, Chetak Bldg., P.B.No. 4, Udaipur. (1971).	Sponsored	B
7.	Can lining composition (based on nitrile rubber latex)	Lining cans for storing mineral oils, greases, food, etc.	M/s. Arya Chemical Works, 141/2A, Dharamtola St., Calcutta-13 (1972).	Non-exclusive	A
8.	Carbazole dioxazine violet pigment	Organic pigment	M/s. Square Chemicals, 73, Sant Tukaram Road, Bombay-9 (1973).	Non-exclusive	A
9.	Cation exchange resins polystyrene base (74356)	Demineralisation of liquids	M/s Tulsi Industries, Poona (1965).	Non-exclusive	C
10.	Cationic dyes for acrylic fibres	Dyes for synthetic fibres	M/s. Sahyadri Dyestuffs & Chemicals Pvt. Ltd., Poona (1972).	Sponsored	B Work being reinstated.
11.	Chlorides from bauxite residue	Industrial inorganic chemicals	M/s. Dharamsi Morarji Chemicals Co. Ltd., Prospect Chambers, 317/21, Dr. D. N. Road, Bombay-1 (1972).	Sponsored	B

1	2	3	4	5	6
12.	Chlorobenzenes	Industrial chemicals	M/s HOC, Rasayani (1969).	Sponsored	B Installation of 4000 TPA plant completed.
13.	Chlorohydroxyquinoline	Pharmaceuticals	M/s. Biological Evans Ltd., 18/1 and 3, Azamabad, Hyderabad (1972).	Sponsored	A
14.	Chloromethanes	Industrial chemicals	M/s. Standard Alkali, Chem. Div., Standard Mills Co. Ltd., Mafatlal Centre, P.B.No. 1038, Bombay-1 (1969).	Exclusive for 2 years	B 1 ton per day semicommercial plant successfully commissioned. 3000 TPA plant under erection.
15.	Coating for oil filter papers	Oil filtration	M/s. White Cloud Paper Mills, 412, Gultekdi Road, Poona-9 (1968).	Non-exclusive	B
16.	Coumarin	Perfumery	M/s. Lona Industries P. Ltd., Ladivali, Tal. Panvel, Dist. Kolaba (1970).	Sponsored	C

1	2	3	4	5	6
17.	Ethylene from alcohol	Organic intermediates	M/s. Industrial Oxygen Co. Ltd., Poona-1 (1972).	Non-exclusive	A
*18.	Ferrites-Hard	Electronics	M/s. Ajanta Enterprises, Mahalaxmi Indl. Estate, Gandhi Nagar, Bombay-13. (1971).	Non-exclusive	B
19.	Flexible magnets	Refrigeration gaskets, toys, educational kits	M/s. Ajanta Enterprises, Mahalaxmi Indl. Estate, Gandhi Nagar, Bombay-13 (1973).	Non-exclusive	A
20.	Foundry core binder	Core binder in steel foundries for high dimensional accuracy	M/s. Cardchem Industries, Hyderabad (1973).	Non-exclusive	A
21.	Hexachloroethane	Industrial chemicals	M/s. Industrial Oxygen Pvt. Ltd., Off Nagar Road, Poona-14 (1971).	Non-exclusive	B Plant of 150 TPA installed.
22.	8-Hydroxyquinoline	Pharmaceuticals	M/s. Alta Laboratories Ltd., Khopoli (1970).	Sponsored	B

1	2	3	4	5	6
*23.	β -Ionone (77225)	Perfumery chemical, intermediate for Vitamin A	M/s. S. H. Kelkar and Co., Bombay (1964).	Non-exclusive	C The firm is producing the item. According to them they are not using NCL technology.
24.	Ketene from acetone	Intermediates	M/s. Sudarshan Chemical Industries, Poona (1971).	Sponsored	B
25.	Liquid stabilizer for PVC	Plastics	M/s. Coated Fabrics (P) Ltd., 426/1, Chatushringi Road, Poona-16 (1969).	Sponsored	B
26.	Megimide	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay-71 (1969).	Sponsored	B
27.	<i>p</i> -Menthane hydroperoxide	Synthetic rubber	M/s. Camphor & Allied Products, Bareilly, (1969).	Exclusive	B
28.	l-Menthol from dementholized peppermint oil	Fine chemicals, drugs	M/s. Bhavana Chemicals, Ltd., Laxmi Ins. Building, Bombay-1 (1968).	Sponsored	B

