ANNUAL REPORT 1973-74

NATIONAL CHEMICAL LABORATORY, POONA

NATIONAL CHEMICAL LABORATORY POONA

1973-1974

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH

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INTRODUCTION

This report describes the work in progress during the year April 1973—March 1974. As in last year's Annual Report, the research activities of the National Chemical Laboratory (NCL) have been grouped together under 19 major areas. The report highlights the major thrusts in each area and the benefits which may be expected, arising out of the R & D inputs. The year under review is also the last year of the Fourth Five Year Plan. R & D work on 31 projects (as against 13 last year) identified by the National Committee on Science & Technology (NCST) has been undertaken. This relates to bulk organic chemicals, petrochemicals, pesticides and agrochemicals, drugs and pharmaceuticals, etc.

Research Utilization

Details of the products manufactured by the NCL technology have been listed in Table I at the end of the report. The total number of processes based, on the NCL know-how in commercial production during the year 1973-74 were 49 with an annual turnover of Rs. 651 lakhs as against 48 processes with a turnover of Rs. 557 lakhs during 1972-73. Last two years have been difficult for the chemical industry which has been plagued with the shortage of raw materials. Hence the rate of growth in the value of annual production over the preceding year, was lower at 16.9% for 1973-74 as against 18.2% for 1972-73 and 66% for 1971-72.

During the year under review, production was reported for the first time for D. C. Recording polarograph, Direct Reading Spectrophotometer, thermosetting resins for industrial laminates and Carbazole Dioxazine Violet, pigment. Trial production of sorbitol and Vitamin C was reported by M/s Hindustan Antibiotics Ltd., Pimpri. M/s Synthetics & Chemicals Ltd., Bareilly, have established a commercial pilot plant of 1 T/day for medium grade nitrile rubber based on the NCL technology. The firm has reported a production of 30 T (Value Rs. 6 lakhs) during the year under review. The firm, in course of the next few years is expected to expand its capacity to 2000 TPA for all grades of nitrile rubber with a turnover of about Rs. 4 crores.

Table II lists 48 NCL processes (including sponsored) that have been released to 54 parties and are awaiting production. 13 processes were released to 16 parties during the year under review. The remaining 38 parties are also taking steps to implement 35 NCL technologies which have been transferred recently. To mention a few notable examples, a 3000 TPA (production value Rs. 1.5 crores) chloromethanes plant has been set up by M/s Standard Alkali and the commissioning runs are in progress. Hindustan Organic Chemicals

Ltd., Rasayani, completed the installation of 4500 TPA (production value Rs. 2.7 crores) Chlorobenzene plant during the year and the plant has recently gone in commercial production. A unit for monochloroacetic acid (300 TPA) was installed by M/s HICO Products (P) Ltd., Bombay, and trial runs were in progress. M/s Atul Products Ltd., Atul, have set up a plant for the manufacture of monoethylaniline (150 TPA). In addition to above, work was also in progress in the installation of the following plants based on the NCL know-how:- Opium Alkaloids (4.7 TPA; Value, Rs. 1 crore) by Ministry of Finance, Govt. of India; Oxalic acid (900 TPA) from bark of Ain tree by M/s Vidarbha Organic Chemical Industries Ltd., Nagpur, and *p*-toluidine (300 TPA plant) from *p*-nitrotoluene by M/s Sudarshan Chemical Industries Pvt. Ltd., Poona.

Table II (A) lists additional 10 new licencees who have not yet reported production for 9 processes (from Table 1) which are however being exploited by other parties.

From the last year's Annual Report a review of industrial utilization of NCL processes is being presented. Table II (B) thus gives data on processes developed by the NCL on its own. Till March 1974, the total number of processes developed was 115 (111) (figures in bracket are the corresponding data for the previous year) and the number of parties that had acquired the NCL know-how was 88 (75). The total number of processes released was 71 (60) and those in production was 32 (29). The value of production for the year 1973-74 was Rs. 388 lakhs (Rs. 401 lakhs).

Sponsored Research— Table II (C) gives a review of the utilization of the processes developed by the NCL on sponsorship by industry. Till the end of March 1974, the total number of sponsored schemes investigated was 55 (51). Of these 16 (15) led to commercial production; the value of production being Rs. 261 lakhs (Rs. 154 lakhs).

Five sponsored schemes were concluded during the year, 6 schemes newly undertaken and 10 schemes continued from 1972-73.

Know-how Available

Table III lists 87 processes on which the NCL know-how is available for commercial exploitation to interested parties. During the year 15 newly developed processes were cleared by the NCL Process Release Committee for release to industry.

Technology Transfer during 1973-74

Eight processes were released to industry as against 17 last year. The process know-how for 9 processes was demonstrated to the licencees.

Premia and Royalties

The total amounts of premia and royalties received during 1973-74 by the NRDC on account of the NCL processes were respectively Rs. 2.39 lakhs

(19 processes) and Rs. 0.74 lakhs (16 processes) as against premia of Rs. 3.79 lakhs (29 processes) and royalties of Rs. 0.97 lakhs (14 processes) received during last year.

Product Oriented Research

The Report describes work carried out on 98 product oriented R & D projects. The number of such projects in each area were as follows:—Petrochemicals and Bulk Organic Chemicals, 5, Pesticides and Agrochemicals, 15; Drugs, Pharmaceuticals and Fine Chemicals, 6; Organic Intermediates Dyes and Industrial Chemicals, 11; Utilization of Plant and Forest Resources, 12; Chemistry of biologically Active Compounds, 4; Studies in organic Synthesis 2; Industrial Polymers, Resins and Elastomers, 10; Inorganic chemicals and Catalysts, 8, Utilization of Mineral Resources, 5; Organometallic compounds, 1; Solid State materials, 7; Instruments Technology, 4; Enzyme Chemistry & Technology, 1; Fermentation Processes, 4; and Process Development and Chemical Engineering Studies, 3. The main thrust of reserach work was in the areas of petrochemicals and bulk organic chemicals (4 projects) and agrochemicals (11 projects) where most of the work undertaken was on important projects identified by the NCST.

Basic Research and Publications

Work on basic research projects has been highlighted under the relevant areas. The total number of research papers published was 88 of which 17 were on solid state materials, 12 on physicochemical studies, 11 on natural organic products, 18 on synthetic organic chemistry, 2 on enzyme chemistry and technology, 7 on polymers, resins, elastomers and synthetic fibres, 11 on inorganic chemistry, 4 on process development and chemical engineering studies and 6 on planning, management and publicity. In addition 12 papers were published in collaboration with outside scientists and 20 papers were presented at symposia, seminars, etc.

40 NCL scientists are recognised by different Indian Universities as research guides. 11 NCL staff members, research fellows and guest workers received post graduate (Ph.D.) degrees during the year.

Patents

As on 31st March 1974, 51 Indian patents (27 sealed and 24 filed) and 6 foreign NCL patents were in force. Three new Indian patents were filed during the year.

Consultancy

During the year, consultancy was offered to 3 public sector (HOC Ltd., HAL and IPCL) and 8 private sector industries through individual scientists or group of scientists. 14 NCL scientists acted as consultants. The consultancy fees received during the year (CSIR share) were Rs. 0.23 lakhs.

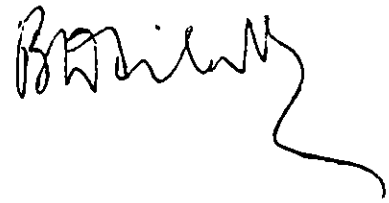
Executive Committee and Advisers

As per the recommendations of the CSIR and Sarkar Committee the Executive Council was replaced by an Excutive Committee which had a majority of internal members. The NCL Process Release Committee was also reconstituted with 7 outside and 5 internal members.

As in previous years the laboratory benefited from advice rendered by 6 Advisory Panels, on Biochemistry, Chemical Engineering and Process Development, Inorganic Chemistry, Organic Synthesis and Natural Products, Polymer Chemistry and Solid State and Physical Chemistry.

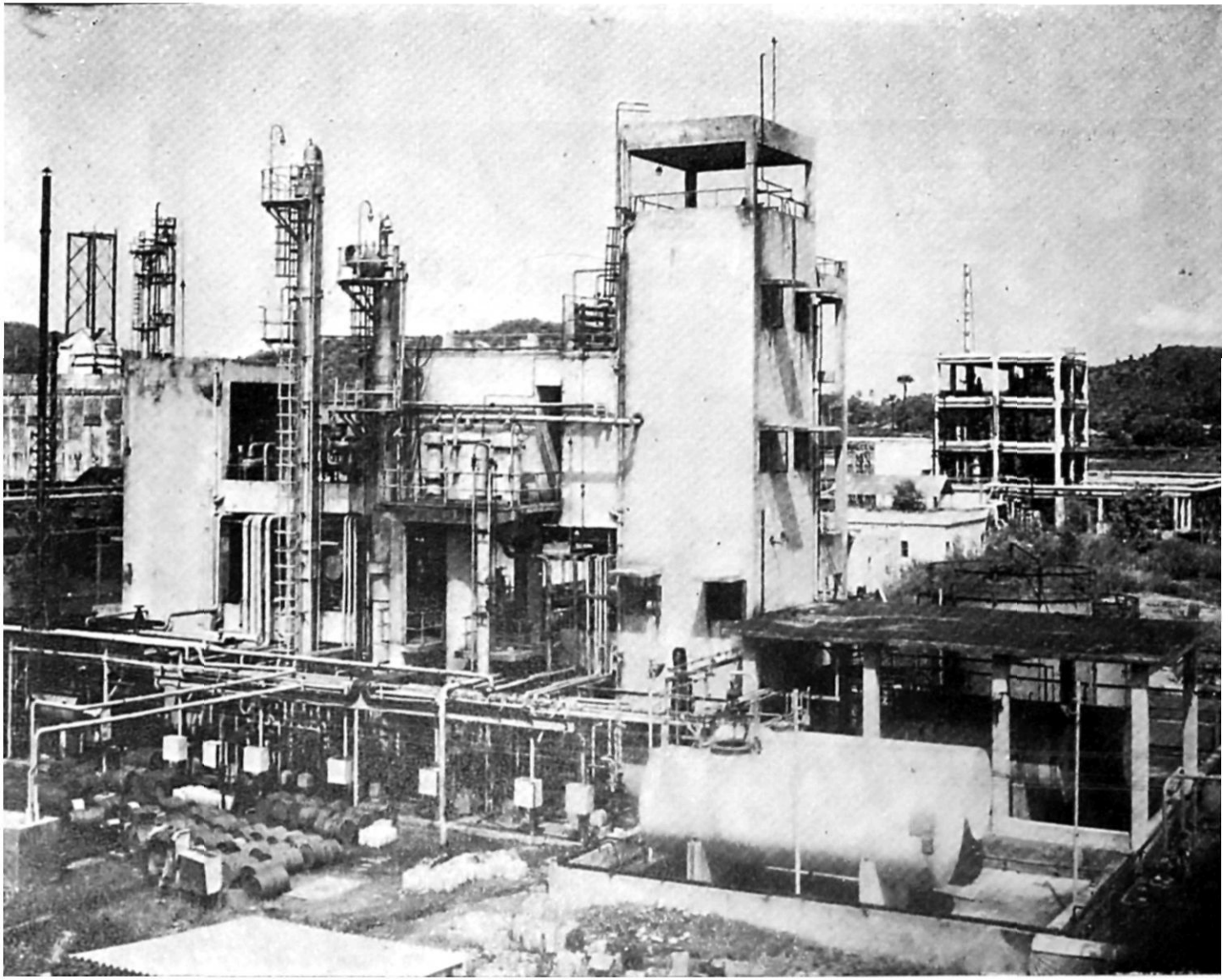
Cost-Benefit Analysis

For the year under review, as against an expenditure of Rs. 90.83 lakhs (Rs. 79.51 lakhs recurring and Rs. 11.32 lakhs capital), the total receipts were Rs. 11.50 lakhs (Rs. 0.58 lakhs NCL's share of premia and royalty, Rs. 4.21 lakhs on sponsored projects, Rs. 0.22 lakhs on analytical and testing charges, Rs. 0.23 lakhs as CSIR share on institutional consultancy, Rs. 1·74 lakhs on Fine Chemicals Sales and Rs. 4.52 lakhs on miscellaneous account). Details of the Costs and direct and indirect benefits have been given on page 130. The value of productions based on NCL know-how for 1973-74 was Rs. 651 lakhs. Most of these production led to import substitution. The value of foreign exchange thus saved is estimated at Rs. 260 lakhs.

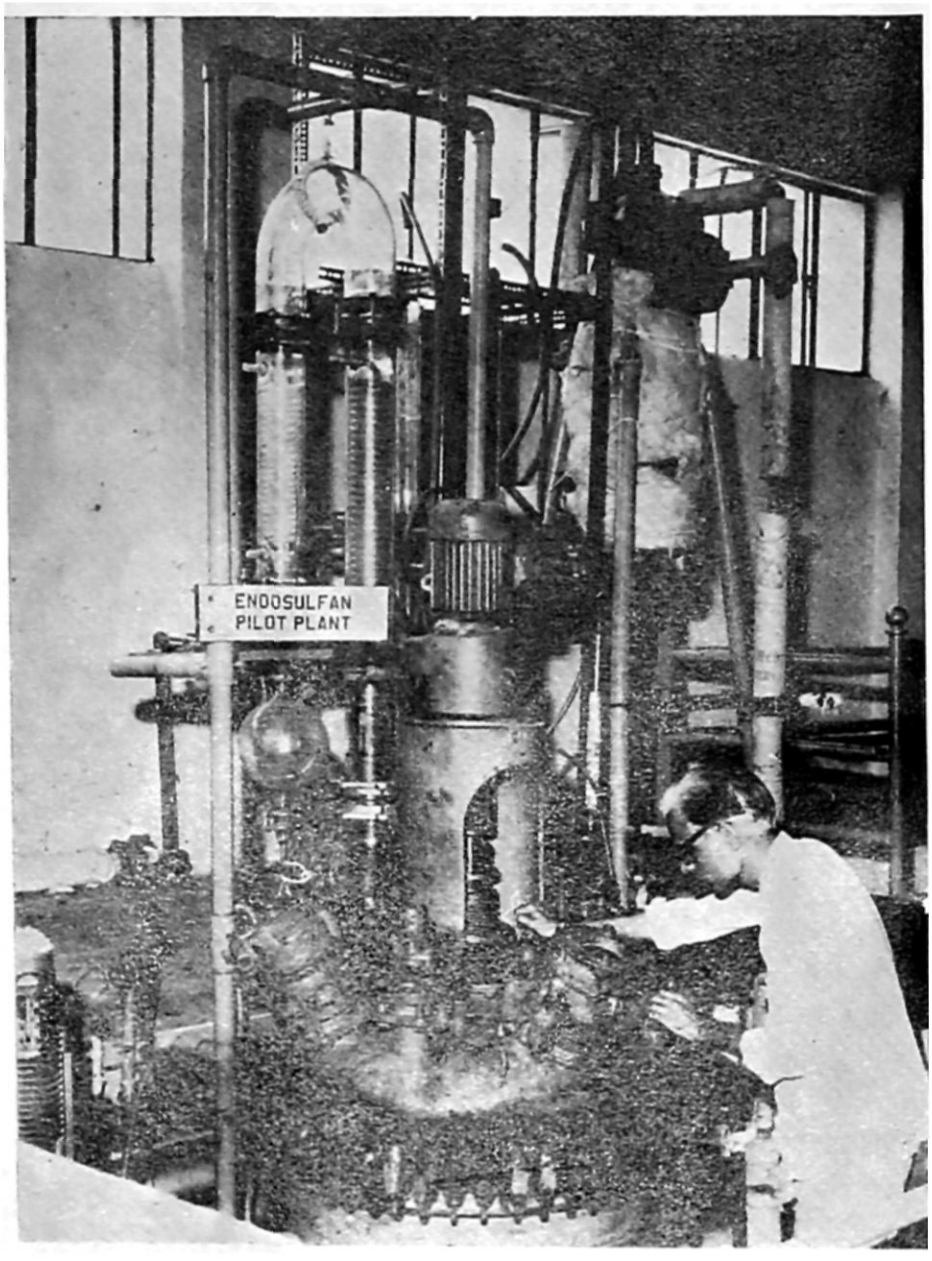


November, 1974.