1	2	3	4	5	6
29.	Monochloroacetic acid	Weedicides, CMC	M/s. HICO Products (P) Ltd., Bombay-16 (1972).	Non-exclusive	B
30.	Monoethylaniline	Intermediates for explosives	M/s. Atul Products Ltd., Atul, Dist. Bulsar (1973).	Non-exclusive	A
31.	Extraction of morphine and other alkaloids from lanced poppy straw	Pharmaceuticals	Ministry of Finance, Govt. of India New Delhi (1972).	Sponsored	A
*32.	Nicotine sulphate	Insecticides, fungicides	(i) Shri P. Jaipuria, Calcutta-14 (1972).	Non-exclusive	A
			(ii) M/s. Tobacco Bye Products (P) Ltd., Fort, Narsarawpet, Guntur (1963).	—do—	C
			(iii) M/s. Corromandal Tobacco Byeproducts, P. O. Gannavaram, A.P. (1970).	—do—	C
33.	Nitrile rubber	Oil resistant rubber formulations, adhesives	M/s. Synthetics & Chemicals Ltd., Bombay-20 (1973).	Non-exclusive	A

1	2	3	4	5	6
34.	<i>p</i> -Nitroacetophenone	Drug intermediates	M/s. Dey-Se Chem Ltd., 8, Rifle Range Road, Calcutta-29. (1971).	Sponsored	B
35.	<i>p</i> -Nitrophenol	Insecticides	M/s. HOC, Rasayani, (1972).	Non-exclusive	B
36.	Nonylphenol	Surface active agents	M/s. Surfactants (P) Ltd., 28, Apollo Street, Bombay-1 (1969).	Sponsored	B
37.	Opium alkaloids	Pharmaceuticals	Min. of Finance, Govt. of India, New Delhi (1966).-	Exclusive	4.7 TPA plant under erection
38.	Oxalic acid from bark of Ain tree	Industrial chemicals	M/s. Vidharbha Organic Chemical Industries, Dhote Bldg. Sitabuldi, Nagpur (1972).	Sponsored	B
39.	Oxy-urea	Textile chemical	M/s. Calico Chemicals & Plastics Divn., Anik-Chembur, Bombay-74 (1971).	Sponsored	B
40.	Pentachlorophenol and other chlorophenolic compounds	Fungicides	M/s. Bombay Chemicals (P) Ltd., Bombay -1 (1969).	Sponsored	C

1	2	3	4	5	6
41.	<i>p</i> -Phenetidine	Pharmaceuticals	M/s. Kabbur Industries Ltd., 3, Bastion Road, Bombay-1 (1971).	Sponsored	B
42.	Phenylacetic acid	Perfumery, penicillin G	M/s. Laxmi Chemical Industries (P) Ltd., Hyderabad-1 (1972).	Non-exclusive	A
*43.	Phthalates-dioctyl and dibutyl	Plasticizers	M/s. Synthetic Organics, Amar Hill, Saki-Vihar Road, Bombay-72 (1971).	—do—	B
44.	DC Recording polarograph	Polarographic analysis	(i) M/s. Elico (P) Ltd., Hyderabad-18 (1972). (ii) M/s. Laxsons Engg. & Electronics (P) Ltd., Bombay-29 (1973). (iii) M/s. Chromatography & Instruments Co., Baroda (1973).	—do— —do— —do—	A A A
*45.	Polyurethane printing rollers (86991)	Prnting rollers	M/s. United Ink & Varnish Co., P.B. No. 6862, Vile Parle, Bombay-57 (1965).	—do—	C The firm is producing the item. According to them they are not using NCL technology.

1	2	3	4	5	6
46.	Potentiometric strip chart recorder	Recording instruments.	(i) M/s. Associated Instruments Manufacturers (India) Pvt. Ltd., New Delhi (1970).	Non-exclusive	B
			(ii) M/s. Bagga Electronics, Bombay-4 (1972).	—do—	B
47.	Propoxyphene	Pharmaceuticals	M/s. Sarabhai Res. Centre, Wadi Wadi, Baroda (1972).	Sponsored	A
48.	Radio opaque dyes	Pharmaceuticals	M/s. Unichem Laboratories, Bombay-16 (1970).	—do—	C
49.	Radiosonde thermistors	Meteorology	M/s. The Bhagyanagar Laboratories, Hyderabad (1973).	Non-exclusive	A
50.	Removal of silica from black liquor	Paper industry	M/s. The Central Pulp Mills Ltd., 1183, Shivajinagar, Poona-4 (1971).	Sponsored	B Commercial plant under installation.
*51.	Sachets-hot and cold	Substitute for hot water bag and ice bag.	M/s. Biswanath Fatesaria, Calcutta-7 (1973).	Non-exclusive	A

1	2	3	4	6	6
52.	Solvent extraction of sandalwood oil	Perfumery	M/s. Govt. Sandalwood Oil Factory, Bangalore (1973).	Sponsored	A
53.	Sisal wax (64958, 65440)	Cosmetics, polishes	M/s. Aphali Pharmaceuticals Ltd., Station Road, Ahmednagar (1966).	Non-exclusive	C
54.	70% Sorbitol	Pharmaceuticals, Vitamin C synthesis	M/s. Maize Products Ahmedabad (1973).	Non-exclusive	A
55.	Direct reading spectrophotometer/Colorimeter	Biochemical research, spectroscopic analysis in visible range	(i) M/s. The Scientific Instruments Manufacturing Co. Ltd., Allahabad (1972).	—do—	A
			(ii) M/s. Neotronics Corpn. Bombay-80 (1972).	—do—	A
56.	Styrene DVB-base cation exchange resin (65976, 98155, 98156)	Demineralisation of liquids	(i) M/s. Industrial & Agricultural Engg. Co. (P) Ltd., 43, Forbes St. Bombay-1 (1967).	—do—	C
			(ii) M/s. Tulsi Industries, Poona-26 (1968).	—do—	All parties are producing the product. According to them they are not using NCL technology.
			(iii) M/s. Bird & Co. Ltd., Chartered Bank Bldg., Calcutta-1 (1963).	—do—	

1	2	3	4	5	6
57.	Sulphacetamide and its sodium salt	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay-71 (1969).	Sponsored	B
58.	Terpineol	Perfumery	M/s. Dujodwala Industries, 14-1 Mile, Mathura Road, Faridabad (1972).	Non-exclusive	B
59.	Thermosetting resins for industrial laminates	Industrial laminates	M/s. Formica India Ltd., Chinchwad, Poona-19 (1972).	Sponsored	B
60.	Vat Golden Yellow GK	Dye intermediates	M/s. Amar Dye-Chem Ltd., P.B.No. 6471, Bombay-16 (1967).	—do—	B
61.	Vitamin C	Pharmaceuticals	M/s. Hindustan Antibiotics Ltd., Pimpri, Poona-18 (1966).	Non-exclusive	B Plant of 120 TPA capacity under commissioning.
62.	Xylit	Pharmaceuticals, fine chemicals	M/s. Unichem Laboratories Ltd., Bombay (1972).	Sponsored	B

A — Processes recently released.

B — Progress reported, likely to be implemented soon.

C — Progress not reported, implementation expected.

Note : 1. Asterisk indicates process already mentioned in Table I.

2. The following 14 processes which were included in Table II of the Annual Report 1971-72 have now been dropped as the licensees have not shown any progress towards implementation for a considerable period.

B.D. catalyst; Conversion of bauxite into anhydrous aluminium chloride; Covering material from CNSL; Dithranol; D.D.T.-Water dispersible; Heat sealable coating compositions; Hexylresorcinol; Isolation of borneol; Liquid rubber; Pulping of Kashmir soft woods; Quinacridone pigments; Rubber based contact adhesive; Terpin G; Titanium tetrachloride.

These processes will again appear in future reviews as and when any progress for their implementation is reported.

TABLE II (A) : REVIEW OF RESEARCH UTILIZATION OF PROCESSES DEVELOPED BY NCL ON ITS OWN.
(Position as on 31st March every year)

Year	No. of processes in production	Value of production Rs. in lakhs.	No. of processes released and not in production	No. of processes available but not yet released**	Total No. of processes developed (2+4+5)	% of the processes in production to the total No. of processes developed.	No. of parties who have acquired NCL know-how
1	2	3	4	5	6	7	8
1969*	26	31.65	10	29	65	40	47
1970	27	60.50	14	34	75	36	48
1971	30	190.43	14	39	83	36	52
1972	29	350.02	22	56	107	27	58
1973	29	401.07	31	51	111	26	75

Break-up of 31 processes which have been released and are awaiting production is as follows:—

A — 15 — Processes recently released.

B — 10 — Processes on which progress has been reported.

C — 6 — Processes on which progress has not been reported or on which production has been discontinued for market/economical/technical reasons.

Total value of industrial production for the years 1950—68 exclusive of those arising from sponsored research schemes and FCP production was Rs. 44.75 lakhs.

These processes have been referred to NRDC for release.

TABLE II (B) : REVIEW OF THE PROCESSES DEVELOPED BY NCL UNDER SPONSORSHIP BY INDUSTRY.
(Position as on 31st March every year)

Year	No. of processes in production.	R & D inputs Rs. in lakhs.	Value of production Rs. in lakhs.	No of processes awaiting production	No. of processes on which work was abandoned for technical/ economical/ market reasons	No. of processes on which no production is envisaged *	Total No. of industrial processes developed (2+5+6)	% utilization of the processes developed.	Total No. of parties who have sponsored the processes
1	2	3	4	5	6	7	8	9	10
Upto									
1968	5	28.34	46.02	9	2	5	16	31	18
1969	13	7.11	48.06	16	2	8	31	42	30
1970	15	7.17	68.08	15	9	18	39	38	42
1971	19	6.68	92.72	15	13	20	47	40	46
1972	16	8.67	120.67	28	13	22	57	28	55
1973	15	4.54	154.39	36	13	22	64	23	61

* This column includes projects where no industrial production was expected to be realised. These include PI-480 schemes, Lac Cess Committee Project, ICMR schemes, Projects for development of analytical procedures, etc.