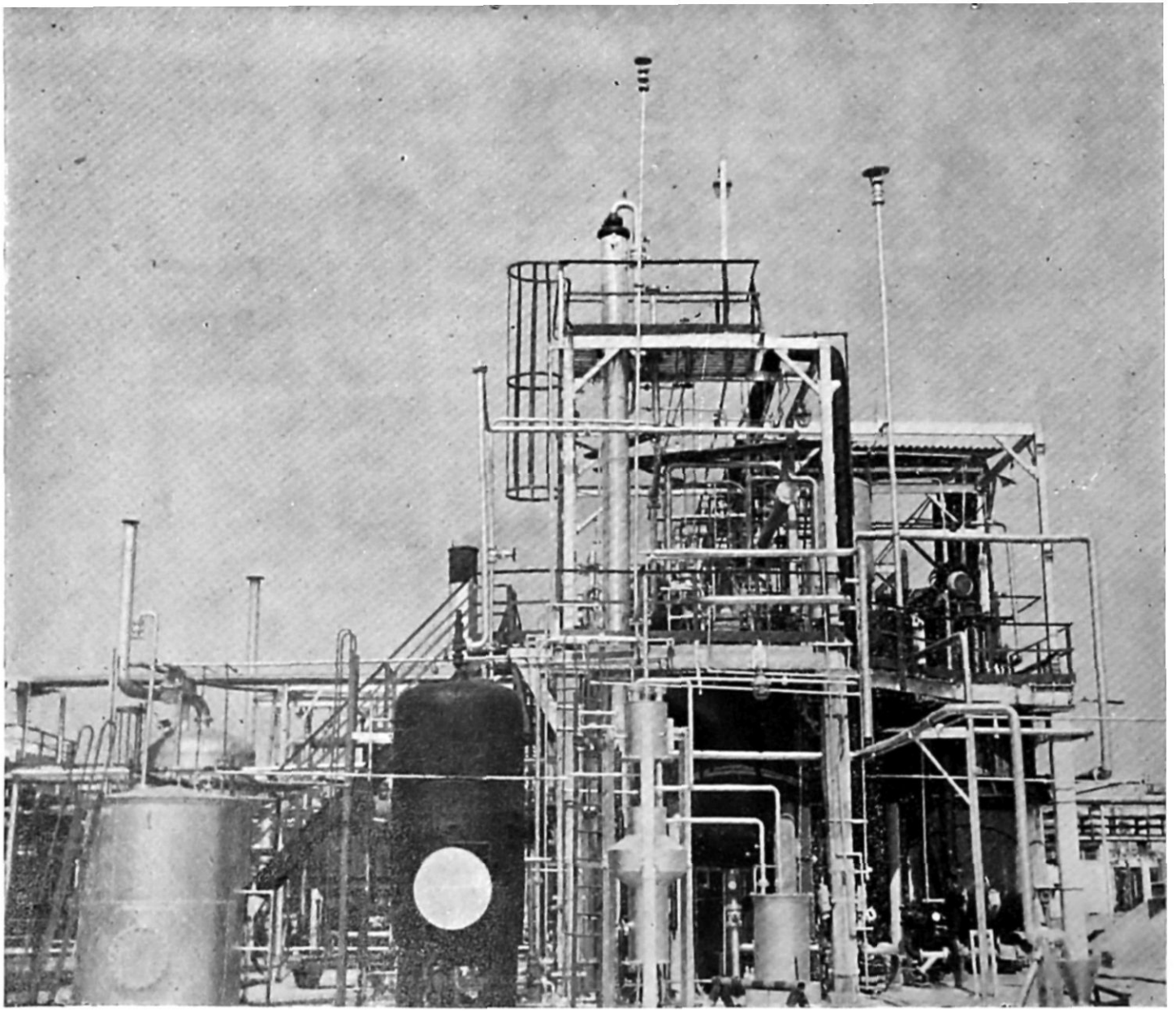
B. D. Tilak
Director



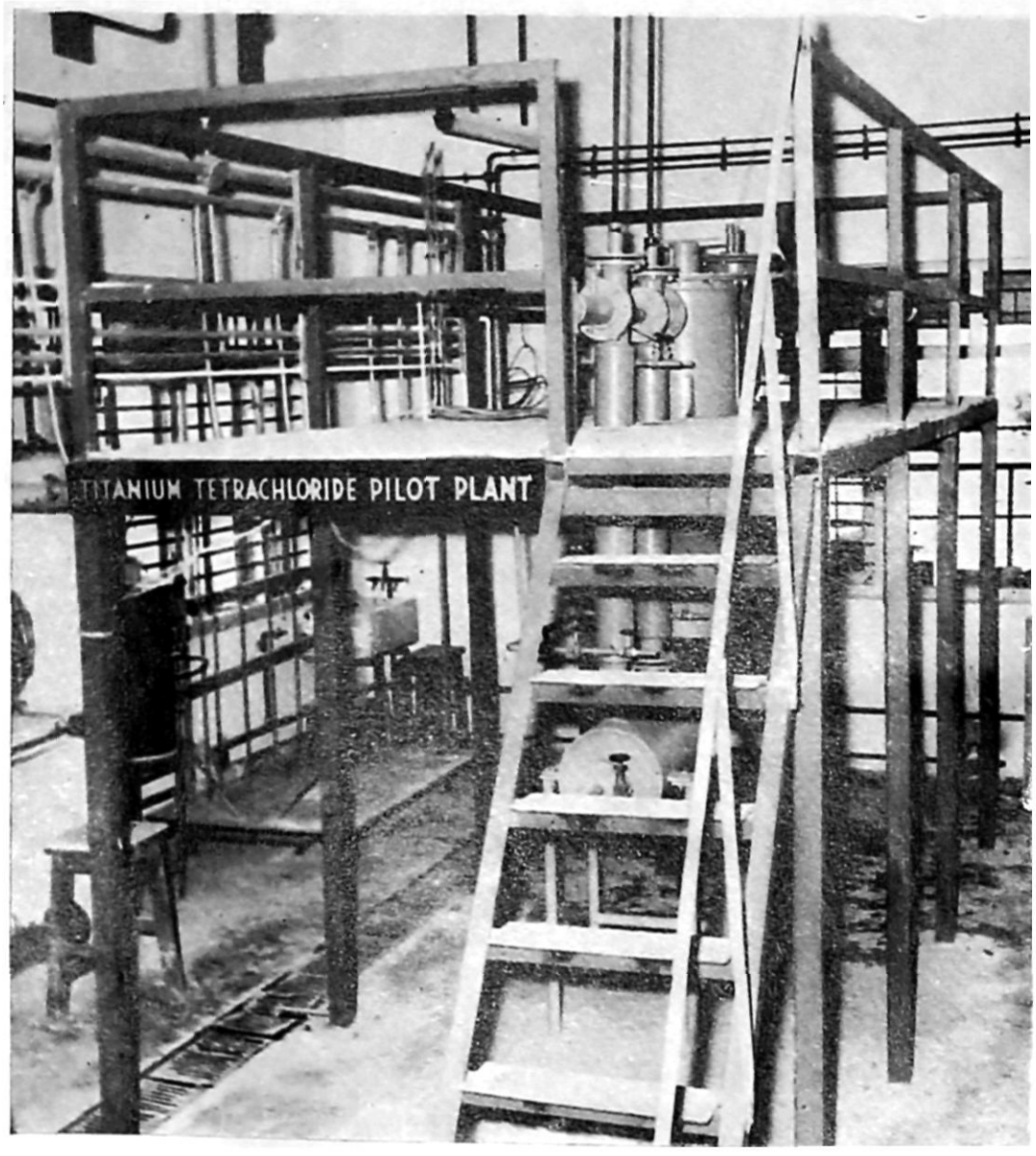
Chlorobenzene Plant (4500 TPA capacity) at Rasayani



Endosulfan Pilot Plant at NCL



Nitrile Rubber Pilot Plant at Bareilly



Titanium Tetrachloride Pilot Plant at NCL

RESEARCH AND DEVELOPMENT PROJECTS

1. PETROCHEMICALS AND BULK ORGANIC CHEMICALS

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

Propylene oxide is not being produced indigenously. The demand for a number of products based on it is met through imports. This sponsored project is aimed at developing the know-how for propylene oxide from propylene that will be available with the sponsor.

After the bench-scale experimentation, the process conditions were standardized on the specially installed pilot plant with an output of 2 kg. of propylene oxide per hour. All the relevant data needed for engineering a plant for propylene oxide has been supplied to the sponsor.

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

This sponsored work was undertaken with a view to utilizing the acrylonitrile that would be available in surplus quantities with the sponsor. Acrylic esters find use in polymer, paint, paper, textile, leather and various other allied industries. The demand is estimated at around 5000-6000 TPA, valued at more than Rs. 5-6 crores. This is likely to increase further in the near future.

The laboratory-scale work on the preparation of glacial acrylic acid, butyl acrylate and 2-ethylhexyl acrylate was completed earlier. A process for methyl acrylate in a counter current extraction cum distillation column was also worked out.

During the year under reference, kinetic data for the preparation of acrylamide sulphate and methyl acrylate was experimentally obtained and finally a method for obtaining pure methyl ester was worked out on a pilot plant scale.

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

Oxides of ethylene and propylene are required in large quantities in the manufacture of glycols, glycol ethers, polyglycols, amino alcohol detergents, surfactants, stabilizers and rocket propellants. This sponsored scheme is undertaken to develop a catalyst suitable for the vapour phase oxidation of ethylene. Such a catalyst is not indigenously available at present.

Various silver oxide catalysts containing different promoters such as Ba, Ca, Sr, Mg and CO_2 inhibitors, supported on silicon carbide were pre-

pared and tested for their activity and selectivity for the oxidation of ethylene to ethylene oxide. Effect of γ-irradiation on the selectivity of the catalyst was also studied.

Work on improving the selectivity of the catalyst and the development of a similar catalyst for propylene oxide is under way.

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

This glycol, mainly used in the manufacture of various polyesters, is not being produced in the country.

The manufacturing process for this glycol consists of aldolization of acetaldehyde and its subsequent hydrogenation. After standardizing the conditions for the aldolization on a bench scale, comparable results were obtained in a continous reactor. The hydrogenation conditions were established in a Parr autoclave to give a 30 APHA (plasticizer grade) 1, 3-butylene glycol. Life test of the catalyst was also carried out. The project is concluded.

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

As reported earlier the benzoic acid route for the manufacture of this industrially important basic chemical was attempted using (a) a stirred tank reactor, as also (b) a tray-type reactor for disproportionation of potassium benzoate. Results in both the cases were not encouraging and the conversions were of the order of only 50% and 55% respectively. Conversion to the extent of 65%, however, was obtained when pellets of potassium benzoate mixed with catalysts were heated in a tower-type reactor. In view of other priority projects, work on this project has been suspended for the time being.

2. PESTICIDES AND AGROCHEMICALS

2.1 *Phenthoate and Thiometon :* (SP-89/73)

Phenthoate is an excellent broad spectrum insecticide with low mammalian toxicity. The estimated requirement of phenthoate by 1978-79 is 50TPA valued at Rs. 50 lakhs as formulated product. A convenient route for the preparation of phenthoate (1kg/batch) was standardized in the laboratory. Based on the process standardized, the sponsors carried out larger scale pilot plant batches at their factory and are now erecting a plant for 300 TPA which is scheduled to go on stream by early 1975.

The estimated demand for the pesticide thiometon in 1978-79 is 100 TPA valued at Rs. 100 lakhs as formulated product. Under this sponsored project work on the preparation of the key intermediate chloroethyl-ethyl-sulphite was standardized on a laboratory scale. Large scale trials on this intermediate and its conversion to thiometon are in progress.

2.2. *Pentachloronitrobenzene (PCNB)* : (ATT-167/70)

PCNB is a soil fungicide for cotton and a variety of vegetables. The

demand for this fungicide is estimated at about 100 TPA valued at Rs. 29 lakhs. PCNB is not manufactured in India.

A process for the manufacture of this fungicide was developed on 20 kg/batch scale and is ready for release to industry.

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

Menazon, a systemic insecticide, is very effective for control of aphids on tobacco, mustard and other similar plants. The projected demand for this product in 1978-79 is 50 TPA with a sale value of Rs. 70 lakhs as formulated product. Menazon is not manufactured in India at present.

A process for its manufacture has been standardized and will soon be released to industry.

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

The sodium salt of dichloropropionic acid is used as a selective weedicide, mainly for the control of grasses in sugar cane, sugar beet, maize, potato and other similar crops. Its action resembles that of 2, 4-D.

A technically feasible process has been worked out.

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

Nitrofen is a modern weedicide especially useful for sorghum, sugar cane, ground-nut etc. The 1978-79 demand is estimated to be nearly 250 tonnes valued at Rs. 75 lakhs of the formulated product.

Process for the preparation of nitrofen has been standardized and is now available for release to industry.

2.6 *Endosulfan* : (ATT-207/73)

Endosulfan (Thiodan) is an excellent broad spectrum contact insecticide with low toxicity to vertebrates. It is effective for the control of aphids, thrips, beetles, foliar feeding larvae, mites, borers, cutworms, bugs, white flies, leaf hoppers and slugs on deciduous, citrus and small fruits as well as vegetables and forage. The consumption of endosulfan in 1972-73 is estimated at 750 tonnes of technical material valued at Rs. 6.9 crores of formulated products and the estimated demand by 1978-79 is 1800 TPA valued at aproximately Rs. 16 crores.

A two-step process starting from hexachlorocyclopentadiene and *cis*-2-butene-1,4-diol was optimized. This project was taken up as a top priority project with a team of scientists from different disciplines and was successfully completed in record time of one year on pilot plant scale.

After successful completion of the study on the kinetics of the two reactions involved in the process, and of the physical properties of the che-

micals under process conditions, pilot plants of 25 kg/batch and 100 kg/batch respectively were designed, erected and operated to test the validity of the process conditions established in the bench scale unit, and to collect engineering data required for the scale up of the process.

The product developed at the NCL was tested for its insecticidal activity and oral toxicity and was found to be comparable to imported product from Israel and West Germany. Studies on choice of the most suitable stabilizer for the finished product are in progress.

Based on the process and engineering data collected on bench and pilot units, complete process design for a 1600 TPA commercial plant was prepared. The process is being offered to industry as a turn-key bid through a firm of project engineers with full guarantees of performance.

2.7 *Vitavax* : (ATT-208/73)

Vitavax, a modern fungicide, is exceptionally active, highly specific and selective against pathogens without injury to hosts and has practically no toxicity to mammals. The demand for Vitavax by 1978-79 is estimated at 100 TPA valued at Rs. 100 lakhs.

Choroacetoacetanilide is one of the intermediates required for the synthesis of vitavax. A process for the chlorination of acetoacetanilide was standardized. Further work is in progress.

2.8 *Ethrel* (*2-chloroethylphosphoric acid*) : (ATT-210/73)

Ethrel acts as a plant growth regulator by virtue of its ability to release ethylene. Its application is found to give about 100% increase in the yield of latex (rubber). It is also used successfully for many other crops. The estimated demand of ethrel by 1978-79 is around 100 TPA valued at Rs. 150 lakhs.

Technical preparation of ethrel is being standardized.

2.9 *Tetradifon* (*Tedion*) : (ATT-213/73)

Tetradifon is widely used as an acaricide which is active on all stages of mites. It is used on fruits, nuts, cotton, tea, vegetables and various other crops. It is harmless to foliage and does not kill beneficial insects. The requirement of Tetradifon in 1978-79 is estimated at 30 TPA valued at Rs. 76 lakhs of the formulated product. Tetradifon is not manufactured in India at present and the requirement is met through imports.

A technical method for the preparation of tetradifon has been established and the process is being released to industry.

2.10 *Carbofuran* (*Furadan*) : (ATT-222/73)

Carbofuran is an excellent insecticide for paddy with effective action against the whole range of paddy insects in India. It has also systemic action.

Laboratory scale work for the synthesis of carbofuran was initiated starting from *o*-nitrophenol and catechol. As model compounds, allyl ethers were made and the Claisen rearrangement of the allyl ethers studied.

At the instance of the Coordination Committee, further work on this pesticide will now be carried out at the Regional Research Laboratory, Hyderabad.

2.11 *Phorate* (*Thimet*) : (ATT-223/73)

The preparation of diethyldithiophosphoric acid—an intermediate for the synthesis of phorate has been standardized. Exploratory experiments for the synthesis of phorate were carried out.

2.12 *Insect hormones and pheromones* : (AB-105/72)

Compounds having mimic insect hormone and insect pheromone activity are likely to prove useful in the selective control of certain harmful insect pests. Moreover such compounds are nontoxic to mammals.

(a) Attempts are under way to synthesize compounds of the above type from intermediate products obtained from plant sources. Starting from geraniol, a naturally occurring terpene, a number of compounds were prepared with various functionalities in the molecule like *cis*- and *trans*-double bonds, ester, alcohol, acetate and epoxide groupings for possible use as juvenile hormone mimics. Preliminary screening tests on nine of these compounds using red cotton bug (*Dysdercus Koenigii*) as the test insect and farnesyl methyl ether, a known J. H. mimic, as the control compound, carried out at B.A.R.C., Bombay, showed that five of them were more active and the others less active than the control. Larger amount of the active compounds were prepared and were sent for detailed investigations. Some additional modified analogues of these compounds are under preparation.

Synthesis of new and potent products, having structural features similar to those present in the juvenile hormone, has been undertaken.

2.13 *Slow-releasing herbicides* : (AB-127/72)

The concept of slow-releasing pesticides is new. It aims at the most economical way of using them with minimum pollution.

Two slow-acting herbicides were prepared by chemically reacting 2,4-dichlorophenoxyacetic acid with saw dust and bagasse. The optimum conditions for the maximum incorporation of 2, 4-D were determined. The rate of release of 2, 4-D from the product under soil conditions was determined by mass spectral techniques. The product was evaluated for its efficacy by field-trials on sugarcane and wheat by the Nimbkar Agricultural Research Institute, Phaltan. In presence of urea the product was found to be more efficient than 2, 4-D as a herbicide.

2.14 *Arsenic pesticides* : (AB-110/73)

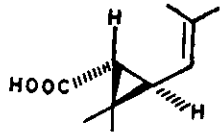
Disodium methyl arsonate (DSMA) and monosodium methyl arsonate (MSMA) are reported to be imported as agrochemicals. These compounds are used as weedicides.

Preparation of DSMA was carried out on laboratory scale starting with arsenious oxide. Methyl arsonic acid was also prepared on small scale from DSMA. Methyl arsonic acid is the starting material for MSMA.

Efforts to collect technoeconomic data on arsenic agrochemicals revealed that these are not much used in agriculture. The project has therefore been discontinued.

2.15 *Pyrethrins* : (AB-123/73)

(+)—trans-chrysanthemic acid is the essential component of pyrethrin and its analogues which are well known for their high insecticidal activity with low mammalian toxicity. A new efficient synthesis of (+)—trans-chrysanthemic acid was achieved starting from (+)Δ^3-carene.



2.16 *Determination of pesticide residues in food, air and water* : (AB-128/73)

The mass spectral technique developed for the estimation of lindane and carbaryl residues in straw, rice and bran was used to analyse a few experimental field samples received from the Rice Research Institute, Cuttack. The residues were found to be below the residue levels prescribed by W.H.O./ F.A.O.

3. DRUGS, PHARMACEUTICALS AND FINE CHEMICALS

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

Glyceryl- ∝-mono-para-aminobenzoate is used in ,cosmetic industry. Work on this sponsored project was completed and the process demonstrated to the sponsor.

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

During the year under review, six new compounds were sent for testing to the sponsor.

3.3 *Papaverine hydrochloride (PPH)*: (SP-90/73)

Papaverine hydrochloride is used in drug industry mainly as an antispasmodic. It is also used along with sedaline as a hypnotic. Imports of this drug during 1971-72 were 4 tonnes valued at Rs. 7.25 lakhs.

Under the sponsored scheme a process for the preparation of the drug from veratrole was developed on a laboratory scale. The product obtained compared favourably with the imported sample. Further standardization of the process is in progress.

3.4 *Cephalexin and 7-aminodeacetoxycephalosporanic acid (7-ADCA)*: (SP-91/73)

Preliminary experiments on the conversion of penicillin V to the corresponding cephalosporin derivative, by oxidation of penicillin V to sulfoxide and ring-expansion rearrangement were carried out. With the object of carrying out the same sequence of reaction on ampicillin, efforts were made to protect the amino and carboxyl function. Reactions were designed to achieve this in one step. The protected ampicillin would be useful for direct conversion to cephalexin, a known cephalosporin antibiotic.

At the request of the sponsor efforts are now concentrated on the synthesis of carbenicillin and hence work on cephalosporin is temporarily suspended.

3.5 1,4-*Benzodiazepines (Diazepam and Chlordiazepoxide)* : (ATT-218/73)

The benzodiazepines are a class of compounds which have been found to show marked antianxiety effect in human beings. Clinical studies indicated that various members of this series also have anticonvulsant, muscle relaxant and hypnotic properties. At present all the drugs of this category are imported to the tune of Rs. 50 lakhs. The main intermediates required for these drugs are 2-amino-5-chlorobenzophenone and 2-methylamino-5-chlorobenzophenone. A two step process for its manufacture was optimized on a laboratory scale. The know-how is being released to industry.

Starting from 2-methylamino-5-chlorobenzophenone a two step process for the manufacture of diazepam is being optimized.

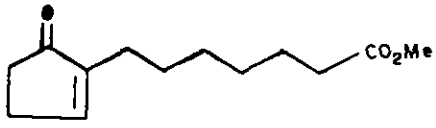
Work on the process development of chlordiazepoxide is in progress.

3.6 *Prostaglandins* : (AB-106/72)

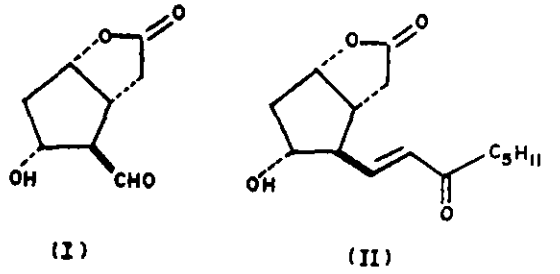
The prostaglandins are a family of oxygenated C_{20}-fatty acids exhibiting a broad range of biological activities, the chief among them being the muscle contracting activity, which has enabled their use as abortive agents for post-conceptive control in family planning. In addition, they are used to

lower blood pressure, prevent blood clots, treat asthmatics and act as long-lasting nasal decongestants.

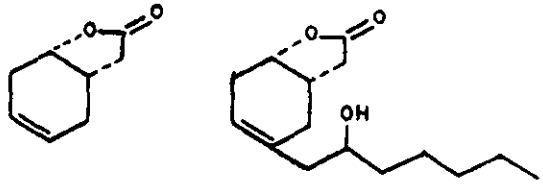
A new simple three step synthesis of the Bagli prostanoid synthon (I) was achieved starting from a readily available fatty acid. There exists precedence in the literature for elaboration of this key intermediate into 11-deoxyprostaglandins as well as the natural (—)-PGE_1.



For the synthesis of Corey's Lactone such as (I) and (II) the following compounds viz. (III) and (IV) were successfully synthesized. Cleavage of the double bond of (III) followed by cyclization of the dialdehyde furnished the desired hydroxy lactone (I) as an epimeric mixture. Work is in progress for the separation of the epimers. For the synthesis of (II) the lactone (IV) was oxidized and the ketone was protected as ethylene ketal. Oxidative cleavage followed by cyclization is in progress.