TABLE III KNOW-HOW AVAILABLE

A — TECHNOLOGIES WHERE CAPITAL REQUIREMENT IS UPTO Rs. 10 LAKHS

S. No.	Name of the process/product	Field of utilization	Imported raw material, if any	Scale of development	Terms of release (provisional)	Remarks
1	2	3	4	5	6	7
1.	Antipriming composition	Antipriming in locomotives	—	30 kg./emulsion per day	Non-exclusive; R.—2%	Released to one party; In production
2.	L-Arabinose (CP)	Biochemical research	—	500 g./batch	Non-exclusive; P—Rs. 15,000 R—2%	Export potential.
3.	Benzoic acid from crude methyl benzoate	Pharmaceuticals	—	2 kg./batch	Non-exclusive; P—Rs. 40,000	Released to one party.
4.	Bisphenol-A	Epoxy resins	—	10 kg./batch	Non-exclusive; P—Rs. 10,000 R—1 %	—

P—Premium
R—Royalty

1	2	3	4	5	6	
5.	Substitute for Bostik sealant	Oil resistant adhesive for fuel tanks	Nitrile rubber	20 kg./batch	Non-exclusive; P—Rs. 2,500 R—2%	Released to one party; In experimental production.
6.	tert-Butyl catechol	Stabilizer and polymerization inhibitor for synthetic rubber	tert-Butyl alcohol	10 kg./batch	Non-exclusive; P—Rs. 10,000 R—1%	Released to one party; In production.
7.	Butyl titanate	Insulating varnishes, special paints, catalyst	Titanium tetrachloride	10 kg./batch	Non-exclusive; P—Rs. 10,000 R—1 %	Released to one party; In production.
8.	Cadmium sulphide photocells	Instruments, photo-electric devices	Indium, Woods metal	50-100 cells/ batch	Non-exclusive; P—Rs. 12,000 R—30 P./cell	Released to two parties; One in production.
9.	Can lining composition	Lining cans for storing mineral oils, greases, food	Nitrile rubber	4 kg./batch	Non-exclusive; P—Rs. 5,000 R—3½%	Released to one party.
10.	Carbazole Dioxazine Violet pigment	Organic pigment	Carbazole, Chloranil, Diethyl-sulphate	1 kg./batch	Non-exclusive P—Rs. 25,000 R — 1 %	—

1	2	3	4	5	6	7
11. Catechol	Organic intermediate		—	7-8 kg./batch	Non-exclusive; P—Rs. 10,000 R—50 P./kg.	Released to one party; In production.
12. Cellulose powder	Chromatography, coating for electrodes, filter media		—	1 kg./batch	Non-exclusive; P—Rs. 5,000 R—2.5%	—
13. Clofibrate	Drug		<i>p</i> -Chlorophenol	1 kg./batch	Non-exclusive; P—Rs. 10,000 R—2%	Released to one party In; experimental production.
14. Coating for oil filter papers	Oil filters		—	6 kg./batch of coating resin	Non-exclusive; P—Rs. 5,000 as technical aid	Released to one party; In experimental production.
15. Costus root oil	Perfumery		—	1 kg. of costus root oil	Non-exclusive; P—Rs. 25,000 for costus root oil;	Costus roots are available in Kashmir and Punjab.
Fructose	Medicine		—	200 g./batch	additional Rs. 5,000 for fructose and chamazulene	
Chamazulene	Cosmetics		—	4 g./batch		

1	2	3	4	5	6	7
16.	D.C. Recording polarograph	Polarographic analysis	—	1 unit/batch	Non-exclusive; P—Rs. 30,000 or P—Rs. 15,000 + R.—3%	Released to three parties.
17.	Dihydroisojasmone and Peach aldehyde chemicals	Perfumery	—	—	Non-exclusive; P—Rs. 5,000 as technical aid	Released to two parties; Both in production.
18.	N,N-Dimethylbiguanide-HCl (DMBG-HCl) Phenethylbiguanide-HCl(PEBG-CI)	Anti-diabetic drugs	Dicyandiamide, Dimethylamine Dicyandiamide, β -phynylethylamine	500 g./batch 140 g./batch	Non-exclusive; P—Rs. 7,500 R—2%	—
19.	Direct reading spectrophotometer/colorimeter	Biochemical research, spectroscopic analysis in visible range	—	1 unit/batch	Non-exclusive; P—Rs. 10,000 R—2%	Released to two parties
20.	Ethylene from ethyl alcohol	Organic intermediate	—	10 kg./hr.	Non-exclusive; P—Rs. 5,000 additional fee of Rs. 7,500 if designs and drawings are required.	Released to one party as technical aid to industry.