(I) (II)



(III) (IV)

For getting the lactone (III) by another route the reaction between 1,4-cyclohexadiene and dichloroketene was also attempted. This reaction is not found encouraging as many side-products are formed.

4. ORGANIC INTERMEDIATES, DYES & INDUSTRIAL CHEMICALS

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

Successful preparation of blue and violet red cationic dyes was reported earlier. During the period under report, laboratory processes for the pre-

paration of eleven dyes ranging in shade from yellow to violet were standardized. The samples are being evaluated by the sponsor.

A modified process for the preparation of a valuable intermediate required for these cationic dyes was also developed. A patent application on the process was filed.

4.2 β-*Naphthol* : (SP-92/73)

Although produced in the country, this valuable dyestuff intermediate is still imported to the tune of 2500 TPA valued at Rs. 100 lakhs.

Under this sponsored project, a laboratory process for the sulphonation of naphthalene and isolation of pure sodium naphthalene sulphonate was optimized. A technically feasible process for the alkali fusion of the sodium naphthalene sulphonate to yield β-naphthol is being standardized.

4.3 *p-Cresol* : (SP-93/73)

p-Cresol is valuable as a dyestuff intermediate and is also used for the preparation of the important antioxidant, ter-butylated hydroxy toluene (BHT).

A bench scale process for the preparation of *p*-cresol from sodium *p*-toluene sulphonate was optimized.

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

Under this programme, a range of azo and anthraquinone dyes containing arylazido, azidoacetamido, carboxyazido and sulphonazido groups were prepared. These dyes and their blends as reactive dyes were applied to a variety of synthetic and natural fibres. The chemistry of the reaction of the dyes with the fibres is being studied. New sulphur and selenium heterocyclic coloured compounds derived from anthraquinone were prepared. The chemistry of these compounds is being studied. A number of aza analogues of phthaloylpyrrocoline were prepared. The pigmentary and dyeing properties of these new compounds are being studied.

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

Trioxane, a key intermediate for the preparation of polyacetal resins, is not manufactured in the country. A convenient method for obtaining pure trioxane has been developed.

4.6 *Chlorinated paraffin wax (CPW)* : (ATT-209/72)

Chlorinated paraffin wax a cheap plasticizer extender, is mainly used for flexible PVC along with dioctyl phthalate. It may also find applications in fire, mould and water proofing of canvas, etc.

A laboratory method (5 kg./batch) was standardized, for chlorinated paraffin wax (CPW) containing 42% chlorine. The product was comparable

to imported commercial samples. The work on the preparation of CPW containing higher percentage of chlorine was discontinued and the project concluded. The process is ready for release to industry.

4.7 *Hexachlorocyclopentadiene* : (ATT-211/73)

Hexachlorocyclopentadiene (HCCP) is an important raw material for the manufacture of several chlorinated insecticides like endosulfan, chlordane heptachlor, aldrin and dieldrin.

Work was underaken to develop a process for HCCP by chlorination of cyclopentadiene. Cyclopentadiene can be obtained from dicyclopentadiene likely to be available from C_5 olefin streams of Indian naphtha cracker units. Work on optimization of the process is in progress.

4.8 *Pentaerythritol (PE)* : (ATT-214/73)

Pentaerythritol is used for the manufacture of resins and the explosive PETN. Imports of this chemical during 1970-71 were 1750 tonnes valued at Rs. 70 lakhs. A process for the preparation of pentaerythritol was optimized. The product containing 98% mono PE was obtained in good yields.

4.9 4-*Nitro*-3-*methylphenol* : (ATT-225/74)

4-Nitro-3-methylphenol, a key intermediate for the manufacture of the pesticide fenitrothion, is at present imported. Estimated demand of fenitrothion in 1978-79 is 700 TPA valued at Rs. 4.9 crores as formulated material.

4-Nitro-3-methylphenol was prepared in satisfactory yields from *m*-cresol. Scale-up work is progress.

4.10 *o,c-Dialkylthiophosphoryl chloride* : (ATT-226/74)

o,o-Dimethyl and diethyl-thiophosphoryl chloride are intermediates in the preparation of pesticides such as fenitrothion and metasystox. Starting with thiophosphoryl chloride and methanol a process was worked out which gave a 90% pure product in good yields. An alternative route using phosphorus pentasulphide, methanol and chlorine is also being explored.

4.11 *Thiodiphenol (4'-4'-dihydrodiphenyl sulfide)* : (ATT-227/74)

Thiodiphenol is the main intermediate for Abate which is a highly effective larvicide for mosquito and midge larvae.

Initial trials with Abate have proved highly successful for malaria control. Abate is proposed to be manufactured in the country form imported thiodiphenol. Starting from phenol a laboratory scale process was successfully developed for the preparation of thiodiphenol. Further work is in progress.

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

A. *Cationic dyes*: Several cationic dyes were crystallized as iodides

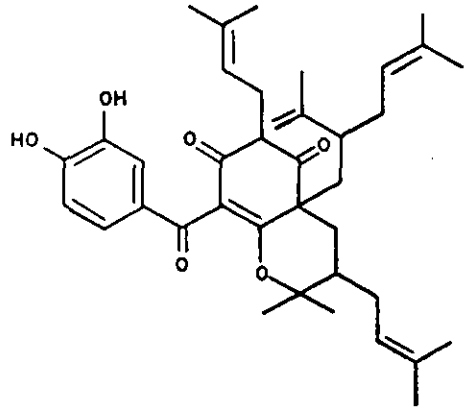
(from water) and picrates (from benzene). The dyes were subjected to sodium borohydride reduction, and the NMR and mass spectra of the products were examined. The data was used for determining the structures of three commercial cationic dyes.

B. *Reduction of aryldiazonium salts to arenes*: In connection with structural problems, this useful reaction was studied. The method mentioned briefly and in general terms in two old German patents, involving the action of dimethylformamide, proved to be most effective. Evidence was obtained for a hydride transfer mechanism. Thus aniline-2,5-disulphonic acid gave a nearly quantitative yield of benzene-l, 4-disulphonic acid. Among several other reagents examined, tetramethylurea proved to be of interest, because the arene is accompanied by acetaldehyde and propionaldehyde suggesting the operation of both ionic and radical mechanisms.

5. UTILIZATION OF PLANT AND FOREST RESOURCES

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

Garcinia species : Xanthochymol and isoxanthochymol, isolated from *G. xanthochymus* fruits are represented by the structures (I) and (II). In the light of their structures the structure of bronianone (Ollis et al., 1969), occurring in the wood of *G. hombroniana*, was revised. Three new xanthones, one of which has an unexpected orientation of hydroxyl groups, were isolated from the heartwood of *G. pedunculata* and *G. xanthochymus*.



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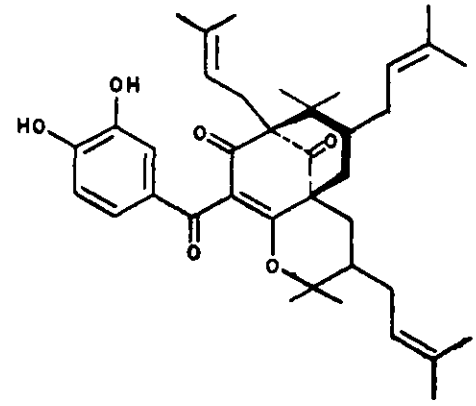
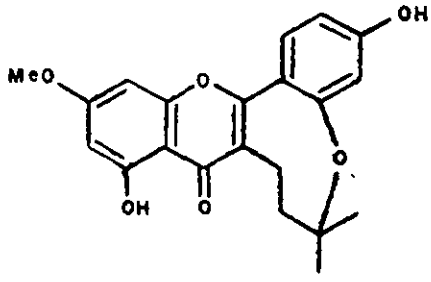


(II)

Artocarpus integer heartwood : From this Indonesian species ten known flavonoids and two new flavones (cyclointegrin and integrin) were isolated. The structures (III) and (IV) of the latter are of special biosynthetic interest because they are the first representatives in nature of flavones which

carry an alkyl or alkenyl substituent in the 3-position in the absence of such substitution in the A-ring derived from phloroglucinol.



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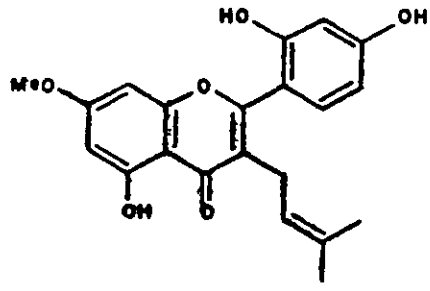

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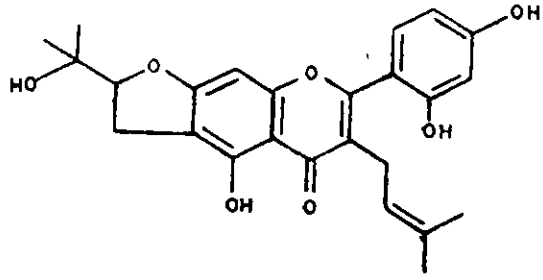
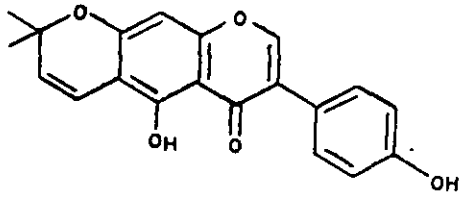
Morus species : In addition to mulberrin, cyclomulberrin, mulberrochromene and cyclomulberrochromene described earlier (*Tet. Letters* 1968, 1715), *M. alba* bark contains a new flavone, mulberranol (V). A new phenol, rubranol (C_{35} H_{28} O_8), was isolated from the bark of *M. rubra* which contains four C_{10}-substituted flavones (rubraflavones A, B, C, D; *Indian J. Chem.* 1974). Based on NMR and mass spectral data rubranol can be assigned one of three or four possible structures in which one molecule of a 7,2', 4'-trihydroxyflavan derivative is coupled with a second molecule carrying a C_5-substituent in the 3-position.



(V)

Albixia species : The bark of *A. procera* contains four known isoflavones, both as glycosides and aglycones. Isoflavones were not encountered so far in *Albizia* species. The heartwood contains the same isoflavones (as aglycones) and also 7,4'-dihydroxypterocarpan (demethyl-homopterocarpin). The heartwood extractives of *A. amara* and *A. lebbek* exhibited identical chromatographic behaviour. Melanoxetin and melacacidin were isolated in agreement with earlier workers; but other results were different. Mollisacacidin (leucofisetinidin a constituent of *Acacia mollissima*) and a new methyl ether of melanoxetin were isolated.

Erythrina variegata : The genus is well known for its alkaloidal content. From the bark four new isoflavones were isolated, the structures of three of which were determined. These are 6-prenyl-5, 7,4'- trihydroxyisoflavone, (VI) and its 5-deoxy-derivative.



(VI)

Chloroxylon swietenia : The bark is found to contain a new alkaloid and at least seven new coumarins, the structures of most of which were determined. In addition to the known alkaloid skimmianine, six known coumarins and two known lignans were present.

Groundnut shell : Work on the possibility of utilizing this waste product is in progress.

Tannins : Work on the tannins of *Terminalia tomentosa, Adina cordifolia* and a few other woods and barks was undertaken with the dual object of determining their structures and (in collaboration with the Central Leather Research Institute, Madras) examining their tanning properties, after chemical modification, if necessary.

5.2 *Preparation of nitromusk 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. A method for the manufacture of musk xylol and musk ketone was standardized. The products compared very well with imported samples. Likewise a process for the preparation of musk ambrette was also standardized. The scheme is now concluded.

5.3 *Synthesis of l-menthol from citronella oil of Indian origin* : (SP-89/72)

A process for the synthesis of *l*-menthol from *d*—citronellal obtained from Indian citronella oil was standardized and demonstrated to the sponsor. The party is taking necessary steps for the production of *l*-menthol from *d*—citronellal in their factory. Work on the project is concluded.

5.4 *Production of xylit from corn cobs* : (SP-81/72)

Xylit, the sweetest of all alditols, is used clinically for correcting abnormal metabolism in diabetics and for replenishing fluid and sugar.

Hydrolytic extraction of the hemicellulosic constitutents of corn cobs gave xylose as the main product contaminated with arabinose, glucose and galactose. Methods are being developed to eliminate the minor sugar constituents to obtain pure xylose which will then be catalytically reduced to xylit according to the process developed earlier.

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

Cotton fabrics were crosslinked with trioxane under different conditions of catalysis and curing. The extent of crosslinking, as determined by chromotropic acid method of estimation of formaldehyde in the treated fabric hydrolysates, was much below the expected level. This was traced to volatilization of trioxane during curing. Work on modification of the curing process is in progress.

Cotton fabrics were subjected to amination with aryl isocyanates to improve their dyeing properties. Aryl isocyanates were reacted with cotton fabrics under different conditions either by a direct method or by generating the reagent *in situ*. Both these methods gave encouraging results and are being pursued further.

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

Jute fabrics (IJIRA bleached) were subjected to crosslinking reactions with trioxane. The degree of crosslinking observed was low. The curing process is being modified to avoid the loss of trioxane.

The nature of the lignin and hemicellulosic constituents of jute (isolated and supplied by the IJIRA) are also under investigation for arriving at the figures of true accessibility of the hydroxyl groups of the cellulosic component of the jute fibre.

5.7 *Dialdehyde starch* : (ATT-219/73)

Dialdehyde starch is required in the manufacture of wet-strength paper for towelling, sanitary tissue paper, maps and the like. It is used in wood adhesive, paper coatings, pretanning of hides, in photographic films and polyvinyl alcohol films.

Dialdehyde starch was produced on 500 g. scale (yield, 95%) using maize and tapioca starch. The aldehyde content was estimated to be 95%. Work up of the spent liquor led to a 93% recovery of the oxidizing agent.

5.8 *Thin boiling tamarind kernel powder (TKP)* : (ATT-220/73)

Thin boiling TKP is needed for cotton and jute sizing, where it is desired to have a higher solid concentration, with a relatively small increment of paste viscosity. It is also used in paper sizing to some extent.

A series of samples of thin boiling TKP were prepared using various oxidizing agents and tested for their viscosity, pH and film-forming characteristics. A sample was sent to industry for evaluation.

5.9 *Cashewnut shell (CNS) gum* : (AB-76/69)

Investigations were conducted to find out suitable solvents to obtain the gum in a granular form. These investigations were useful to avoid the time consuming and tedious procedures involved in the precipitation of the gum. The conditions to obtain the gum in a granular form on 1 kg. batch were standardized.

Large scale experiments to process 45 kgs. of CN shells to obtain about 4.0 kgs. of CNS gum per batch were conducted. The final gum was obtained as a grey material. In all seven batches were processed. The product will be evaluated for different end uses by a firm interested in acquiring the know-how.

5.10 *D-Galactose* : (AB-97/70)

A process for the preparation of D-galactose in 35-40% yield from CNS gum, as well as the extract of CN shells was developed.

Experiments were successfully conducted to get about 400 gms. of D-galactose per batch. D-Galactose is usually prepared by hydrolysis of lactose which is however costly and has several other uses in pharmaceutical industries. Hence CN shell (an industrial waste) appears to be one of the cheap sources for the manufacture of this rare sugar.

5.11 *Synthesis of D-Fucose* : (AB-111/72)

Optimum conditions were established for the displacement of the 6-tosyloxy group from 1:2,3:4-di-*o*-isopropylidene-6-*o*-tosyl-*D*-galactopyranose.

5.12 *L-Rhamnose* : (AB-112/72)

Experiments were conducted with an indigenous gum to obtain L-rhamnose. It was observed that crystallization of L-rhamnose from the mixture of D-galactose and L-rhamnose was very difficult and the yield of crystalline L-rhamnose was about 1%. Further work is discontinued.

5.13 *Dissolving pulp* : (AB-114/73)

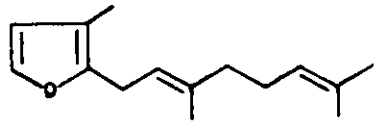
Terminalia tomentosa, Anogeisus latifolia, Madhuca latifolia and *Cleistanthus collinus* species from Chanda forest of Maharashtra were investigated for the production of high wet modulus pulp in mixture according to the latest percentage availability data in the forest. The results of the above tests confirmed the earlier results obtained under various percentage mixtures. Further, these species were separated into sapwood and heartwood chips

and studied for various chemical constituents. These separated chips of individual species of heartwood and sapwood were pulped under established conditions and the pulps evaluated for various chemical constituents. Similar study was also carried out on mixture of sapwood chips and heartwood chips.

Preliminary study of above species individually and in mixture by sodium base acid sulfite pulping process gave encouraging results.

5.14 *Chemical examination of essential oils, transformation products and synthesis of terpenoids* : (B-7·4/60)

Synthesis of sesquirosefuran (I) : Sequirosefuran, a furanoid sesquiterpene was recently isolated by Japanese workers and the structure was established on the basis of spectral data as (I).



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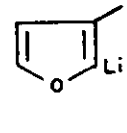

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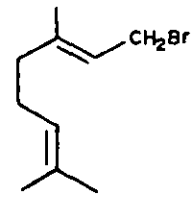

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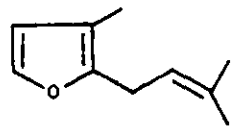
Synthesis of sesquirosefuran was successfully completed to confirm its structure, by condensing geranylbromide (III) with 2-lithio-3-methylfuran (II).

Synthesis of Elsholtzidiol (II)

Elsholtzidiol (II), a furanoterpenediol, was isolated from the essential oil of *Elsholtizidensa benth* and the structure was assigned on the basis of spectral data. It was synthesized in one step from Rosefuran (I) to provide synthetic confirmation of its structure.



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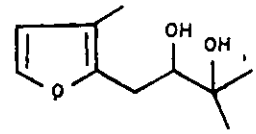

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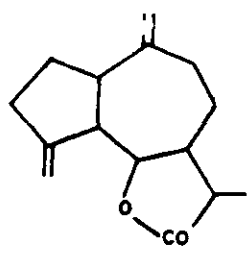
5.15 *Transformation products of costunolide and dehydrocostus lactone* : (B-8.15/66 and B-8.19/65)

Dihydrodehydrocostus lactone (I) was partially hydrogenated using platinum catalyst to give lactone (II) as one of the products. Epoxidation of lactone (II) followed by BF_3 etherate-rearrangement of the epoxide furnished a keto lactone (III) of known structure. This revealed the stereochemistry of (I) and of dehyroscostus lactone (IV) at the asymmetric centres C_5, C_6 and C_7.



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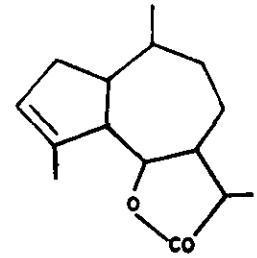

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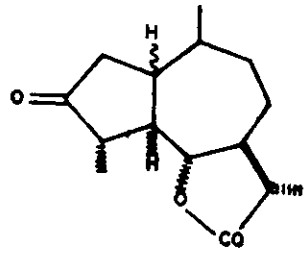

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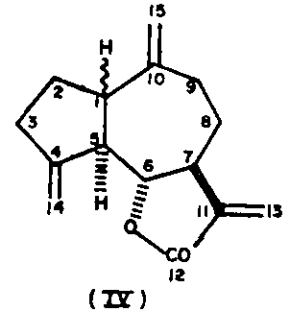

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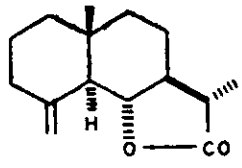
β-Cyclodihydrocostunolide (V) on epoxidation furnished two stereo-isomeric epoxides (VI & VII). These were isolated and characterized. Some more transformation of these epoxides are under study.