1	2	3	4	6	6	7
21. Ferrites—Hard	Electronics	—	—	1 kg./batch	Non-exclusive; P—Rs. 5,000 R—2 %	Released to three parties. Two in production.
22. Ferrites—Soft	Electronics	—	—	1 kg./batch	Non-exclusive; P—Rs. 5,000 R—2%	—
23. Flexible magnets	Refrigerator gaskets, toys, educational kits	Chlorinated polythene	—	500 g./batch extrusion of small pieces of 1-2 ft.length.	Non-exclusive; P—Rs. 5,000 R—2%	Released to one party.
24. Foundry core binder (sinol core binder)	Core binder in steel foundries for high dimensional accuracy	Catalyst	—	5 kg./batch	Non-exclusive; P—Rs. 5,000 R—2%	Released to one party.
25. Gaskets from coir pith	Gaskets	Neoprene	—	Gaskets of 18" × 18" × 1/8" size	Non-exclusive; P—Rs. 10,000	—
26. D-Glucosamine hydrochloride (C.P.)	Biochemical research, pharmaceuticals	—	—	250 g./batch	Non-exclusive; P—Rs. 10,000 R—2%	—

1	2	3	4	5	6	7
27.	Glyceryl guaiacolate	Drug-expectorant, intestinal antiseptic	Epichlor- hydrin, sodium nitrite	Laboratory scale	Non-exclusive; P—Rs. 25,000	—
28.	Gum arabic substitute	Adhesives	—	3 kg./batch	Non-exclusive; P—Rs. 5,000 R—2%	—
29.	Hexachlorethane (Ind. Pat. No. 92997)	Pyrotechnics, smoke screen, veterinary medicine, fluxing agent in foundries	—	10 kg./hr.	Non-exclusive; No premium Rs. 2½ %	Released to one party; In experimental production.
30.	β-Ionone (Ind. Pat. No. 77225)	Perfumery chemical, inter- mediate for Vitamin A	—	Laboratory scale	Non-exclusive; No premium R—2½ %	Released to two parties; One in production.
31.	Linseed oil emulsion paint (Ind. Pat. No. 117403)	Emulsion paints	—	8·5 kg./batch	Non-exclusive; P—Rs. 10,000 R—2%	—

1	2	3	4	5	6	7
32.	Microfilters	Industrial filtrations	Melamine	1 kg. of pulp per batch	Non-exclusive. P—Rs. 15,000 R—10 P. per cartridge.	—
33.	Monoethylaniline	Intermediate for explosives	—	15 kg./hr.	Non-exclusive; P—Rs. 25,000 R—2.5%	Released to one party.
34.	Nicotine sulphate (40% nicotine)	Insecticide	—	Plant designs for processing 1 T of tobacco waste	Non-exclusive. on zonal basis. P—Rs. 2000 R—1%	Released to four parties: One in production.
35.	Optical whitening agent for synthetic fibres	Whitening agent for synthetic fibres	Acenaphthene methylamine	1 kg./batch	Non exclusive; P—Rs. 30,000	—
36.	Civetone & Dihydro-Perfumery civetone		—	—	Non-exclusive, Negotiable	Know-how on Items 36-40 could be offered as an integrated project.
37.	Dihydroambrettolide & Isoambrettolide	„	—	—		

1	2	3	4	5	6	7
38. Exaltone	Pfumery	—	—	—	Non-exclusive Negotiable	See remark of item 36
39. Exaltolide (Ind. Pat. No. 73702)	”	—	—	—	”	”
40. Neo-Lavandulol	”	—	—	—	”	”
41. Phenylacetic acid	Perfumery, Penicillin G	Sodium cya- nide catalyst	1 kg./batch	—	Non exclusive; P—Rs. 10,000 R—1½%	Released to one party
42. Phenoxyacetic acid	Penicillin V	—	50 kg./batch	—	Non-exclusive, P—Rs. 10,000	—
43. Polyurethane coatings (Ind. Pat. No. 121538)	Leather, rubber wood, glass, Nylon fabrics	Toluene di-isocyanate, Adipic acid	7 kg./batch	—	Non-exclusive; P—Rs. 10,000 R—1%	—
44. Polyurethane printing rollers (Ind. Pat. No. 86991)	Printing rollers	Di-isocyanates, polyethylene glycols	15 lb./batch	—	Non-exclusive; P.—Rs. 5000 R.—5%	Released to 3 parties; One in production.

1	2	3	4	5	6	7
45.	Radiosonde thermistors	Meteorology	Platinum foil	100 pieces/batch	Non-exclusive; P—Rs. 25,000 R—5%	—
46.	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; P—Rs. 10,000.	—
47.	Rubber blowing agent (Dinitrosopentamethylene tetramine)	Rubber chemical	Sodium nitrite	10 kg./batch	Non-exclusive; P—Rs. 5,000 as technical aid	Released to one party; In production.
48.	Rubber reclaiming agent	Rubber chemical	—	20 kg./batch	Non-exclusive; P—Rs. 30,000	Released to one party; In production.
49.	Rubberized cork sheets from cork waste	Gaskets	Neoprene	Sheets of 6" × 9" × $\frac{1}{8}$ size	Non-exclusive; Premium Rs. 10,000	—