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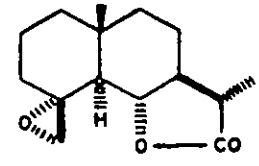

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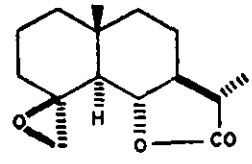

(VII)

5·16 *Compounds related to selinane, elemane and p-menthane* : (B-8.16/67)

Since a number of ∝,β-unsaturated γ-lactones showed cytotoxic activity, a new route was developed for the synthesis of ∝-methylenelactones. The new route will be employed to synthesize ∝-methylene lactones having *p*-menthane and selinane skeleton. β-Eudesmol was synthesized starting from santonin.

5.17 *Reagents for improving the wear-life and other surface properties of cotton and synthetic fabrics* : (B-8.58/72)

Several new chemical reagents containing reactive groups such as carbonylazido, sulphonazido, alkylazido and aminodicholorotriazinyl groups were prepared from long chain hydrocarbons and fatty acids. These compounds were found to improve the surface properties of cotton. The azido derivatives could also be combined reactively with synthetic fibres such as nylon and polyester resulting in their improved surface properties such as hydrophobicity and antistatic properties. Further work is in progress.

5.18 *Synthesis and reaction of epoxides and* α,β*-unsaturated ketones* (B-8.57/73)

A new route involving oxidative decarboxylation of β-aroylpropionic acids with lead tetraacetate in the presence of cupric acetate was developed for the synthesis of aryl vinyl ketones.

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

On the basis of fresh experimental evidence, alternative mechanistic pathways were suggested for the stannic chloride catalyzed glycosidation reaction.

Both of the anomers of several aryl and aralkyl hepta-*o*-acetyl maltosides were synthesized by the stannic chloride catalyzed glycosidation under different reaction conditions.

A modified procedure was developed for the preparation of penta-o-acetyl-α-D-mannopyranose in about 96% yield. Earlier methods gave yields of only 40-45%.

One of the new α-thioglycosides was found to be a successful competitive inhibitor for the separation of some enzymes by affinity chromatography.

6. CHEMISTRY OF BIOLOGICALLY ACTIVE COMPOUNDS

6.1 *Composite drug research scheme on Indian medicinal 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.

Asparagus racemosus Willd (*Shatavari*)

Mass spectrometric study of permethylated shatavarin-Ia, obtained by the partial hydrolysis of shatavarin-I and subsequent permethylation, indicated a possibility that the six-membered ring in the spiroketal system

might have opened. In order to verify this, it was attempted to get the genuine sapogenin by sodium metaperiodate oxidation. Besides this, shatavarin-I was peracetylated and hydrolyzed to yield a sapogenin. Sapogenin samples obtained by both methods are being studied further.

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

A number of extractions (40 kg. capsules/batch) were carried out and the extracts worked up further for the isolation of alkaloids. While the morphine recovery in the total extract was to the extent of 80-85% of that present in the capsules, the actual recovery of morphine from the extract was low. Work on the project is suspended for the time being pending further sponsorship.

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

Scale up work on the process of extraction of colchicine from *Iphigenia stellata* seeds was undertaken. After standardizing the process for the extraction of 14 kg. seeds/batch, it was further scaled upto 25 kg. seeds/batch size.

Colchicine was also extracted from the seeds and vegetative parts of *Gloriosa superba* separately, in order to explore some new and easily available sources.

6.4 *Chemistry of folk medicines in veterinary use* : (ATT-192/72)

Promotion of cattle wealth need not be stressed. 'Mange ' is a disease affecting the cattle; the hybrid ones being more prone to this disease. The skin is affected severely and may often cause death of the cattle. Hence, its control is of vital importance. The essential oil of *Cedrus deodara*, which is commercially available has been known to cure this disease.

With a view to identify, the component(s) of the essential oil responsible for this activity, the essential oil was fractionated and an oxygenated fraction was isolated which was found active. From the oxygenated fraction, a number of fractions were further separated by chromatography. These fractions are being tested for their veterinary activity.

6.5 *Chemistry of plant hormones* : (AB-104/72)

Plant hormones play an important role in the field of agriculture, horticulture and forestry.

The present work was undertaken to study the chemistry and physiological activity of different pollen extracts.

The ether extract of pollen/anthers from *Sorghum vulgare* (Jowar) was chromatographically separated into various fractions which were tested

by different assay methods. One of the fractions exhibited 4 times the growth regulating activity against the control in the rice callus tissue. Elaborate experiments are under way to confirm these results.

From the other fractions two hydrocarbons with the molecular formulae $C_{31}H_{64}$ and $C_{31}H_{62}$ and also a mixture of stigmasterol, sitosterol and campesterol were isolated. Preliminary work indicated that these two hydrocarbons possessed some insect attractant property. A systematic examination of these compounds is in progress.

6.6 *Plant growth regulators* : (AB-115/73)

Plant growth regulators are used for the controlled growth of plants as well as to aid the production and harvesting of various crops.

Maleic hydrazide is a plant growth regulator used mostly to prevent storage sprouting of onions, potatoes and certain other root crops; to increase sugar content in beet roots; to suppress suckering in tobacco leaves and to retard the growth of grasses and trees along the railway lines, highways and high power lines.

A process for the preparation of maleic hydrazide has been standardized and work on other plant growth regulators is in progress.

6.7 *Biologically active compounds of plant origin* : (AB-115/73)

The weed popularly known as 'Gajar gavat' (*Parthenium hysterophoras* Linn) spread all over the country is a menace to the agriculturist. It is found to be allergenic, insect-resistant, insect repellant, drought resistant and found to be very sturdy in its growth. In view of these properties and its availability in large quantities work was initiated to isolate and identify the active principles.

The screening of the various constituents from 'Gajar gavat' has been undertaken with a view to find out the toxicity of allergies and to find out whether any of the constituents possess insect repellant or insecticidal and/or plant growth regulating activity.

7. STUDIES IN ORGANIC SYNTHESIS

7.1 *Synthesis of polypeptides* : (ATT-215/72)

Peptide hormones like oxytocin and vasopressin are of therapeutic importance and are imported. Data on imports are however not available.

Solid phase peptide technology for the synthesis of protenoid hormones is being developed. A protected nonapeptide amideoxytocin—a peptide hormone—was synthesized on a polystyrene support.

7.2 *Peptide antibiotics* : (ATT-216/73)

Recently many peptide antibiotics have been discovered. Antiamoebin,

a peptide antibiotic supplied by Hindustan Antibiotics Ltd., has been reported to be of use in increasing production of milk in cows and buffaloes. This compound was found to be a mixture of five different compounds. Attempts are being made to purify and establish the amino acid sequence of the five components and to study the structure-activity relationship.

7.3 *Oxidation of olefins* : (AB-102/72)

Oxidation of cyclic olefins, in the presence of transition metal salts affords α, β-unsaturated ketones. A method was standardized to obtain these compounds in good yields.

This method was successfully applied for the oxidation of a few indigenously available olefins. Oxidation of isolongifolene and β-himachalene gave the corresponding α, β-unsaturated ketones as the major products. However, Δ^3-carene on oxidation gave a number of products, including the desired compound, partly due to the opening of the cyclopropane ring.

The project is concluded.

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

Catalytic oxidation and photo-oxidation were tried for the synthesis of a number of alcohols, ketones and acids starting from *p*-menthane, carane and pinane.

Oxidation of *p*-menthane resulted in a 35% conversion consisting of 25% neutral and 10% acidic products. The neutral products consisted of menthone, isomenthone, carvomenthone, etc.

Carane gave a very complex mixture of products.

Pinane, on oxidation, yielded three major components. The total conversion was 40%, consisting of 30% neutral and the rest acidic fraction. The major neutral component was identified as *cis*-2-pinanol.

The project is concluded.

7.5 2-*Ethylhexanoic acid* : (AB-126/73)

2-ethylhexanoic acid in the form of salts is used as a PVC stablilizer. Present demand is estimated at about 100 TPA and the landed cost of the imported product is about Rs. 19/- per kg.

After surveying the available literature and trying out some catalysts in preliminary experiments it was decided to explore the following two routes to the acid:

(a) Single stage catalytic conversion of 2-ethylhexanol to the acid and

(b) Vapour phase conversion of the alcohol to aldehyde and further oxidation to acid.

Encouraging results were obtained in both the series of experiments, but some attention was focussed on the latter on considerations of costs of materials. The catalyst chosen was found to have good activity. A number of experiments were carried out for finding out optimum conditions for conversion of the alcohol to aldehyde. Since catalyst activity fell with time, conditions for activation of the catalyst and time cycles required for obtaining high conversions were worked out. In an attempt to obtain better catalyst efficiency more finely divided forms of the catalyst mounted on suitable supports were made. Some of these are being tried and the results are still under evaluation.

Experiments were also done on the conversion of the aldehyde to the acid and almost quantitative conversions were obtained.

7.6 *Studies in Heterocyclic chemistry* : (B-8.7/65)

7.6.1 *Synthesis of compounds with potential biological activity*:

While studying the mechanism of the cyclodehydration reactions of β-phenylmercaptoethyl aryl/alkyl/cycloalkyl ketones, 2, 4-disubstituted-1-S-phenyl-l-thionium cyclobert-2-ene salts were envisaged as the intermediates. Some members of this new series of compounds were prepared and shown to rearrange to the respective 2, 4 -disubstituted thianaphthalenium salts and the corresponding 2, 4-disubstituted thiachromans.

Nitrogen heterocyclics: (B-8.50/69) Stereospecific synthesis of 2-methyl-4-phenyl-lN-(3'-methoxy) phenylazetidines was achieved. The stereospecificities in the photolytic, pyrolytic and acid catalyzed ring openings leading to various isomeric 2, 4-disubstituted 1, 2, 3, 4-tetrahydroquinolines were studied.

The scope of the cyclodehydration of arylaminomethylene cycloalkanones for the synthesis of novel polycyclic ring compounds was further extended to include polyheteroatomic systems.

7.6.2 *s-Trithianes*: A number of triaryl-s-trithianes were prepared starting from aromatic aldehydes and treating them with 0-0,-diethyldithiophosphoric acid. The conversion of s-trithianes and thioacetals into the corresponding aldehydes or ketones in good yields was achieved by novel oxidative desulphurisation [Tet. Letters. 3735 (1973)]. s-Trithianes can also be converted to the corresponding carbonyl compounds by using silver oxide in aqueous methanol.

7.6.3 *Synthesis of heterocyclic compounds using phenylisothiocyanate*: Making use of 2-amino-5-chlorobenzophenone (I), an intermediate used for diazepam and chlordiazepoxide, some new heterocyclic compounds were

prepared. Thus when (I) was treated with phenylisothiocyanate in benzene, a colourless product was obtained which was identified as 6-chloro-2-amino-4-phenyl-4-hydroxy-4H-3, 1-benzothiazine. 4H-3,1-benzothiazines are known to have very good tranquilizing properties.

The reaction of phenylisothiocyanate on the oxime of (I) resulted in the formation of a mixture of 6-chloro-2-anilino-4-phenylquinizoline and its N-oxide.

7.7 *Synthesis of steroids* :

Work on a new short synthesis of estrone has been undertaken with an object to develop a practical synthetic route to steroidal starting materials for drugs. The method involves setting up of the steroidal ring system in a simple, efficient and stereospecific way. This will have a great impact on synthetic steroidal drugs.

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

The reaction of 2-α-10 epoxypinane and 2-β-10-epoxypinane with alumina impregnated with NaOH or NaCl was studied. Both epoxides, irrespective of their difference in stereochemistry behaved in a similar manner giving myrtenal, 2-pinene-10-ol and trans-myrtanol. The percentage composition of these products was different with Al_2O_3 -NaOH and Al_2O_3 -NaCl.

It was observed, previously, that the carbonium ion rearrangement constituted the chief pathway open to oxirane ring isomerization in a silica gel matrix. In this connection, the behaviour of 2-α-10-epoxy and and 2-β-10-epoxypinane and 3-β-,4-β-epoxycarene towards silica gel was studied. As expected, both the pinane epoxides afforded trans-myrtanal as a major product besides 2-pinene-10-ol and perilla alcohol in minor amounts.

The project is concluded.

8. PHYSICO-CHEMICAL PROPERTIES OF MATERIALS

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

The studies on the isobaric vapour liquid equilibria (T, X, Y diagram) over the entire range of compositions of the binary mixtures, of the main components of the turpentine oil, viz, α-pinene, β-pinene and Δ^3-carene were completed. With the data thus obtained, a plant design for the fractional distillation of α-pinene, β-pinene and Δ^3-carene from the turpentine oil will be offered to the sponsor.

8.2 *Polyesters as stationary phases in GLC analysis* : (AB-33/73)

This is a continuing project and developmental work is undertaken along with routine analysis of samples.

Developmental work on the analysis of various pesticides, now being developed in the laboratory, was carried out.

8.3 *Diffusion of Co in Ag* : (B-2.1/67)

In an earlier study on the diffusion of ^{60}Co in Ag, it was shown that the diffusion of cobalt in silver took place by the formation and the motion of vacancies. To obtain more definitive information on the mechanism of diffusion of Co in Ag, the mass dependence for the diffusion of Co in Ag by studying the diffusion of ^{59}Co and ^{60}Co together, was determined in the temperature interval 814°-910°.

The experimental value of the correlation factor 'f' was found to vary from 0.74 at 814° to 0.60 at 910°. The variation in the value of 'f' was explained arising out of simultaneous operation of single and divacancy diffusion mechanism. It was also shown that there was a large contribution of the divacancy to the diffusion process and this contribution increased sharply with the rise of temperature.

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

In continuation of the earlier work, the heats of mixing of the binary mixtures of water, methanol, ethanol, 1-propanol and 1-butanol with normal butylamine at 25° were determined. The results could be described by the equation:

$$\Delta H^m(\text{K.J. Mole}^{-1}) = x_i\, x_2\, B + C(x_1\text{-}x_2) + D(x_i - x_2)^2$$

where x_2 is the mole fraction of n-butylamine, B,C and D are constants whose values are tabulated below:

Systems	B	C	D
H_2O-n-$BuNH_2$	—13.8003	—3.1626	+3.1717
CH_2OH-n-$BuNH_2$	—15.4021	—3.5341	+1.7200
C_2H_5OH-n-$BuNH_2$	—11.9015	—2.2767	+1.015
n-C_3H_7OH-n-$BuNH_2$	—12.2746	—1.685	+3.4692
n-C_4H_9OH-n-$BuNH_2$	—10.157	—0.3868	+0.3687

Isothermal vapour-liquid equilibrium measurements at 40° were completed on a Jone's still for the systems: (i) n-butylamine-n-propanol and (ii) n-butylamine-n-butanol throughout the concentration range of the binary mixtures. It was observed that the n-$BuNH_2$-n-C_3H_7OH mixture formed an azeotrope at ca. 0.11 mole fraction of n-$BuNH_2$.

8.5 *Thermodynamic properties of complex ions in solutions* : (B-2.15/70)

A comparison of the enthalpy changes accompanying the formation of bis-monothiodibenzoyl methanato-Ni(II) and bis-dibenzoylmethanato-

Ni(II) in 75 volume % dioxane + 25 volume % water medium showed that the Ni(II)-sulphur bond in the former chelate is nearly one and half times stronger than the Ni(II) oxygen bond. Equal entropy changes accompanying the formation of the above two chelates indicated similar stereochemical arrangements of the donor atoms around the central Ni(II) atom in the dioxane-water medium.

A comparison of the enthalpy and entropy changes associated with the formation of 1 : 1 complex of Ni(II) and Cu(II) ions with N, N'-bis (3-aminopropyl)-1, 2-ethanediamine (designated as 3,2,3-Tet) with the corresponding values available in the literature for the other linear tetramine ligands; N,N'-bis(2-aminoethyl)-1, 2-ethanediamine (designated as 2,2,2-Tet), N, N'-bis-(2-aminoethyl)-1,3-propanediamine (designated as 2,3,2-Tet) and N,N'-bis-(3-aminopropyl)-1, 3-propanediamine, showed the following order: $-\Delta H$,

$$2,3,2\text{-Tet} > 3,2,3\text{-Tet} > 2,2,2\text{-Tet} > 3,3,3,\text{-Tet}$$

and $+\Delta S$,

$$3,3,3\text{-Tet} < 3,2,3\text{-Tet} < 2,3,2\text{-Tet} < 2,2,2\text{-Tet}$$

The above order of decreasing values of exothermic enthalpy changes could be explained as a result of the two opposing contributions to the total enthalpy change viz. (i) increase in the exothermic enthalpy of formation as a result of the relief in the cumulative ring strain in fused three-5 membered metal chelate ring system when 5-membered chelate ring is replaced by the 6-membered chelate ring, (ii) the lower exothermic enthalpy of formation of a 6-membered chelate ring relative to a 5-membered chelate ring.

The above increasing positive entropy changes arose as a result of increasing loss in the internal degree of freedom of the ligands on coordination to a metal ion, as the chain length of the ligands is increased.

8.6 *Thermodynamic properties of binary molten salt mixtures* : (B-2.14/72)

The work on the measurement of the E.M.F. of the formation cell for the system $AgI+CdI_2$ was continued for various mole fractions and at different temperatures. The formation cell used for the experiments was of the type.

$$-Ag \;\Big|\; \underset{x_1 \qquad x_2}{AgI + CdI_2} \;\Big|\; I_2(g) \quad {}^{+}C \quad (1\ atm)$$

where Ag^- was silver electrode and C^+ graphite electrode and x_1, x_2 represent mole fractions of AgI and CdI_2 respectively. Activities of the AgI in the molten mixture AgI + CdI_2 were determined for 11 mole fractions between 0.18 to 0.87 of AgI. The values of the activities of CdI_2 were

calculated from the Gibbs-Duhem equation. The data collected so far showed large deviations from the ideal behaviour in the region of 0.6-0.7 mole fraction of AgI suggesting a possibility of the formation of the species Ag_2 (CdI_4). Further work is in progress.

8.7 *Mossbauer and X-ray diffraction studies on the ferrite,* FeV_2O_4: (B-3.13/73)

The ferrite FeV_2O_4 had a cubic spinel structure and was paramagnetic at room temperature. Earlier crystallographic and magnetic studies reported in the literature gave conflicting information on its cation distribution.

Mossbauer spectra of FeV_2O_4 determined at 298°K and 193°K could be resolved into a pair of two quadrupole split doublets of unequal intensities. The outer pair of lines could be attributed to the Fe^{3+} ions at B sites while the inner pair to the F^{2+} ions at A sites. These results gave an evidence of a partially inverse cation distribution in FeV_2O_4. By comparing the relative areas under the two sub-spectra, the degree of inversion in FeV_2O_4 was found to be 10 and 13 per cent at 298°K and 193°K respectively.

Cation distribution in FeV_2O_4 at room temperature was also determined by X-ray diffraction by calculating theoretically intensities of reflections from different hkl planes for 5, 8, 10, 12, 15 and 20 per cent inversion and 'u' parameter in the range of 0.375—0.390 in the interval of 0.0025. The combination of u=0.385 and the degree of inversion equal to 8% gave the least value of the reliability factor.

$$R = \frac{\sum \left[\left| I_{hkl} \right|_{obs.} - \left| I_{hkl} \right|_{cal.} \right]}{\sum \left| I_{hkl} \right|_{obs.}}$$

The degree of inversion of 8% at room temperature determined by X-ray powder diffraction was in good agreement with the value of 10% determined by Mossbauer spectroscopy.

8.8 *Thermodynamic properties of solutions—Studies on adiabatic compressibility of macromolecules* : (B-2.16/65)

A considerable amount of work was reported earlier on adiabatic compressibility of polyanions but investigation on polycations was limited. A study of adiabatic compressibility of anionic and cationic polymers separately and then in combination in the form of amphoteric polyelectrolyte is underaken to elucidate the nature of interaction between the acid and base groups and the resultant molecular configuration when they are put together on the same polymer chain. This study was further extended to a cationic polymer and the copolymers made with this cationic polymer and other anionic polymers studied before.

The adiabatic compressibility of a cationic polymer, poly (N-di-methyl-aminoethyl methacrylate) (PDAM) and of three copolymers of N-dimethyl-aminoethyl methacrylate and acrylic acid, ranging in composition from 33 to 58 mole percent of amino groups, were studied. The ϕV_2° of the polymer showed a slight decrease (2.4 cc/mole) while the ϕK_2° was found to have increased considerably (51×10^{-4} cc/bar/mole) compared to that of the monomer, DAM. Since the magnitude of electrostriction was higher in fully neutralized salt than that of the unneutralized polymer, the ϕK_{2f}° and ϕV_{2f}°'s were found lower in salts. The difference in these values for the polymer and its hydrochloride salt was 23.7×10^{-4} cc/bar/mole and 7.5 cc/mole respectively which could be due to electrostrictive effect.

The experimentally observed ϕK_2° and ϕV_2°'s for the three copolymers containing 58%, 43% and 33% amino groups were 2.5×10^{-4} cc/bar/mole and 164.5 cc/mole, -32×10^{-4} cc/bar/mole and 177.5 cc/mole, and -55×10^{-4} cc/bar/mole and 211 3 cc/mole respectively whereas the calculated values were less by 19.4×10^{-4} cc/bar/mole and 3.2 cc/mole, 49.5×10^{-4} cc/bar/mole and 19.9 cc/mole and 73×10^{-4} cc/bar/mole and 16.4 cc/mole respectively. This decrease was ascribed as due to the interaction of the acid and base groups in the molecules. Further work for the sodium and hydrochloride salts of the amphoteric polyelectrolytes is being carried out.

A second ultrasonic interferometer was calibrated for the compressibility measurements of polymer solutions in nonaqueous medium.

8.9 *Crystallography* : (B-2.5/67)

During this period, precise structures of 12-ethoxy-2,3-benzo-6,5-naphtho (b) (7,12)- thiophosphorin-7, 7, 12-trioxide and 2-nitro-4-methyl-benzoic acid were determined with the help of full three dimensional X-ray data. The structure of *o*-nitrobenzoic acid which was determined earlier was further refined using anisotropic thermal parameters in order to have a better comparison with the structures of *p*-and *m*-nitrobenzoic acids.

12-Ethoxy-2,3-benzo-6,5-naphtho (b) (7,12)-thiophosphorin-7, 7,12-trioxide was a phosphorus analogue of phenothiazine and was found to be biologically active. The crystals were monoclinic, space group $P\frac{2_1}{n}$ with $a = 14.71 \pm 0.01$, $b = 8.07 \pm 0.02$, $c = 14.30 \pm 0.01$ Å; $\beta = 94.0 + 0.2^\circ$; ρ_o, 1.406 g.cm^{-3} and ρ_c for Z=4, 1.405 g. cm^{-3}. The structure was solved by direct methods using visually estimated data, and refined by the least squares method to an R value of 0.115 for 1907 observed reflexions. The dihedral angle between the plane of the naphthalene ring and that of the benzene ring on either side of the sulphur and phosphorus atoms was 133.8°; P lay in the naphthalene plane and S was 0.15 Å away from it, whereas S lay in the benzene plane and P was 0.15 Å away from it. The S = O and S-C bonds around sulphur atom had a slightly distorted tetrahedral configura-

tion suggesting greater *S* character of S=O bonds than S-C bonds; the distortion in tetrahedral configuration around P was more severe. The ethoxy group underwent large anisotropic thermal vibrations owing to loose van der Waals contacts with atoms of the neighbouring molecules.

The structure analysis of 2-nitro-4-methyl benzoic acid was undertaken in order to study the influence of methyl group substitution in para position (with respect to COOH) on the structure of *o*-nitrobenzoic acid. The compound crystallizes in two forms: form I in space group $C\frac{2}{c}$ and form II in space group $P\frac{2_1}{c}$ The detailed investigation of form I was undertaken. The crystals were monoclinic, space group $C\frac{2}{c}$ with a = 13.26, b=11.03, c=11.42 Å; $\beta = 94.6°$ and Z = 8. The phases of 150 reflexions were obtained by direct method; the three dimensional Fourier map revealed the structure completely. The structure was refined by the least squares method to an R value of about 0.106 for 1323 observed reflexions.

8.10 *Spectrochemical studies* : (B-5/63)

8.10.1 *Infrared spectra and Molecular structure*

Detailed infrared analysis of benzbetaines and their hydrohalides revealed that the benzbetaines did not form inner salt structures even in the ortho derivative, but existed as dipolar ions in the solid state. Characteristic frequencies of N $(CH_3)_3$ group were found in the region 1280-1240, 100-950, 820-775 Cm^{-1} in these compounds.

8.10.2 *Association behavior of alkoxyethanols*

The self association of methoxy, ethoxy and butoxy ethanols in carbon tetrachloride solution was investigated in the temperature range 25-65°. The equilibrium constants were found to be independent of the chain length. The AH°, AG° and AS° of the triangular equilibrium of Dimer (D), bonded monomer (B) and free monomer (F) were $\triangle H°$ 13,6.8 and 3.2 K.cal/mole $\triangle G°$ 3.4, 0.66 and 1.38 K.cal/mole; $\triangle S°$ 36, 21 and 6 e.u./mole. The intramolecular and intermolecular hydrogen bond strengths were found to be 3.2 and 6.5 K. cal respectively.

9. STUDIES IN PHYSICAL ORGANIC CHEMISTRY

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

(a) *Reactivity of o-hydroxyarylamides in alkaline solution*

The reactivity of *o*-hydroxyarylamide in alkaline solution towards reactive halides like 2, 4-nitrochlorobenzene was interpreted in terms of different mechanisms in earlier investigations. The claims made were apparently

conflicting. A thorough study was made of the nature of the anions of these amides using NMR and IR spectroscopy. Data of considerable importance were obtained and their significance for chemical reactivity is under assessment.

(b) *Reaction of carbon tetrachloride with anions*

The reaction between carbon tetrachloride and sodium phenoxide in DMSO solvent was studied in some preliminary experiments. Several other anions were found to react with carbon tetrachloride. Work on the identification of the products involved and the general course of these reactions is under way.

9.2 *Studies on conjugated systems* : (B-5.2/62)

Extensive studies on measurement of the strength of conjugation between donor and acceptor groups in benzene derivatives were made in earlier investigations. 4-Substituted phthalic esters from one series of compounds were used in this connection. It was noted that the NMR spectrum of the phthalic ester could not be analyzed well as an AA' BB' system. The possibility that the spectrum is an average of two identical ABCD spectra is being examined. A suitable modification of the computer programme in use is being made.

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

9.3.1 *Electron impact, photolysis and pyrolysis of organic compounds*: The pyrolytic and mass spectral behaviour of 1,2-dimethyl-4-nitro-5-phenyl-$\triangle^1$-cyclohexene was studied. The major pyrolytic product was 3,4-dimethylbiphenyl which was formed by the loss of nitrous acid and a molecule of hydrogen. The mass spectral behaviour was similar to the pyrolytic decomposition.

9.3.2 *Electron impact, chemical ionization and field desorption spectral studies* : The fragmentation reaction of isomeric 3,5,6-triphenyl-4-amino- $\triangle^1$-cyclohexenes was studied under electron impact, chemical ionization, and field desorption conditions. The differences observed were rationalized on the basis of the different competing fragmentation modes which operated under these ionizing conditions. It was shown that these techniques were complementary.

9.3.3 *Ion kinetic energy (IKE) spectroscopy* : The metastable ion decompositions in the first-field free region of the mass spectrometer of some isomeric pesticides (aldrin, isodrin, endrin and deldrin) were studied by this technique. A study of the IKE spectra of some di-saccharide acetates with different glycosidic linkages showed that it was possible to differentiate them by this method.

9.4 *Mass spectral techniques* : (B-5·8/65)

Efforts were made to improve the ion current integration method of quantitatively estimating organic compounds by mass spectrometry.

9.5 *Electron impact fragmentation of triaryl-s-trithianes*

The mass spectral fragmentation of seven substituted 2,4,6-triaryl-s-trithianes were studied and the fragmentation modes were confirmed by deuterium labelling. The prominent radical ions in the mass spectrum of triphenyl-s-trithiane were at m/e 180 (52.3%) and m/e 186 (45.2%) corresponding to stilbene and $PhCHS_3^+$. The formation of stilbene indicated a relationship between pyrolitic and electron impact studies. The origin of $PhCHS_2^+$ suggested that one of the sulphur atoms attaches itself to the other two sulphur atoms in the molecular ion, eliminating a stilbene radical ion. The other important fragmentation corresponded to the monomer radical ion (thiobenzaldehyde) and the thiobenzoyl cation.

10. INDUSTRIAL POLYMERS, RESINS AND ELASTOMERS

10.1 *Expandable polystyrene* : (ATT-34/66)

As reported earlier the project was undertaken to develop indigenous know-how for the preparation of expandable polystyrene beads by suspension polymerization technique.

The technology was standardized on 6 kg/batch of expandable beads and samples were sent to several actual consumers for evaluation. The product compared well with a similar product available in the market.

The process is offered for commercial implementation through NRDC.

10.2 *Polyurethane coating compositions for textiles and other substrates* : (ATT-46/72)

As reported earlier work on the preparation of colourless, odourless polyurethane coating formulations for water proofing of textiles was continued. A rubber composition was prepared on a scale of 2 to 4 kg. per batch which was found to possess excellent adhesion to natural and synthetic fabrics, low temperature flexibility, good abrasion resistance and water-proofing properties with low proofing weight.

The water proofing properties of the nylon and rayon fabrics coated with the above rubber formulation was found quite satisfactory.

Applications of the rubber formulation in the field of conveyor belt and bonding paper manufacture are being evaluated.

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

Nitrile rubber, a copolymer of acrylonitrile and butadiene is extensively used where oil resistance is of primary importance. Nitrile rubber is not manufactured in the country at present. Its import is estimated to be 500 TPA valued at Rs. 50 lakhs. A major proportion of the nitrile rubber is of medium nitrile content (34% bound acrylonitrile).

The process developed in the laboratory for the medium nitrile content rubber (34%) was demonstrated to M/s. Synthetics and Chemicals, Bombay, who have now commercialized the process at their Bareilly pilot plant.

The process conditions, optimum conversions, efficient short stop combinations for polymerization and the coagulation conditions were standardized for the preparation of high nitrile content rubbers (8 kg. rubber/ batch). Samples of the rubbers were sent for evaluation to M/s. Synthetics and Chemicals, Bombay. Their evaluation report is awaited.

The process conditions are also being optimized for the low nitrile content rubber (combined acrylonitrile content 25± 1%). The reaction rates were slightly slower than desired. Three samples are being evaluated by the above party.

10.4 *Synthetic polymers for sugarcane juice clarification* : (ATT-86/68)

A synthetic imported polymer (Separan AP-30) is presently used in the Indian sugar industry as a flocculating agent for the clarification of sugarcane juice. The estimated present annual demand for this product is valued at Rs. 10 lakhs. The project was undertaken to develop a substitute for the imported flocculating agent.

High molecular weight polyelectrolytes based on acrylamide and acrylic acid were found highly effective flocculating agents for the clarification of sugarcane juice.

A laboratory-scale process (1.5 kg./batch) was developed for the preparation of a co-polymer equivalent to Separan AP-30. Samples of different grades were prepared of which two types were found suitable. In factory trials their performance was found comparable with the imported ones. The process is offered to industry for commercial implementation.

10.5 *Polysulphide rubber* : (ATT-89/67)

Polysulphide rubbers are mainly used for the preparation of oil and fuel resistant sealing materials and adhesives. These rubbers are mostly needed by the Defence Dept. and the present requirement is met totally by imports.

Some exploratory work for the preparation of liquid polysulphide rubber was done earlier. Scale-up experiments are in progress.

10.6 *Sulphochlorinated polyethylene elastomer (SCPE)* : (ATT-90.1/68)

This polymer finds a variety of end uses due to its excellent properties

viz., light weight, chemical resistance, thermal stability and weatherability. The estimated demand of SCPE is about 200 TPA valued at Rs. 40 lakhs. The development of a one step laboratory process for the production of general purpose SCPE was reported earlier. This was found suitable for the manufacture of moulded rubber goods, as a paint base, and as a modifier for other elastomers and plastics.

The sulphochlorination step was successfully carried out on a large scale to obtain the engineering data. However, difficulties were encountered in the recovery of the polymer from the reaction product. The wet precipitation method is being tried under different conditions and the products obtained are being evaluated.

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

The processes developed earlier for the preparation of dibutyl tin maleate (liquid) and dibutyl tin laurate were scaled up to 5 kg./batch scale. The products were found acceptable by the industry. The process is being offered to industry for commercial implementation.

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

The resin is primarily used for imparting a protective and glossy surface finish to paper and in printing inks. Its present imports are of the order of 250 TPA valued at Rs. 25 lakhs.

As reported earlier efforts were made to prepare a product with high molecular weight, high gloss and good transparency. Samples were prepared accordingly and are being sent to industry for evaluation.

10.9 *Polyacetal resins* : (ATT-196/72)

These are linear thermoplastic polymers of formaldehyde. In view of their superior properties these polymers are good substitutes for non-ferrous metals and are finding increasing use in various fields such as fabrication of gears, pumps, speedometers, etc. Polyacetal resins are not yet manufactured in the country and the indigenous demand is estimated as 1000 TPA valued at Rs. 2 crores.

Polymerization of trioxane was successfully carried out to obtain 80-95% yields of a product with intrinsic viscosity of 1.5 to 1.7. Experiments for stabilizing the polymer are in progress. Exploratory experiments to obtain ethylene oxide copolymer of trioxane have shown encouraging results.

10.10 *Polyurethane rigid foam* : (AB-96/71)

Rigid urethane foams have unique properties like high strength to weight ratio, low thermal conductivity, low water vapour transmission, buoyancy and ideal electrical properties. Earlier work done in this field was

connected with the use of these foams for artificial limbs. The objective of the present work is to develop rigid foam for spraying purposes needed by Defence and Atomic Energy Establishment, Trombay.

Moldable and sprayable rigid foams based on CNSL shellac polyester were prepared. A machine for this purpose has been fabricated. Trials with the machines and with different polyester compositions are under way.

10.11 *Physico-chemical studies in polymers* : (B-13.1/60)

Copolymerization of ionizing monomers : In copolymerization initiated by a free radical, the monomer reactivity ratios are independent of the dielectric constant of the solvent and any other polar, dipolar or induced dipolar interactions. Several combinations of monomers have already been studied extensively in the organic media. Very little work however is reported with ionizable monomers in aqueous systems.

The reported values of ionic monomers varied widely with variation in ionization. The monomer reactivity ratio of the system acrylic acid-acrylamide changed drastically with the degree of ionization of acrylic acid. Thus, a detailed study was undertaken to understand the effect of ionic change in the monomers like acrylic acid, methacrylic acid, on their free radical copolymerizations. The copolymerization behaviour of the system acrylic acid, N-vinylpyrrolidone was studied as a function of the degree of ionization of acrylic acid and N-vinyl pyrrolidone. The effect of the dielectric constant of the media and electrolyte monomer interactions on the monomer reactivity ratios was also investigated. The copolymerizations were carried out to high conversions and it is proposed to use the integrated form of the copolymerization composition equation to avoid the errors due to (i) small amount of impurities which alter the initial reactivities of monomers,etc., (ii) large difference in the two monomer reactivity ratios, and (iii) improper graph fitting.

The solution behaviour of the copolymers is proposed to be studied. Light scattering and osmometric measurements throw light on polymer solvent interactions of the copolymers in comparison with those of the homopolymers. The excess thermodynamic functions of the copolymers and homopolymers prepared in aqueous medium gave useful information about the structure of the polymers.

10.12 *Thermodynamic properties* : (B-13.5/69)

The new bond-energy/group-contribution scheme for calculating standard heat of formation of monomers and polymers, was extended to halocarbons. The work has been forwarded for publication.

A novel analytical procedure for calculating monomer reactivity ratios (kinetic parameters) in copolymerization reaction was evolved. The

method was computerized along with other published procedures and its efficacy was tested. The results however were not encouraging. A comprehensive research paper on the comparative study of these methods was published indicating the best available procedure for precisely recalculating the existing copolymerization data.

10.13 *Radiolytic polymerization of trioxane* : (B-3.11/71)

The polymer obtained by the radiation method was highly crystalline and resisted acetylation. Upon reprecipitation it could be readily acetylated. Thermal decomposition in vacuum was found less than 0.1 per cent per minute. The acetate was stabilized by incorporating stabilizers. The finished stabilized polymer compared favourably with industrial Delrin in respect of molecular weight and thermal stability. Cost estimates for the production of polyoxymethylene polymer by gamma-ray polymerization of solid trioxane are being worked out.

11. INORGANIC CHEMICALS AND CATALYSTS

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

Silicon tetrachloride is an important chemical used in the manufacture of fumed silica which in turn is used as a filler in plastics and as a dispersing agent for insecticides. It is also used for the production of ethyl silicate (ES-40), which finds application as a binder in precision casting and as a bonding agent for commuted materials.

The country's demand for silicon tetrachloride is estimated to be about 2000 TPA valued at Rs. 40 lakhs. Demand for ethyl silicate is estimated to be about 100 TPA valued at Rs. 10 lakhs.

A continuous process for the manufacture of silicon tetrachloride in a fluidized bed was developed on a pilot plant (70 TPA capacity), and the necessary process and engineering data were obtained.

Starting with silicon tetrachloride a process for the production of ES-40 (ethyl silicate with 40% silica by weight) was standardized. Both the processes are being offered to industry for commercial exploitation.

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

Sodium hydrosulphite is used in large quantities in the textile, paper, sugar and leather industry and is in short supply at present. The combined production using zinc and electrolytic methods is around 8000 TPA. The country's current requirement is 11000-12000 TPA and the internal demand is expected to go up further to 14000 tonnes by 1975-76.

Manufacture of sodium hydrosulphite using sodium formate would avoid the use of zinc. Know-how for this process was developed on a laboratory

scale (1/2-1 kg./batch). The price, purity and stability of the product were comparable with those obtained by the coventional zinc process.

11.3 *Molecular sieve catalysts for alkylation reactions* : (AB-84/70)

The project aims at the development and testing of catalysts for alkylating aromatics with olefins to obtain mono-alkyl aromatics such as ethyl benzene, propyl benzene, etc.

Based on the X type (developed at NCL) and Y type (imported) zeolites several new catalyst samples were prepared. The chemical composition, X-ray structure, and characteristic adsorption properties of the catalysts were evaluated.

A modified all glass reactor assembly for testing the catalysts has been fabricated and the determination of the catalytic activity of the above improved catalysts is in progress.

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

Pearl pigments are used to impart lustre, brilliance, depth and reflectance to plastic, leather, artificial pearls, polyester moulding for buttons, novelties, nail polish, paints, printing inks, etc.

Laboratory scale preparation of bismuth oxychloride was reported last year.

Basic lead carbonate is the major inorganic pearl pigment essence used in plastic industries. The preparation of this material was standardized optimising precipitation conditions, such as pH, concentration and temperature. However, the preparation of a paste of this material to get perfectly aligned parallel layer of substantially uniform crystals offered some difficulties. Use of suitable surface active agents to counteract the agglomeration of the platelets is being tried.

11.5 *Synthesis of molecular sieves* : (AB-107/72)

Preparation of molecular sieves types 3A, 4A, 5A, 10X and 13X both in the powder form and in the form of 1/16" diameter extruded pellets has been standardized. The sieves thus produced, were comparable to the corresponding grades of imported Union Carbide Linde Division Molecular Sieves, in respect of crystal structure, chemical composition, surface area and adsorption of suitable gases of varying nominal molecular diameters. The know-how is being released to industry.