1	2	3	4	5	6	7
50. Sachets-Hot and Cold	Substitute for Hot water bag/Ice bag	—	—	20 cm. × 10 cm. size sachets	Non-exclusive; P—Rs.2,000 5 paise per sachet	Released to three parties; Two in production.
51. Sisal wax (Ind. Pat. Nos. 64958 & 65440).	Cosmetics, polishes	—	—	2 kg./batch	Non-exclusive; R-2½%	Released to four parties.
52. Staple pin adhesive	Adhehive for staple pins	—	—	10 kg./batch	Non-exclusive; P—Rs. 6,000 as technical aid	—
53. Tamarind kernel powder-phosphate and borate	Textile sizing, substitute for hydrolyzed maize starch	—	—	5 kg./batch	Non-exclusive; P—Rs. 5,000 R—2%	—
54. Terpeneol	Perfumery	α-Pinene	—	1 kg./batch	Non-exclusive; P—Rs. 35,000	Released to one party.
55. Thermistors	Temperature measurement and control, electronic devices, etc.	—	—	1 kg./batch	P—Rs. 10,000 as technical aid	Released to three parties; Two in production.

1	2	3	4	5	6	7
56.	Thioglycolic acid	Cosmetics, catalyst	Isopropyl ether	1 kg./batch	Non-exclusive; P—Rs. 25,000 or P—Rs. 10,000 + R—1%	—
57.	Xanthates-Potassium ethyl (PEX) and Potassium amyl (PAX)	Froth-flotation	—	500 g./batch	Non-exclusive; P—Rs. 40,000	—
B—TECHNOLOGIES WHERE CAPITAL REQUIREMENT RANGES BETWEEN Rs. 10—25 LAKHS						
58.	Anion exchange resin from melamine (Ind. Pat. No. 71190)	Demineralization of liquids	—	12 lb./batch	Non-exclusive; P—Rs. 2000 R—3%	Released to one party; In production.
59.	Atrazine	Herbicide	Monoisopropylamine	1 kg./batch	Non-exclusive; P—Rs. 3,500 R—2.5%	—
60.	Cashewnut shell gum (Ind. Pat. No. 123638)	Binder, thickening agent in food and pharmaceuticals	—	1 kg./batch	Non-exclusive; P—Rs. 10,000 R—2%	—

1	2	3	4	5	6	7
61. Dimethylaniline	Dyestuff and explosives intermediate		—	9 kg./batch	Non-exclusive; P—Rs. 1 lakh for 600 TPA	Released to one party; In production.
62. Ethylenedichloride from ethyl alcohol	Solvent, organic intermediate		—	3 kg./hr.	Non-exclusive; Negotiable	—
63. Monochloroacetic acid	Organic intermediate for weedicides, CMC, etc.		—	600 g./batch	Non-exclusive; P—Rs. 30,000	Released to one party; In experimental production.
64. Morpholine	Intermediate for rubber chemicals, textile chemicals, optical brighteners etc.	Diethanolamine		400 g./batch	Non-exclusive; P—Rs. 50,000	—
65. p-Nitrophenol	Intermediate for parathion and paracetamol	p-Nitrochlorobenzene		25 kg./batch	Non-exclusive; P—Rs. 40,000	Released to one party.
66. Phenacetin	Drugs	p-Phenetidine		60 kg./batch	Non-exclusive; P—Rs. 60,000	—

1	2	3	4	5	6	7
67. Potentiometric strip chart recorder	Instrument for use in research and industry	Chopper	One unit per batch	Non-exclusive; P—Rs. 10,000 R—Upto 1,000 pieces — 3% above 1,000 pieces — 2%	Released to three parties.	
68. Simazine	Herbicide	—	1 kg./batch of 50% simazine	Non-exclusive; P—Rs. 3,500 R—2.5%.	—	
69. Mannitol/sorbitol from cane sugar	Pharmaceuticals (Mannitol) Pharmaceutical syrups, Humectant (Sorbitol)	Catalyst (Raney Nickel)	5 kg./batch of sugar	Non-exclusive; P—Rs. 50,000	—	
70. 70% Sorbitol from dextrose monohydrate	Pharmaceuticals, Vit. C synthesis	Catalyst (Raney nickel)	75 kg./batch dextrose monohydrate.	Non-exclusive; P—Upto 500 T-Rs. 50,000. For larger capacities, on sliding scale upto Rs. 75,000/-.	Released to one party.	

1	2	3	4	5	6	7
71. Synthetic gemstones	Jewellery, electric meters		—	—	Non-exclusive; P—Rs. 30,000 R—1%	—
72. Theophylline Aminophylline and Caffeine	Drugs (Caffeine also used in beverages)	1, 3-Dimethyl-urea Raney nickel, Sodium nitrite, Sodium cyanide, Dimethyl sulphate, Ethylene-diamine		Theophylline, Caffeine— 1 kg./batch, Aminophylline 500 g./batch	Non-exclusive; P—Rs. 25,000 R—1½%	—
73. 2, 2, 4-Trimethyl-6-ethoxy-1, 2-dihydro-quinoline.	Rubber antioxidant	p-Phenetidine		1 kg./batch	Non-exclusive; P—Rs. 50,000.	—
74. 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) Chromatographic adsorbents & standardizing agents (b) Industrial organic, pharmaceutical intermediates.	Rs. 1,00,000 Rs. 60,000

1	2	3	4	5	6	7
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(c) Analytical reagents indicators & laboratory chemicals.