11.6 *Preparation of dicyandiamide* : (AB-119/73)

Dicyandiamide is used for the manufacture of melamine, amino resins and also finds application as an intermediate in drugs, explosives, plastics,

etc. Practically the entire demand of dicyandiamide (2000 TPA valued at Rs. 6 crores) is met by imports.

The preparation of dicyandiamide from calcium carbide involves three steps viz., azotization, extraction and isolation of cyanamide and dimerization.

Azotization of calcium carbide was studied using different flux and mixture of flux materials. The experimental conditions such as flow rate, temperature, types and quantity of the flux were optimized, to get 80% conversion efficiency.

Extraction of calcium cyanamide and its isolation as sulphate,oxalate or carbonate are under study.

11.7 *Precipitated silica* : (AB-121/73)

Precipitated silica is used as a filler in rubber, plastics and pigments and as additive in greases and paper coating. At present the value of annual imports is about Rs. 30 lakhs.

Silica aerogel prepared was found to be comparable with an imported sample (Aerosil 200).

11.8 *Special grade alumina for electronic industry*

Special grade alumina of high chemical purity, high thermal conductivty, low electrical conductivity, low moisture retention and comparatively low sintering temperature, is required for the housing of transistor diodes, for making substrates for integrated printed circuits, etc. Other uses include printing inks, paper, rubber and plastics. It is estimated that Rs. 1.5 crores worth of special grade alumina is being imported for these uses.

The various types of alumina being imported by Bharat Electronics, Bangalore, were analysed and their physical characteristics were determined.

12. UTILIZATION OF MINERAL RESOURCES

12.1 *Titanium tetrachloride* : (ATT-224/73)

Titanium tetrachloride is used in the manufacture of rutile grade titania. It also finds application in metallurgy and in the manufacture of some intermediates and polymerization catalysts. The demand for titanium tetrachloride in the country is of the order of 2000 TPA valued at about Rs. 1 crore.

A continuous process for the production of titanium tetrachloride (2.5 kg/hr) by the chlorination of beneficiated ilmenite in a fluidized bed in

the presence of oxygen acceptors like coke at high temperature was developed. The necessary process and engineering data required for the design of a commercial plant was collected. The process is now being scaled up in a pilot plant of 150 TPA capacity.

12.2 *A cyclic process for getting soluble P_2O_5 from Udaipur rock phosphate* : (AB-117/73)

Udaipur rock phosphate is at present being tried for phosphoric acid production by the conventional method, which involves use of sulphuric acid. This can be avoided if the IMI process using hydrochloric acid and solvent extraction is adopted. There is also a possibility of developing a cyclic process using ammonium bisulphate which is regenerated and recycled.

The reaction between ammonium bisulphate and high grade rock phosphate proceeded satisfactorily (over 95% efficiency) in the presence of a minimum amount of water. The phosphoric acid was then shaken with a solvent and water. The suspended solid calcium sulphate was removed. The liquid portion separated into two layers. The upper layer contained about 60% of the original P_2O_5 and very little sulphate. The lower aqueous layer contained almost all the sulphate and the residual P_2O_5. Further studies on the extraction of the major portion of the phosphoric acid either by crystallizing the ammonium sulphate or by cycling the liquor rich in ammonium sulphate back into the process is under study.

12.3 *Studies on the utilization of Indian fluorspar* : (AB-118/73)

Fluorspar produced by the Gujarat Mineral Development Corporation was assessed for its use as a potential source of hydrofluoric acid. The project was undertaken to study the extraction and volatalization of fluoride from Indian flurospar and its conversion to cryolite.

Experiments were carried out with the crude (20% CaF_2) fluorspar, as well as with the partially upgraded material (90% CaF_2). In both cases, over 95% of the fluorine could be volatilized. The crude fluorspar needed higher temperatures than the upgraded one. After absorption in water, the hydrofluoric and hydrofluosilicic acids were converted to sodium fluoride (solution). This step as well as preparation of cryolite from the sodium fluoride was standardized on a laboratory scale.

12.4 *Beneficiation of ilmenite* : (AB-129/73)

Ilmenite is abundantly available in India and beneficiated ilmenite has a dependable export market. A plant with a capacity of 10,000 TPA will have a turnover of about Rs. 1 crore.

A new technology for continuous beneficiation of ilmenite by chlorination of ilmenite at high temperature in a fluidized bed reactor was esta-

blished on a laboratory scale (1.5 kg./hr.). A pilot plant of 10 kg./hr capacity is now being operated for collection of design data.

12.5 *Bacterial leaching of ores* : (AB-67·7/73)

This is an interlaboratory project taken up to isolate and identify bacteria capable of leaching out copper from low grade copper ores. This is expected to utilize the available as well as the potentially available low grade ores of copper.

Some of the copper, coal and zinc mining authorities were contacted and mine water samples from different locations were collected. These mine water samples were utilized to isolate bacteria of the *Thiobacilli* and *Ferrobacilli* groups. The initial experiments were fairly successful since a few bacterial species in some of these waters showed a potential for leaching out copper.

13. ORGANO METALLIC COMPOUNDS

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

The import of diorganotin oxides required for the preparation of stabilizers for plastics is estimated to be 50 T/year valued at Rs. 2 crores. These oxides, when synthesized starting from tin, would cost probably only half. Research on new stabilizer compositions in which tin is partly replaced by titanium will help to reduce the import of tin.

Work on organotin stabilizers for PVC was continued. Starting from dibutyl tin oxide preparation of dibutyltin dilaurate and maleate was standardized. The process is now available for commercial implementation. A cheaper method for making dibutyltin oxide from tin and butyl chloride is under study.

Partial replacement of tin by titanium in stabilizers was achieved. The economics of the process is under study. Some new compounds containing titanium and silicon were also prepared. Their properties are under study.

Di-α-thienyltin diiodide was synthesized for the first time from tin metal and α-iodothiophene. Several chelated α-thienyltin compounds were prepared. The chelating ligands used were salicylaldehyde, benzoylacetone, dibenzoylmethane, acetoacetanilide, 8-hydroxyquinoline and diphenyldithiophosphinic acid. Some benzyltin chelates were also prepared.

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

As part of investigations on the reactivity of chelated ligands, studies on the electrophilic substitution reactions, such as nitration, bromination, iodination and thiocyanation of various beryllium (II) chelates of N-substi-

tuted acetoacetamides were continued. More of new bromo- and nitro-group containing chelates such as bis (3-bromo-2', 4'- dichloroacetoacetanilidato) beryllium (II), bis (3-bromo-2', 5'-dichloroacetoacetanilidato) beryllium (II), bis (3-nitro-3'- chloroacetoacetanilidato) beryllium (II), bis (3-nitro-2', 4'-dichloroacetoacetanilidato) beryllium (II), bis (3-nitro-2', 5'-dichloroacetoacetanilidato) beryllium (II) etc. were isolated and characterized by IR and NMR studies. The mass spectral study of these chelates showed essentially the same break-down patterns and ion rearrangement reactions which were noted in the case of other previously reported similar chelates. The beryllium chelates of 2-chloro-3-chloro-4-chloro-2, 4-dichloro-2, 5-dichloro-acetoacetanilides and also of 2-methoxy-, 4-methoxy- and 2, 5-dimethoxy-acetoacetanilides underwent iodo-and thiocyano-substitution reactions when treated N-iodosuccinimide in chloroform and thiocyanogen in 1,2-dichloromethane. New chelates were obtained containing iodo- or thiocyano- group, as expected, at the central carbon atom in the chelate ring as revealed by IR and NMR studies.

In addition to bromination and nitration, iodination reactions of metal complexes of *o*-vanillin were studied.

The infrared spectral studies on the acylated compounds derived from copper (II) and beryllium (II) chelates of 5-nitro-β-resorcylaldoxime showed that the acetylation occurred at oxime hydroxyl group in preference to the phenolic hydroxyl group.

Ligand exchange-reactions of some rare earth (Nd, Pr, Sm, La) and other metal (Pd, Mo, Cu, Ni, Co) chelates of 2-hydroxy- 1-naphthaldehyde were investigated. These chelates were found to undergo ligand replacement with stronger chelating ligands such as 1, 3-diamino-2-hydroxy propanol-N, N, N', N'-tetraacetic acid.

The condensation reactions between diaminophenol and a dicarbonyl compound (terephthaldehyde or glyoxal) were carried out and selectivity of these polymeric products was examined. However, these were found to be not selective in their reactions with metal ions. The adsorption capacities of these polymers were determined in a glass column (30 cm×2 cm) filled with the dry polymer. The metal containing solution was first passed through the column (0.5-0.3 ml/min.) and after rinsing the column with distilled water, the metal ion was eluted later with an acid solution. About 80% of uranium from solution was retained on the column packed with the polymer obtained from diaminophenol and glyoxal. Other metals found to be adsorbed on the column were Ni(II) -80%, Cu(II) -30% and Co(II) -15-20%. In the case of the polymer from diaminophenol and terephthaldehyde, Ni(II)-6%, Cu(II) -39% were found to be retained on the column.

14. SOLID STATE MATERIALS

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

Potentiometers are vital components of all electronic systems and assemblies. With the development of integrated circuits, the need for increased performance from other passive components has been demonstrated. Thus the demand for smaller sized high reliability components like trimpotentiometers is expected to increase substantially. A new technology is attempted to be developed based on the properties of tin oxide degenerate semiconductors. The latter have already been proven (in fixed resistors) to have the following advantages: very low and consistent resistor noise level, imperviousness to moisture, wide operating temperature range, negligible voltage coefficient of resistance, excellent high frequency performance and tight design tolerance.

The Bhabha Committee report had predicted a total demand of Rs. 3 crores worth of potentiometers of all types by 1975.

A rotating annular sector furnace was fabricated and was used to deposit tin oxide tracks on ceramic substrates. This will enable deposition of 50 to 100 tracks at a time.

Electrical contacts are vital part of potentiometers and ohmic contacts are indispensable for the successful operation of these potentiometers. Tests were carried out using a Tektronix oscilloscope to record the I-V characteristics photographically. A linear I-V characteristic contact was obtained. A surface temperature monitor was developed specifically for obtaining precise temperature information on the heated substrate surface. This will enable close control to be maintained on the tin oxide deposits on the substrates.

14.2 *Photoconductive materials* : (AB-5/72)

Electrophotographic materials find predominant use in the reproduction of images by electrostatic charge processes. The investigation of new materials is useful as the material used at present, amorphous selenium, has some drawbacks like poor sensitivity to incandescent light sources and compartively slow speed of response.

A new technique for the preparation of large area photo conducting layers of CdS by a chemical deposition method was reported earlier. It was possible to charge these layers by the application of high voltage and to store this charge for some time.

Further experiments are planned to study the decay rate and the strength of induced charge in order to assess its suitability for embodiment in a process similar to Xerography. Besides, some basic data on these

amorphous layers is also being collected with a view to understanding the behaviour of these materials.

During the period under review, a new photothermoelectric voltage measurement system was constructed and measurements of photothermoelectric power with a view to determine carrier mobility in the chemically deposited CdS layers are in progress. A new piece of equipment designed and built in the laboratory is the photo Hall effect apparatus which features an all metal cryostat with a provision for intensifying the magnetic field in the cryostat by bringing the pole pieces as close as 1.5 cms. It also features an offset-voltage-nulling bridge with a sensitivity of a few micro volts. These innovation are expected to help in the study of low mobility materials.

14.3 *Thin film sensors, resistors, etc.* : (AB-9/62)

In continuation of the work reported earlier preparative conditions for sensors with varying resistance but having high stability were standardized. Resistance varied from 15 K Ω to 50 M Ω and TCR was in the range -2.0 to-3·0%/°.

Several samples of metal film resistors were prepared and the properties such as resistance, TCR and stability were studied. Almost all the resistors had the value of TCR between -0.04 to-0.06. In some cases the extreme value of TCR was in the range -0.1 to + 0.1. Value of resistance varied from 20 ohms to 200 K. ohms. For accelerated stabilization of metal film resistors different methods were adopted. Arrangements were also made for the measurement of the above properties of the resistors on load.

14.4 *Ferroelectric materials* : (AB-2/71)

The project is aimed at developing suitable ceramics which can be used in phonograph cartridges, ultrasonic transducers, wave filters etc.

A few compositions of modified PZT (lead titanatezirconate) were reported earlier. A data sheet for a typical material is given below.

Density (10^3 kg/m^3)	7.5
Curie temperature (°C)	280
Relative Dielectric Constant, $\varepsilon_{33} / \varepsilon_0$	1800
Dielectric Dissipation Factor tan δX 10^{-2}	2.6
Coupling Factor k_P	0.50
Mechanical Quality Factor, Q_M	97
Piezoelectric Charge Constant,	

$d_{31} \times 10^{-12}$ C/N	— 115
Piezoelectric Voltage Constant, $g_{31} \times 10^{-3}$ VM/N	—10.9
Frequency Constant, Hzm, N*p*	2100

The strips of the ferroelectric material are being evaluated for their performance.

14.5 *Thick film materials* : (AB-75/71)

Thick film passive elements like conductors, resistors, and capacitors are widely used in hybrid microelectric circuits along with active devices. Their applications are on the increasing trend in the manufacture of integrated circuits in television, radio and other fields in the electronic industry.

Silver paste of different types required for ceramic/mica capacitors were formulated. M/s. Bharat Electronics Limited, Bangalore, have evaluated these samples and found them to be satisfactory. Large scale trials are proposed.

A special type of silver paste was developed which can be used in gold electroplating of printed circuits. The product was evaluated by a local firm engaged in making printed circuits on a large scale and was found satisfactory. The process for the preparation of silver paste for mica capacitors has been offered to industry. It is proposed to complete the work on different types of silver paste for various commercial applications.

Work on steatite was initiated. This is required for substrata on which the passive elements are screen printed and are studied for their electrical properties.

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

High-permeability ferrities are annually imported especially in the form of pot-cores to the tune of not less than a crore of rupees. These are used in tele-communication extra-high tension transformers, low loss filters, television sets, etc.

The preparation of the ferrite powder which on pressing into toroids and sintering gave satisfactory magnetic properties was reported earlier.

It was found that the toroids invariably had some cracks in the interior though outwardly they looked satisfactory. It was necessary to press toroids without such discontinuities before pressing complicated shapes such as pot-cores. This was done with the help of the Department of Metallurgy, Engineering College, Poona and toroids of satisfactory specifications and without any cracks were successfully prepared.

The reproducibility in preparing such crack-free toroids is being checked.

14.7 *Liquid crystal display devices (LCD)*: (AB-120/73)

Because of their low cost, low power consumption (<1 μw), low operating voltages (<6 volts a.c./d.c.) and currents (<10μ amps.) and excellent readability even in sunlight, LCDs are preferred for many applications such as digital indicators in watches, calculators, computers, meters, advertising displays, aircraft cock-pits, and are likely to enter in TV. It is planned to construct a liquid crystal digital display using imported room temperature liquid crystal materials.

During the period under review, the literature survey on the construction of various types of LCD cells, organic materials used in them, the physical properties of these materials, and the driving circuitry used for LCD for various electronic applications was completed. For the study of mesomorphic behaviour of the liquid crystals at various temperatures, an universal microscope stage capable of varying its temperature from -100° to 400° was designed, fabricated and tested.

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

In recent years studies of semiconducting compounds especially in thin film form have assumed importance because of their practical applications in various electrical and other devices. In continuation of the structural studies reported earlier of these films, further investigations were made on various intermetallic compounds, chalcogenides, oxide films, especially with respect to substrate temperatures which have a dominant role in determining not only the phase changes but also their physical and other properties. Indium arsenide films showed the normal cubic (ZnS type) structure growing epitaxially on (100), (110) and (111) faces of sodium chloride crystals but occasionally accompanied with an hcp phase Cobalt telluride films (NiAs type hexagonal structure) were formed at about 300-350°, but at a higher temperature say about 400° the deposits were cubic (a=6.04 Å). This phase also grew epitaxially on all the rocksalt faces. Vapour phase deposits of MnSe developed a cubic structure (a=5.48 Å, ∝—MnSe) at about 350°, but at a lower temperature it was amorphous. MnS developed, on the other hand, a cubic structure (a=4.58 Å) and hexagonal structure at higher substrate temperature on the rocksalt faces. MnTe films having hexagonal structure at about 350°, however, developed a cubic phase (a=5.92 Å) at about 400°. All phases developed appropriate orientations on the different faces of NaCl at higher substrate temperature. Nickel sulphide also showed peculiar phase changes with the substrate temperature. The bulk having normal millerite structure (∝—NiS, hexagonal) showed a phase change to ZnS type of structure (a=5.46 Å) along with an hcp form between 150-350°. At higher substrate temperature a phase change of nickel sulphide having spinel type of structure was found. All these phases, however, grew epitaxially with appropriate orientations at suitable substrate temperatures. Various other compounds such as indium

sulphide, copper sulphide, thallium sulphide, indium oxide and bismuth oxide were also studied in details for their phase changes, orientation, relationship and crystal growth process at different substrate temperatures.

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

In continuation of studies reported earlier on dielectric and optical properties of the evaporated films further investigations were made on various oxides such as praseodymium oxide, dysprodium oxide and Sb_2O_3 at room as well as higher temperatures. Praseodymium oxide films which were amorphous at room or higher substrate temperatures were found to have dielectric constant which was thickness independent when the film thickness was higher than, say, about 1000 Å, Measurements of tan δ showed a minimum at a frequency ranging between 100 to 10_5 H_z depending on film thickness. Tan δ (minium) was also found to shift with the increase of temperature to the higher temperature side following the model previously suggested for ZnS films. Capacitance also became invariant with frequency at low temperature regions. The breakdown field was about 3 meV/cm. Refractive index of the deposited films varied from 1.56 to 1.8 and absorption coefficient was very low about 0-0.1 in the visible region. Sb_2O_3 film also behaved in a similar manner except that loss factor was indeed very low and less than 0.005 and independent of film thickness. TCR of this capacitor was less than 200 ppm/°K and capacitance was practically invariant with frequency (100 H_z to 10_5 Hz). Breakdown field was about 0.1 meV/cm and obeyed the Forlani-Minnaja relation. Refractive index was found to vary from 1.9 to 1.98 in the visible region and the absorption coeffiicient was very low indeed. Similar a.c. behaviour was also studied for Dy_3O_3 films and the variation of tan ∂ and capacitance with frequency and temperature were similar to those observed for praseodymium and Sb_2O_3 films. Forlani-Minnaja law was also found to be valid for these films. It was also found that the conduction in the dielectric films was space charge limited (SCL).

Investigations are under way for the semiconducting and other properties of organic semiconductors such as copper phthalocyanine, indanthrone, violanthrone and hydroxyviolanthrone.

14.10 *Ternary semiconducting compounds* : (B-2.10/72)

Thin films of stoichiometric $CdSnAs_2$ (a ternary semiconductor belonging to the chalcopyrite family) were prepared and characterized by X-ray diffraction, electron diffraction, optical and electrical properties. These films when deposited on cold substrata were amorphous but developed one degree orientation with (112) plane of the chalcopyrite structure corresponding to (111) of zinc blende structure parallel to the glass substrate after baking at 350° in N_2 or Ar atmosphere.

An examination of the I-V characteristics with different electrodes showed

that with copper, indium, silver and aluminium, ohmic contacts were obtained while with gold it exhibited non-linearity. The resistance values of the films were highly dependent on baking conditions and varied from 10^5—10^8 ohms. Activation energy was calculated from the linear portion of the log ρ Vs. 1/T curve and found to be varying from 0.23 to 0.6 ev. depending on several deposition parameters. Thermoelectricity measurements have revealed the samples to be n-type. Infrared transmission studies of the films have shown that absorption edge varied from 2.5 to 4.0μ.

Several of the above studies indicate the usefulness of these semiconducting compounds for several applications.

$CuGaS_2$, $CuInS_2$ and $CuGa1-\times INS_x$ which are members of I-III-VI_2 series have been successfully prepared. Single crystals of these compounds have been grown by chemical transport technique using iodine as a transporting agent. Electrical and optical properties like electrical conductivity thermoelectricity, Hall effect, optical absorption, etc., have been carried out. The resistivity of $CuGaS_2$ crystals is of the order of 10^7-10^8 ohm-cm. However, annealing under sulphur pressure at 700° for 3 days produced p-type materials and resistivity reduced to 1-10 ohm-cm.

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

Study of the magnetic properties of the amorphous transition metal oxides/sulphides were undertaken to investigate experimentally the consequences of breakdown of the Neel's model. Study of amorphous gamma iron oxide revealed the absence of regular crystalline structure, however, it had interesting magnetic properties. Similar studies were carried out on iron-cobalt oxide mixture. When cobalt was varied from 1-25%, the composition remained magnetic and slowly became non-magnetic and at 35% cobalt it was completely non-magnetic.

It is proposed to study the Mg-Fe and Ni-Fe oxide systems in amorphous state and correlate the results thus obtained.

Dielectric and optical properties of several oxides and fluorides were also studied in details for the capacitance loss factor with varying frequency at different temperatures. Corresponding refractive indices of these compounds at different wavelengths were determined.

Investigation on the semiconducting properties of several chalcogenide, oxide, etc. films were carried out. It was observed that semiconducting behaviour of bismuth oxide films deposited as such and also after oxidation behaved quite differently thus indicating that the nature of the films was different. Activation energy for films deposited from silica boat were about 2.7 eV whereas oxidized films had 1.2 eV. Hall coefficient, resistivity, mobility, etc. were also measured for the films prepared by direct evaporation. For all these

films mobility followed T^{-1} law. It was observed that the oxidized films which were transparent but on vacuum at about 400° became blackish and tend to have activation energy similar to the films obtained by direct evaporation. This suggests that the vacuum treated films were the dissociated product of Bi_2O_3 the optical behaviour of which also confirmed the above view.

Investigations on vacuum deposited As_2S_3 films showed that these were p-type. Refractive index was found to increase with increasing wavelength whereas k decreased. From the absorption coefficients optical energy band was found to be about 2.5 eV. These samples were photo conducting having a trap depth of about 0.6 eV. Bi_2S_3 films were found to be n-type with activation energy about 0.5 eV depending on film thickness. Thermoelectric power was found to vary from 50 to 100 μ V/°C. R_H was, however, very low. Refractive index was found to vary from 3-3.5 and k 0.1 to 0.7 with increasing wavelength in the visible region. Optical energy band gap estimated from the absorption coefficient with the photo energy was found to be about 1.0 eV for thinner films.

15. INSTRUMENTS TECHNOLOGY

15.1 *Electron Spin Resonance Spectrometer* : (ATT-64/68)

A prototype spectrometer was assembled and tested. Solid DPPH sample was used to analyze the performance. It was decided to carry out modification in the console and magnet to improve the performance and signal to noise ratio. New cavity and sweep circuits are under development.

15.2 *Solid State Recorder* : (ATT-195/72)

The prototype was successfully completed and tested with various analytical equipments. Response time was improved and the recorder now meets the following specifications:

Sensitivity — 1 mV	—100 mV in 7 steps
Response time	— 0.5 sec. for F.S.D.
Input Impedance	— Off Null 50K at Null more than 1 M

Three more recorders are being constructed for assessing reliability and reproducibility.

15.3 *UV Visible Spectrophotometer* : (ATT-173/71)

A majority of mechanical mounts and stands were fabricated for the various mirrors. The slit mechanism is under fabrication and electronic assemblies are being planned and designed. A working prototype model will be ready by next year.

15.4 *Solid State Chopper* : (ATT-221/73)

A substitute for electromechanical chopper used for lmV recorders, MV/I converters, and precision temperature controllers was fully developed and tested. The know-how will be made available soon.

16. TISSUE CULTURE STUDIES

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

This project includes basic studies on the establishment, differentiation and somatic hybridization of plant cells and the production of plantlets of virus-free sugarcane and hybrid plants of agricultural importance.

The high yielding sugarcane strain CO-740 grown in Maharashtra is infected with sugarcane mosaic virus. Over 1000 plants were raised by tissue culture which were free from symptoms of mosaic virus. These plants are now being grown at the Nimbkar Agricultural Research Institute, Phaltan, for obtaining enough stock for large scale field trials.

Methods were also standardized for obtaining sugarcane variety CO-419 from apical meristems. Plantlets were also obtained from callus cultures of CO-740 and CO-419. Some of these plants appeared to be mutants. A screening programme has been undertaken to obtain non-flowering sugarcane which will be of agricultural importance.

About 250 plants of high yielding hybrid cabbage were obtained from two seeds and further scaling up is in progress for large scale field trials. Methods were also standardized for obtaining plantlets from several different strains of wheat, and these are being screened for agriculturally useful mutants.

Methods were standardized for obtaining protoplasts from ten different plants. Further work on obtaining protoplasts from other plant cells and for standardizing the conditions for maximum yield of protoplasts is in progres. Work on the regeneration of protoplasts and cloning of plant cells and on somatic hybridization of protoplasts is being continued.

A large number of compounds isolated from pollen in the Natural Products Division was assayed for effect on the growth of callus tissues and for other physiological effects such as altering the sex ratio of cucumber plants. A few promising compounds are being further investigated.

During the course of work on acylphosphatase over twenty different inhibitors of proteolytic enzymes were found to be present in crude extracts of *Vigna catjang*. These included inhibitors of subtilisin, papain, trypsin and chymotrypsin. Such a large number of different inhibitors from one tissue were not hitherto reported. The inhibitors were found specific for the corresponding proteolytic enzymes. Eight of these inhibitors were purified and

three were obtained in homogeneous form and their molecular weights, isoelectric points, stability and stoichiometry were studied. A papain inhibitor was found in all plants and tissue cultures which were examined. Inhibitors of subtilisin and trypsin occurring in some of the plant tissues were also examined. The papain inhibitor content of maize roots was three times higher than that of the callus from which the roots were derived.

The effect of these inhibitors on the growth and differentiation of the tissue culture is being examined.

16.2 *Insect Tissue Culture* : (AB-96.2/72)

A screening programme for antiviral and antitumour compounds was undertaken (in collaboration with Virus Research Centre, Poona). Methods are being standardized for the assay of Sindbis virus and interferon. The cells isolated from *Corcyra cephalonica* earlier did not multiply *in vitro* after a few subculturing. The growth of cells from potato tuber moth (*Gnorimoschema operculla*) and red cotton bug (*Dysdercus cingulatus*) *in vitro* is being examined.

17. ENZYME CHEMISTRY AND TECHNOLOGY

17.1 *Matrix—Bound Enzymes* : (SP-74/73) (Formerly AB-97/72)

The microbial enzyme penicilln acylase (penicillin amidohydrolase EC 3.5.1.11) is used industrially to hydrolyse benzylpenicillin and other natural penicillins to 6-aminopenicillanic acid, a key intermediate in the production of semisynthetic penicillins like Ampicillin.

Two novel systems of immobilised penicillin acylase were developed. Under this sponsored scheme scale-up studies on one of the systems are in progress.

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

A bacterial phytase which is specific for inositol polyphosphates and requires calcium for its activity was obtained in homogeneous form. The amino acid composition of the purified enzyme was determined. The use of the enzyme for the rapid determination of inositol polyphosphates in seeds and other materials was standardized. The pathway of hydrolysis of inositol hexaphosphate involved triphosphate which was not cleaved by periodate. The identity of the monophosphate which is the final product of action of phytase is being studied.

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

A specific acyl phosphatase was obtained in homogeneous form from *Vigna catjang*. None of the acylphosphates which occur in plant materials, such as carbamyl phosphate and 1, 3-diphosphoglyceric acid, was hydrolyzed

by the enzyme. Since acetyl phosphate is not known to occur in plants, the function of this enzyme, which is specific for acetyl phosphate, is not known. Its molecular weight was determined by ultracentrifugation as well as by chromatography on Sephadex and acrylamide gel in the presence of sodium dodecyl sulphate and was found to be 6000—7000. This is one of the smallest known enzymes.

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

A. fischeri nitrite reductase does not dissociate in the presence of 6M guanidine HCl or 6M urea. The enzyme however splits into two physically indistinguishable subunits upon treatment with 6M guanidine HCl in combination with 1% 2-mercaptoethanol or 1% sodium dodecyl sulphate and 1% 2-mercaptoethanol.

In both the systems the subunit molecular weight was found to be approximately 39000. No peak corresponding to native enzyme was observed suggesting that the dissociation of the enzyme was complete. These results together with the data obtained earlier on sulfhydryl content indicated that *A. fischeri* nitrite reductase was composed of two subunits of equivalent size which are covalently bonded by a disulphide bridge. The amino acid composition of the enzyme was determined.

17.5 *Plant Protease* : (B-12.9/72)

An acid protease from germinated matki (cymopsis) was isolated and purified to homogeneity but the yield of pure enzyme was quite small. Attempts at different methods of purification did not very much significantly change the yield. Further work has been discontinued.

18. FERMENTATION PROCESSES

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

During the period under report 125 new cultures were added to the National Collection of Industrial Microorganisms. The collection now contains over 1700 nonpathogenic yeasts, bacteria and fungi. They are supplied free of charge to research institutions and industries. 779 cultures were sent to different institutions. 40 compounds synthesized in the laboratory were tested for their antibacterial and antifungal activity. The isolation of cultures obtained from different mine waters for the project 'Bacterial Leaching of Ores' was undertaken.

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

As indicated in an earlier report, some more experiments were carried our for mutation of fungus spores with a view to obtain better yielding strains. They did not yield the desired result. Since preliminary cost calculations based

on the analyses of some imported as well as of local amylase samples showed that the laboratory cultures need quite extensive improvement. The project has been suspended for the present.

18.3 *Cellulase* : (AB-67.2/68)

18.3.1 *Protein food from cellulosic plant materials*

A CSIR grant under the Silver Jubilee Scheme has been made to this laboratory, CFTRI, Mysore and Professor G. N. Ramachandran, Indian Institute of Science, Bangalore, for a joint programme on the utilization of cellulosic materials, especially for their conversion into protein food for use either by cattle or by human beings. A screening programme for high cellulase producing microorganisms has been undertaken.

A few cultures which produced very high cellulase activity (both C_x and C_1) are being studied more intensively. Preliminary work and purification of the enzyme from one of the strains was undertaken. Methods for increasing the yield of enzyme from a *T. viridae* strain are also being standardized. One culture which growed rapidly on cellulose without the production of high cellulase activity was tested and found to be non-toxic in preliminary trials. Cellulase is also being produced routinely for work on the project on tissue culture for the preparation of plant protoplasts.

18.3.2 *Studies on cellulases*

The main objective of the project is to make use of cellulases of microbial origin for conversion of cellulosic material into food especially in the form of protein.

Submerged fermentation: Growth and sporulation characteristics of *Trichoderma* varied with the media were used for inoculum development. This in turn might influence the subsequent performance of the culture and also affect the behaviour of the culture in the long run. A more favourable medium for the growth of the inoculum than being previously used was established.

Studies regarding the effects of incorporating several trace metals and some constituents to cellulose powder media resulted in the increase of all the three activities namely C_x, filter paper and cotton-degrading activities to about 1.5 to 2 fold as compared to cellulose powder medium. These were the highest activities obtained so far for this culture in this laboratory. Besides the above, stimulation of C_x activity in particular was observed by anti-sporulating agents. Further studies on these aspects and to establish the optimum conditions to get uniformly high yields of the enzyme are in progress.

Surface fermentation: Experiments to produce cellulase by surface fermentation by some promising new fungal isolates in combinations showed that the fermentation period for maximum activity could be shortened by this procedure but the activities per ml. could not be increased further.

Of the many new strains tested, the fungus belonging to *Polystictus* species grew well and digested the cellulose powder completely.

A cellulase which exhibited optimal activity at a lower pH range was obtained from an isolate belonging to *Basidiomycetes* (tentatively identified as *Sclerotium rolfsii*) species. Conditions for obtaining maximum cellulase production were determined. Work on the purification of cellulase and study of the component nature and characteristics of the cellulase system from this isolate is in progress.

18.4 *Retardation of loss of ammonia applied as fertilizer in soil* : (AB-67.5/70)

Ammonia fertilizers applied to the soil are oxidized microbiologically to nitrite and then nitrate which are partly lost by leaching or by denitrification. The ammonia oxidizing organisms were found difficult to maintain in pure culture owing to their failure to survive on subculture. The project has been undertaken to find cheap inhibitors which will retard or prevent ammonia oxidation in soil.

Methods were standardized for the isolation of ammonia and nitrite oxidizing organisms. 15 cultures of *Nitrosomonas* and *Nitrobacter* were isolated in pure form. Out of 32 cultures which were isolated earlier 26 have been maintained *in vitro* without diminution in vigour on subcultures for two years.

19. PROCESS DEVELOPMENT AND CHEMICAL ENGINEERING STUDIES

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

This is an important chemical finding use in the dyestuff industry and in the production of tetryl.

A new catalyst was developed for the vapour-phase condensation of methanol and aniline to predominantly dimethylaniline. The investigation was conducted in a single tube reactor using an oil circulating unit, and the process conditions were standardized to get over 90% dimethylaniline in the product. The design of a 300 TPA plant has now been undertaken by the NCL and the process will soon be commercially exploited by the sponsor.

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

The indigenous production of this important intermediate is at present based on a batch process. A continuous process for the chemical by vapour phase catalytic hydrogenation of *p*-nitrotoluene is being developed under this sponsored scheme.

The work on pilot plant was completed after testing the activity of the

catalyst for a period of 1,000 hours. The same catalyst was tested for its suitability to *o*-nitrotoluene reduction, and was found satisfactory. The designs for a 300 TPA plant have been submitted to the sponsor.

19.3 *Alkylation of naphthalene and aromatic hydrocarbons :* (AB-92/71)

Studies on propylation of naphthalene using sulphuric acid as catalyst were continued. The work indicated that conversions upto 70% of naphthalene to mono-isopropyl naphthalene could be obtained under certain conditions.

This was confirmed by carrying out some larger laboratory-scale batch runs. Process improvements for removal of sulphur compounds formed during reaction and unreacted naphthalene from the product are being looked into.

Laboratory scale work on propylation of toluene using sulphuric acid as catalyst was carried out to select suitable process variables for obtaining satisfactory conversion of toluene to cymene. A series of over 20 laboratory scale runs were conducted and the composition of the product mixtures obtained was determined by GLC analysis. The product mixtures were separated into different fractions to recover toluene, cymenes and higher propylated products. The cymenes mixture (B.P. 172-175°) thus recovered is being used in the hydroperoxidation studies now in progress.

Work on utilization of the bye-product, higher propylated toluenes will be considered later.

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

19.4.1 *Studies in gas-liquid reactions* : A detailed analysis of gas-liquid reactions has been made and a mathematical model which explains the diffusion-reaction behaviour of hetrogeneous systems has been developed. Gas-solid catalytic reactions represent a limiting situation of this genera, model. The effectiveness factor for gas-liquid reactions are represented by regions which, under appropriate limiting conditions, degenerate to a line for gas-solid catalytic reactions. The model is now being extended to a general p^{th} order reaction developed by Aris for gas-solid reactions. The results obtained so far show that the generalized approach can lead to a unified diffusion-reaction theory for all hetrogeneous systems.

19.4.2 *Simulation of a catalytic reactor*: A procedure has been developed for the simulation of a fixed bed catalytic reactor based on a semi-two-dimensional approach to the modelling of fixed beds. The results obtained are very close to those predicted from rigorous but time-consuming two-dimensional models. The semi-two-dimensional model is a significant improvement over the simple one-dimensional model and errs on the conservative side so that designs based on this model can be considered to be safe.

19.4.3 *Measurement of catalyst properties under reaction conditions:* Several experiments were carried out on the standardization of catalysts for the isomerization of butenes to isobutylene. Important properties of the catalyst have been determined based on which the best catalyst will be selected. Measurement of these properties under reaction conditions will then be undertaken. The experimental assembly for these studies is ready.

19.4.4 *Modelling of catalytic reactions:* A reactor assembly has been set up for studying the kinetics of oxidation reactions. This assembly utilizes a flow system with on-line analysis of the gas-product. The oxidation of o-xylene to phthalic anhydride is being studied in this assembly. This reaction has been identified as being of great industrial importance for which indigenous technology should be made available. After the kinetics of the reaction have been worked out, complete modelling and simulation studies will be carried out.

Studies on the ammoxidation of propylene are in progress. Detailed and precise analytical procedures have been worked out and a micro-reactor assembly is being set up together with a stirred reactor. The object of this investigation is to try some new catalysts and study the mechanism of ammoxidation.

19.4.5 *Solid-solid reactions:* Very little work has been reported in the literature on the modelling of solid-solid reactions. A detailed programme of research on solid-solid reactions has therefore been undertaken not only with the object of studying the kinetics of this reaction but also of exploring in detail the influence of diffusion. To start with, the addition product formed by reacting maleic anhydride with aminophenols is being studied in a differential thermal analysis unit using particles of the two solids. The studies will be extended to pellets to examine the effect of diffusion. Theoretical analysis has also been carried out and equations have been developed for the effectiveness factor. The object of these theoretical studies is to see if the diffusion-reaction behaviour of solid-solid reactions can also be included in the general approach mentioned above under 19.4.1

20. FOLLOW-UP ACTIONS

20.1 *Monochlorobenzene (MCB)*

Based on the know-how and process design supplied, a 4500 TPA commercial plant was erected at HOC, Panvel, by R. L. Dalal and Co. NCL scientists visited the plant several times during the erection stage and provided necessary suggestions and advice on erection, layout and related matters. After taking several trial runs, necessary modifications were made in the plant. Further trial runs and training of HOC personnel are in progress.

20.2 *Hexachloroethane (HCE)*

Process design for a 300 TPA plant of HCE was supplied to the licen-

cee. A 100 TPA plant was erected at the site of the firm and operated to test the process.

20.3 *Chloromethanes*

As per the designs supplied, a 4500 TPA chloromethane reactor was fabricated abroad and erected at M/S Standard Alkali, Thana. Erection of the whole plant was completed. Continuous assistance at site and advice are being provided and commissioning of the plant is in progress.

20.4 *Monoethylaniline (MEA)*

The process was demonstrated to the licencee on a 15 kg/hr scale. Complete process design for a 300 TPA commercial plant was prepared and given to the licencee-firm.

20.5 *Ethylenediamine (EDA)*

The process was demonstrated to representatives of the firm who have purchased this technology, in the presence of a representative of their project engineers. Full process package with all necessary details was prepared and sent to the party.

20.6 *Vitamin C*

Assistance was rendered to Hindustan Antibiotics Ltd., Pimpri, in connection with large scale runs on the manufacture of vitamin C by the NCL process.

21. NEW ANALYTICAL METHODS

Basic and applied research in analytical chemistry is being pursued with the objective of keeping abreast of developments in this field and of evolving new analytical techniques and procedures for the process development groups of the laboratory.

21.1 *Aqueous chemistry of transition metal chelates of polyaminocarboxylic acids*

Diethylenetriamine pentaacetic acid (DTPA) is known to form more stable chelates than EDTA. Spectrophotometric studies of DTPA chelates of lead and bismuth at various hydrogen ion concentrations showed that at pH 3.6 the two chelates had widely separated absorption maxima. The Pb-DTPA chelate had the absorption maximum at 244 mμ ($\varepsilon = 10.7 \times 10^3$) while the Bi-DTPA chelate showed at 277 mμ ($\varepsilon = 9.2 \times 10^3$) and both the chelates obeyed Beer's law in the wavelength region of interest. Analytical method was established for the simultaneous estimation of these two metals based on the above phenomenon. Synthetic mixtures of Pb and Bi containing, in addition the commonly associated elements, Sn (II), Sn(IV) and Sb (III) indicated the non-interference of the latter elements. The validity of

this method was also checked by estimation of bismuth in pig lead samples and the results were in good agreement with, and quite comparable to, the standard thiourea method. The DTPA method of estimation of bisumth had the added advantage over the EDTA method of non-interference from the associated elements, Sn(II), Sn(IV) and Sb(III). The formation constants of Pb/Bi-DTPA chelates were determined by ion-exchange and spectrophotometric method.