Rs. 90,000

C—TECHNOLOGIES WHERE CAPITAL REQUIREMENT EXCEEDS RS. 25 LAKHS

75. Acetanilide	Drugs and dye intermediate	—	60 kg./batch	Non-exclusive; negotiable.	Released to one party. In production. Turnkey plant available through Project Engineer.
76. Aniline	Organic intermediate	—	5 kg./hr. continuous	Non-exclusive; negotiable.	Released to one party.
77. Dissolving grade pulp (Ind. Pat. No. 82822)	Rayon, tyre cord	—	100 kg. of chip charge per batch	Non-exclusive; negotiable.	The process is offered on turn-key basis through Project Engineers.

1	2	3	4	5	6	7
78. Ethylenediamine	Bulk organic chemical		—	5-10 kg./day continuous	Non-exclusive; P—Rs. 50,000 R—1½%.	Released to one party. Turn-key plant available through Project Engineers.
79. Phthalate-butyl octyl	Plasticizer in non-electrical applications		—	45 kg./batch	Non-exclusive; P—0-1500 TPA Rs. 35,000 For larger capacities, on sliding scale upto Rs. 1 lakh.	—
80. Phthalates-dibutyl/dioctyl	Plasticizers		—	60 kg./batch (DBP) 70 kg./batch (DOP)	Non-exclusive; P—Upto 1500 T Rs. 35,000. For larger capacities, on sliding scale upto Rs. 2.5 lakhs.	Released to three parties; Two are in production.
81. Phthalates-dimethyl/diethyl	Plasticizers		—	60 kg./batch (DMP) 40 kg./batch (DEP).	Non-exclusive; P—Rs. 2 lakhs	Released to one party; In production

1	2	3	4	5	6	7
82. Vitamin B ₆ (Indian Patent No. 127750)	Drugs	Sodium cyanide, Phosphorus pent- oxide, 2-Butene 1, 4 diol, Paraformalde- hyde.	Laboratory scale (1 kg./ batch in progress)	Non-exclusive; P—Rs. 50,000; R— 1%.	—	
83. Vitamin C.	Drugs	—	—	Non-exclusive; R—1%.	Released to one party; In experimental production.	

COMPARATIVE COST-BENEFIT ANALYSIS FOR 1971-72 & 1972-73

	1971—72 <i>(Rs. in lakhs)</i>	1972—73 <i>(Rs. in lakhs)</i>
ACTUAL EXPENDITURE		
1. Recurring expenditure	70.41	69.52
2. Capital expenditure	12.42	7.87
3. Pilot plant expenditure	2.84	2.34
	85.67	79.73
BENEFITS		
<i>Receipts</i>		
Premia and royalty	1.42	1.07
Receipts on account of sponsored projects	8.67	4.53
Analytical/testing charges	0.32	0.30
Institutional consultancy (CSIR share)	0.38	0.36
FCP (Fine Chemicals Project) sales	2.67	2.40
Miscellaneous receipts including job work	1.97	5.99
	15.43	14.65
<i>Indirect benefits</i>		
1. Total number of processes in production	51	48 *
2. Value of production based on NCL know-how	471.20	557.11
3. Estimated savings in foreign exchange on account of above production	188.48	222.84
4. Total number of NCL processes released and awaiting production.		
(a) NCL processes.	20	36
(b) Sponsored schemes	79	86

5. Total number of parties who have taken up NCL processes for exploitation.	107	136
6. Total number of processes which were not released but which were available for commercial exploitation	56	51
7. Number of processes released		
(a) NCL processes	9	17
(b) Sponsored processes completed	12	7
8. No. of processes newly added to the list of NCL processes available for exploitation	21	4
9. Research papers published	111	66
10. Doctorate & Masters degrees awarded	20	22
11. No. of recognised guides for Doctorate & Masters degrees	38	39
12. <i>Patents</i>		
(a) Indian patents in force	52	58
(b) Foreign patents in force	7	7

Premia and Royalties received from NRDC through NCL processes

(a) Premia	1,03,500·00	3,79,249·00
(b) Royalties	64,388·00	97,290·00
No. of processes assigned to NRDC	29	15

*Parties who have not reported production for two consecutive years are excluded from this total.

Cumulative Cost-Benefit Analysis (1950-1973)

(Rs. in lakhs)

COST

1. Recurring expenditure	816·73
2. Capital expenditure	162·47 **
3. Pilot plant expenditure	71·47
	<hr/>
Total cumulative expenditure	1050·67

BENEFITS

1. <i>Total cumulative receipts</i>	
(a) Total premia	6.79
(b) Total royalty	3.39
(c) Receipts from sponsors	62.51
*(d) Miscellaneous	53.54
	<hr/>
	126.23
	<hr/>
2. Total cumulative value of production based on NCL know-how	1605.12
3. Total no. of research papers published	2,646
4. Total no. of degrees received	339

* This includes CSIR share of consultancy, analytical and testing charges FCP sales and other receipts.

** This figure does not include capital expenditure on NCL buildings amounting to Rs. 30.76 lakhs incurred by CSIR during 1949-50. Total investment Rs. 1081.43. lakhs.

NCL EXECUTIVE COUNCIL MEMBERS

1. Shri Arvind N. Mafatlal (Chairman)
Mafatlal House,
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2. Prof. B. K. Bachhawat,
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Sturdia Chemicals Ltd.,
Naval House, Ballard Estate,
Fort, Bombay-1.
4. Dr. T. R. Govindachari,
Director, CIBA Research Centre,
Goregaon, Bombay-63.
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Adviser (P.A.D.), Planning Commission,
Yojana Bhavan, New Delhi-1.
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I. I. T., Kanpur-16.
9. Shri Darbari S. Seth,
Director,
Tata Chemicals Ltd.,
Bombay House, Bruce Street,
Bombay-1.
10. Shri D. M. Trivedi,
Project Manager,
Synthetics & Chemicals Ltd.,
7, Jamshedji Tata Road,
Bombay-20.

11. Director General, Scientific & Industrial Research.
12. Financial Adviser, CSIR.
13. Director, NCL.

ADVISERS

Process Release Committee

1. Shri Arvind N. Mafatlal,
Mafatlal House,
Bombay-20.
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ARDE, Poona-21.
3. Shri V. G. Rajadhyaksha,
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Bombay-1.
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Director, Tata Chemicals Ltd.,
Bombay-1.
7. Shri D. M. Trivedi,
Project Manager,
Synthetics & Chemicals Ltd.,
Bombay-20.
8. Dr. H. E. Eduljee,
Sturdia Chemicals Ltd.,
Bombay-1.
9. Dr. C.V.S. Ratnam,
Managing Director, NRDC.
New Delhi-24.
10. Secretary, CSIR.
11. Director, NCL.

ADVISORY PANELS

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1. Dr. S. Ramachandran,
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Research, Bombay.
3. Dr. M. B. Sahasrabudhe,
Head, Chemotherapy Divn., Cancer Research Institute,
Bombay-12.
4. Prof. G. N. Ramachandran,
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Indian Institute of Science,
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Director, Virus Research Centre,
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Director, Hindustan Lever Research Centre,
Bombay-69.
7. Dr. P. K. Bhattacharyya,
Indian Institute of Science,
Bangalore-12.

II. *Chemical Engineering and Process Development*

1. Dr. C. J. Dadachanji,
Administrative Managing Director,
National Organic Chemical Industries Ltd.,
Bombay-18.
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Managing Director,
Aniline Dyestuffs & Pharmaceuticals Ltd.,
Bombay-1.
3. Prof. N. R. Kamath,
Indian Institute of Technology,
Bombay-76.

4. Dr. P. K. Mukhopadhyaya,
Engineers India Ltd., New Delhi-1.
5. Dr. H. E. Eduljee,
Sturdia Chemicals Ltd., Bombay-1.
6. Shri V. K. Dikshit,
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Kalyan.
7. Prof. M. M. Sharma,
Dept. of Chemical Tech.,
Bombay University, Bombay-19.
8. Shri J. J. Mehta,
Chairman & Managing Director,
Indian Petrochemicals Corpn. Ltd., Baroda,

III. *Inorganic Chemistry*

1. Dr. H. K. Chakravarty,
Head, Chemistry Division,
National Metallurgical Laboratory,
Jamshedpur-7.
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Min. of Petroleum & Chemicals,
New Delhi.
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Superintendent, (Chemistry Research Wing),
Fertilizer Corporation of India, Sindri.
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Director, Indian Institute of Science,
Bombay-32.
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Industrial Adviser (Chemicals), D.G.T.D.,
New Delhi.
6. Shri K. S. Hinge,
Chief Executive Officer (Development),
Tata Chemicals Ltd.,
Bombay-1.

7. Dr. Jagdish Shankar,
Chemistry Division,
Bhabha Atomic Research Centre,
Bombay-85.
8. Shri B. K. Kamat,
Production Manager (Works),
Dharamsi Morarji Chemical Co. Ltd.,
Ambernath (Dist. Thana).
9. Dr. H.V.R. Iyengar, (Jr)
Head of Research Dept.,
EID-Parry Ltd., Madras-1.

IV. *Organic Synthesis and Natural Products*

1. Prof. A. B. Kulkarni,
Dept. of Chemistry,
Bombay University,
Bombay-19.
2. Shri Jogindar Singh,
Industrial Adviser (Chem.), D.G.T.D.,
New Delhi.
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