21.2 *Estimation of thionyl chloride and HCl in endosulfan reaction mixture*

A method was developed for the estimation of thionylchloride and HCl in the endosulfan reaction mixture.

21.3 *Water hardness monitoring solution*

An indicator formulation was developed for continuous monitoring of hardness in water samples. The formulation which will be useful in water softening plants indicated the exact degree of hardness from 1-100 ppm in a matter of seconds. This composition is superior to the soap solution testing method in many respects.

21.4 *Microanalytical methods*

Work on simultaneous microdetermination of chlorine and bromine in organic compounds reported last year was successfully completed.

21.5 *Development of new analytical methods for the applied projects in NCL*

Suitable methods of analysis were developed for the acrylates project using NMR for the compositions obtained in the processes for methyl acrylate and butyl acrylate. A number of analyses were carried out for the study of partition coefficient and reaction kinetics.

A UV method was developed for the estimation of HEX (hexachlorocyclopentadiene) in the products obtained at different stages in the process for the production of Endosulfan. A number of analyses were carried out using this method.

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. Chemical examination of complicated samples from industries are also entertained to a limited extent.

Analytical method was developed and standardized, for the estimation of propionaldehyde (PPM levels) in the propylene oxide project.

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

The group is also concerned with the development of new analytical methods for 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, 2408 samples were analysed for various elements (2292) and functional groups (116).

22.1.3 *Mass Spectrometry :* During the period under review 1452 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 programme.

Number of samples studied

NMR	3412
IR	2344
VPC/GLC	2100
X-ray powder patterns	309
Determination of surface area by BET technique	7
Thermogravimetric analysis (DTA, TGA, TG, etc.)	48

22.2 *Instrumentation section*

The section is primarily engaged in maintenance and upkeep of special type of analytical instruments like IR, UV, NMR spectrometers, visible gas chromatographs, x-ray machines etc., besides normal routine maintenance and repairs of smaller instruments like potentiometers, pH meters, conductivity bridges, furnaces etc.

Total number of jobs and test reports completed during the year were 385.

22.3 *High Pressure Laboratory*

This section primarily provides equipment for carrying out experiments involving high pressure, relating to the various projects in the laboratory.

During the period under reference 324 experiments were carried out both for various staff members of the laboratory and for private parties.

22.4 *Engineering section*

22.4.1 *Mechanical Engineering* : Apart from maintenance of the laboratory's installations, utility services and equipment the following special fabrications were carried out for laboratory and sponsored projects :

(i) Fabrication of one 40 L.M.S. reactor with stirring arrangement and filteration unit for 'Pentachloronitrobenzene' project and its installation with all necessary services.

(ii) Fabrication of special type of horizontal 'Epoxydater' (300 mm. dia. and 900 mm. length) and two condensers with assembly as a unit on a suitable stand for 'propylene oxide' project.

(iii) Assembly and erection of all glass unit for 'Acrylate' project and giving all necessary service conditions.

(iv) Fabrication and installation of 2 Nos. of fume extractors (approx. 950 cf/m. cap.) for general research use in the laboratory.

The total job orders executed by this section during the year were over 3114.

22.4.2 *Civil Engineering* : During the period under review the following constructions were carried out :

(i) Construction of a shed for pump house to accommodate the 40 H. P. pump.

(ii) Construction of a shed for organic synthesis division.

(iii) Construction of a wing for the Division of Technical Services.

In addition to the above, construction work of a new building for stores is in progress.

22.5 *Glass blowing section*

The section primarily undertakes repairs of glass apparatus for various research groups in the laboratory. During the period under report 4447 jobs were completed.

Glass assemblies of various types were fabricated as per specific drawings furnished by the scienstists. About 2305 fabrications of glass assemblies were carried out. Of these, 160 fabrication jobs were related to the sponsored projects. About 6143 standard glass joints were fabricated for internal use.

22.6 *Library*

The library houses about 63,000 accessioned items consisting of books, periodicals, patents, standards and technical reports, etc., in stock. 2659

volumes comprising of (i) bound volumes of periodicals—2000 and (ii) books, patents, standards and technical reports—659 were added during the period under review. 820 journals including annual reports of various institutions were received on continuation basis. Library facilities are extended to readers from Govt. departments, universities, colleges and other research institutions. Library facilities are also made available to the representatives of industry on membership basis. 447 persons from these organisations made use of the library facilities during 1973-74. In addition to the above a monthly current awareness service on 'Pesticides and Agrochemicals' was started from January 1974.

NCL is a Patent Inspection Centre for Indian patents. During the year under review 2000 Indian Patents were received by the library.

22.7 *Division of Technical Services*

Commercial intelligence

Techno-economic information on the following items was collected as a follow-up action of Advisory Panel recommendations.

Trimethylol propane, adhesive for metal to rubber bonding (Chemlok 220, 205 and 402) and hylak polyester resin required for the manufacture of resin treated glass tapes.

A comparative study of landed prices and Indian manufacturer's bulk prices for more than 80 selected chemicals was carried out.

Techno-economic and market data related to Indian chemical industry in general and NCL programme in particular and socio-economic data on Indian States was routinely compiled.

Industrial liaison and co-ordination

Over 3900 enquiries peritaining to NCL projects, general technical enquiries from different individuals, industries and government organisations and starred questions from the Parliament were attended to. 200 applications in chemical and allied industries for industrial licences with foreign collaboration were scrutinized and comments forwarded.

Over 2700 visitors from educational institutions, industries and other organisations were shown round the laboratory. More than 200 enterpreneurs/ parties interested in exploiting know-how available from NCL were attended to and required information in the form of non-technical notes or otherwise was furnished.

Non-technical notes on processes approved by Process Release Committee (PRC) were prepared along with tentative cost estimates. The total number of non-technical notes which are at present available for circulation to interested parties is 86.

Contractual research proposals were prepared for the following projects:

(1) Fumed silica (2) Testing the suitability of Bastar hard woods for the manufacture of rayon grade pulp (3) Butyl and 2-ethyl hexyl acrylates (4) Ethyl and methyl acrylates (5) Vinyl acetate monomer (6) 1-Naphthalene acetic acid (7) β-Naphthol (8) 7-Amino Deacetoxycephalosporanic acid (7-ADCA) and cephalexin (9) Daptazole (10) Matrix-bound penicillin acylase systems (11) Xylit from corn-cobs and (12) Vanillin from lignin sulphonate.

A list of NCL technologies available for export was prepared and circulated to interested parties.

Liaison with CSIR

The following processes were referred to CSIR for assignment to NRDC :

(1) Molecular sieves (2) Sodium hydrosulphite (3) Diethyl-toluamide (4) Reactive dyes (5) Flocculating agents for sugar cane juice clarification (6) 2-amino-5-chlorobenzophenone and 2-methyl-amino-5-chlorobenzophenone (7) Dibutyl tin stabilizers for PVC and (8) Expandable polystyrene beads.

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 1973 and NCST Annual Report 1971-72 was sent.

In connection with the Vth Five Year Plan of NCL data required by the Planning Group of CSIR was sent in prescribed proformae.

Research schemes submitted to CSIR for financial assistance were scrutinized taking into consideration their feasibility and relevance to CSIR's and country's research and industrial programmes. Comments on 150 such schemes were sent to CSIR.

Assistance to States/Regions

For the CSIR Get-together at Madras, problems/projects where NCL could assist were identified after considering the "Report on Survey of Industries in Tamilnadu" prepared by Madras Complex of CSIR.

A note on industries which could be established in Vidharbha region was prepared for Development Corporation of Vidharbha Ltd. A similar note was also prepared for Konkan Development Corporation.

Publicity

NCL Documentary

A 35 mm. coloured documentary film on NCL was prepared under the title "NCL in the service of nation." The film depicts the role played

by chemical industry in the uplift and betterment of the life of our people and has a reference to socio-economic benefits accrued through the manufacture and use of a variety of chemical products. The working of the NCL and its capabilities for undertaking R and D work in chemical technology have been highlighted. The film highlights the achievements of NCL in the establishment of chemical plants on the basis of NCL know-how.

Plastic Age Exhibition

NCL participated in the Plastic Age Exhibition which was held in Poona from 3-7th March 1974. The NCL pavilion highlighted the R & D programme carried out in the Polymer Chemistry Division of NCL.

Research Analysis

Techno-economic feasibility reports were prepared for the items mentioned below for the consideration of the Process Release Committee. (1) Pentachloronitrobenzene (2) 7-dehydrocholesterol (3) Molecular sieves (4) Sodium hydrosulphite (5) Diethyl toluamide (6) Reactive dyes (7) Ethanolamines (8) Colchicine (9) Flocculating agents for sugar cane juice clarification (10) 2-amino-5-chlorobenzophenone and 2-methylamino-5-chlorobenzophenone (11) Silicon tetrachloride (12) Chlorinated paraffin wax (13) Ethyl silicate (14) Dibutyl tin stabilizers for PVC (15) Expandable polystyrene beads (16) Silver paste for mica capacitors and (17) Ferroelectric ceramics for gramophone pick-ups.

In order to facilitate the early release of NCL processes to industry several projects were referred to different project engineering firms for their technical and economic evaluation. DTS assisted the project engineers in the preparation of feasibility reports on (1) Simazine and Atrazine (2) Guaiacol, glyceryl guaiacolate, potassium guaiacol sulphonate, vanillin and thioglycolic acid and (3) Reactive dyes, based on the processes developed at NCL.

Cost estimates for the following items were made (1) Theophylline and caffiene (250 TPA plant) (2) Sandalwood oil by solvent extraction of sandalwood and (3) Recosting for optical whitening agents.

The Efficiency cum Performance audit party of CSIR visited NCL. The enquiries raised by them were attended to and the data required by them was compiled and furnished to them.

Research Planning and Management

The study on (i) Transfer of technology and (ii) Patents was concluded. The findings were summarised in the form of papers.

DTS provided secretarial assistance to the Director, NCL, in technical follow-up work of (i) Science and Technology Plan for Chemical Industry and (ii) Futurology Panel.

With a view to evaluate the projects of the NCL in respect of research programme during the period 1968-73, a study was initiated to analyse the progress made by the institute in commercializing these projects. Projects on which commercial production has been established, projects awaiting production and projects offered to industry are separately listed. Effeorts are being made to study the reasons for non-exploitation of completed projects and projects that were discontinued before completion.

Reports

The following reports were compiled, edited and produced :

(i) Report on Science and Technology Plan for Chemical Industry (Vols. 1 and 2), published by NCST.

(ii) NCL Annual Report 1972-73 and Achievements 1972-73.

(iii) NCL Research Programme 1974-75.

APPENDICES

1. SERVICES RENDERED TO INDUSTRY, RESEARCH INSTITUTES, UNIVERSITIES, ETC.

1.1 *Supply of cultures*

During the year under report, 779 cultures from the National Collection of Industrial Microorganisms (NCIM) were supplied free of charge to various institutions in India and abroad.

1.2 *Analytical Services*

A large number of analyses were carried out on payment for universities, research institutions, Govt. departments, private parties, etc.

Microanalysis	40
NMR	46
IR	57
Mass Spectra	86
Thermogravimetric analysis (Viz. DTA, TGA, TG, etc.)	34
X-ray powder patterns	18
VPC/GLC	7
Ultracentrifugal analysis	2

Technical aid was rendered to about 55 agencies including universities, Government 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 maintenance of instruments and chemical and spectroscopic analytical methods was given to 20 representatives of IIT's, colleges and universities.

High pressure reaction facilities were extended to 6 parties of carrying out 31 experiments.

2. SPONSORED PROJECTS

2.1 *Sponsored projects concluded during* 1973-74

1.	Synthesis of resin for friction material	M/s. Hindustan Ferodo Ltd., Ghatkopar, Bombay-86, AS.
2.	Preparation of l-menthol from citronella oil of Indian origin	M/s. Bhavana Chemicals Ltd., Bombay-1.

3.	Preparation of nitromusk compounds	M/s. Opal Fine Chemicals, Bombay.
4.	Vapour phase reduction of p-nitrotoluene to p-toluidine	M/s. Sudarshan Chemical Industries P. Ltd., Poona.
5.	Fractionation of turpentine (thermodynamic studies)	M/s. J & K Industries Ltd., Srinagar.

2.2 *Sponsored projects undertaken during* 1973-74

1.	Preparation of 7-ADCA and cephalexin	M/s. Hindustan Antibiotics Ltd., Pimpri, Poona-18.
2.	*p*-Cresol	M/s. Hico Products (P) Ltd., Bombay-16.
3.	Preparation of matrix-bound penicillin acylase systems	M/s. Hindustan Antibiotics Ltd., Pimpri, Poona-18.
4.	Papaverine hydrochloride	M/s. Suneeta Aromatics, Indore.
5.	β-Naphthol	M/s. Hindustan Organic Chemicals, Rasayani.
6.	Technical preparation of xylit from corn cobs	M/s. Unichem Laboratories, Ltd., Bombay.

2.3 *Sponsored projects continued from* 1972-73

1.	Acrylic acid/acrylates from acrylonitrile	M/s. Indian Petrochemicals Corporation Ltd., Baroda.
2.	1, 3-Butylene glycol	,, ,,
3.	Catalytic vapour phase epoxidation of olefins	,, ,,
4.	Cationic dyes for acrylic fibres	M/s. Sahyadri Dyestuffs and Chemicals (P) Ltd., Poona.
5.	Composite drug research scheme on Indian medicinal plants	Ministry of Health, Govt. of India, New Delhi.
6.	Manufacture of dimethyl-aniline by continuous process	M/s. Sahyadri Dyestuffs and Chemicals (P) Ltd., Poona.
7.	Flavonoids, tannins, stilbenes, lignans and quinones in some Indian forest trees (PL-480)	U. S. Deptt. of Agriculture, Washington.

8.	Glyceryl- ∝-mono-paraamino-benzoate	M/s. Indian Schering Ltd., Bombay.
9.	Preparation of propylene oxide from propylene	M/s. Indian Petrochemicals Corporation Ltd., Baroda.
10.	Synthesis of potential pharmacologically active substances	M/s. Sarabhai Research Centre, Baroda.

3. TECHNOLOGY TRANSFER

3.1 *Demonstrations :*

Process know-how for the following products was demonstrated :

Process	*Name of the party*
1. Can-lining composition	M/s. Arya Chemical Works, Calcutta.
2. CDV Pigment base	M/s. Vapson Products, Bombay.
3. Ethylenediamine	The Bharat Vijay Mills, Ltd., Kalol.
4. Foundry core binder	M/s. Card-Chem. Industries, Hyderabad.
5. Gum arabic substitute	M/s. Karnatak Adhesives, Bangalore.
6. Microfilters	M/s. Sona Microfilters, Poona.
7. Monoethylaniline	M/s. Atul Products Ltd., Bulsar.
8. Nitrile rubber-medium grade	M/s. Synthetics & Chemicals Ltd., Bombay.
9. Phenyl acetic acid	M/s. Orient Aroma Chemical Industries, P. Ltd., Bombay.

3.2 *Processes leased out during* 1973-74

Process	*Name of the party*
1. Ethylenediamine	The Bharat Vijay Mills Ltd., Kalol.

Process	*Name of Party*
2. Flexible magnets	i) Ferrites and Electronic Components Pvt. Ltd., Balmiki Marg, Lucknow. ii) M/s. V. P. Nijhawan, III-F/5, Lajpatnagar, New Delhi-24.
3. Gaskets from coir pith	M/s. Oberoi Industries, 12/37, Tilak Nagar, New Delhi.
4. Gum arabic substitute	i) M/s. Karnatak Adhesives, 19, Mysore Deviation Rd., Gopalapuram, Bangalore-23. ii) M/s. Bal Krishan Mital, Quarter No. CN-2/I, NRC Colony, Mohone, Kalyan. iii) M/s. Supreme Enterprises, Bhagwan Nivas, Kailash Cinema Chowk, Ludhiana.
5. Microfilters	M/s. Sona Microfilters, Joseph House, Padamjee Park, Poona-1.
6. Phenylacetic acid	M/s. Orient Aroma Chemical Industries, Pvt. Ltd., 27, Chinchbunder Road, Bombay-9.
7. Rubberized cork sheets from waste cork granules	M/s. Cork Products (P) Ltd., 9, Jor Bagh, New Delhi.
8. Vitamin B_6	M/s. Indian Drugs & Pharmaceuticals Ltd., New Delhi.

3.3 *Processes assigned to NRDC during* 1973-74.

1. Molecular sieves
2. Reactive dyes
3. Sodium hydrosulphite
4. Dibutyltin stabilizers for PVC

3.4 *Premia and royalties received by NRDC through NCL processes during the year* 1973-74.

Premia

S. No.	*Name of the process*	*Name of the firm*	*Premium received (Rs.)*
1.	Benzoic acid from crude methyl benzoate	M/s. Indian Petrochemicals Corpn. Ltd., Baroda.	40,000.00
2.	Butyl titanate	M/s. Synthochem, 33-A, Laxmi Nagar, Indore-1.	4,000.00*
3.	Cadmium sulphide photoconductive cells	M/s. Chinoy Electronics, 64, Koregaon Park, Poona-1.	4,500.00*
4.	Dimethylaniline	M/s. Sahyadri Dyestuffs & Chemicals (P) Ltd., 117, Vithalwadi Road, Poona-9.	22,500.00*
5.	Ethylene diamine	The Bharat Vijay Mills Ltd., Kalol, North Gujarat.	25,000.00*
6.	Flexible magnets	M/s. Ferrites & Electronics Components Pvt. Ltd., Lucknow.	1,000.00*
7.	Foundry core binder	M/s. Industrial Chemicals Co., Mangalore.	2,500.00*
8.	Gaskets from coir pith	M/s. Oberoi Industries, 12/37, Tilak Nagar, New Delhi.	5,000.00*
9.	Gum arabic substitute	(i) M/s. Karnatak Adhesives, 19, Mysore Deviation Rd. Gopalapuram, Bangalore-23.	5,000.00
		(ii) Mr. B. K. Mital, Quarter No. CN-2/1, NRC Colony, Mohone, Kalyan.	5,000.00

S. No.	*Name of the process*	*Name of the firm*	*Premium received (Rs.)*
		(iii) M/s. Supreme Enterprises, Bhagwan Niwas, Kailash Cinema Chowk, Ludhiana.	5,000.00
10.	Hard ferrites and flexible magnets	M/s. V. P. Nijhawan, III-F/5, Lajpatnagar, New Delhi-24.	5,000.00
11.	Nicotine sulphate	(i) M/s. Agro Chemical Industries, Parchuru, Dist. Prakasam (AP).	2,000.00
		(ii) Shree Ganesh Tobacco Bye-products Industries Pvt. Ltd., Borsad Dist. Kaira, Gujarat.	2,000.00
12.	Nitrile rubber	M/s. Synthetics & Chemicals Ltd., Bombay-20.	20,000.00*
13.	Phenylacetic acid	M/s. Orient Aroma Chemical Industries Pvt. Ltd., 27, Chinch Bunder Road, Bombay-9.	1,000.00*
14.	Phthalates-dioctyl and dibutyl	M/s. India Carbon Ltd., Bombay through R. L. Dalal & Co, Bombay.	12,500.00*
15.	Radiosonde thermistors	The Bhagyanagar Laboratories, Hyderabad.	10,000.00*
16.	Rubberised cork sheets	M/s. Cork Products (P) Ltd., 9, Jor Bagh, New Delhi.	10,000.00*
17.	Sorbitol	M/s. Maize Products, Ahmedabad.	37,500.00*
18.	Terpineol	M/s. Dujodwala Industries Faridabad.	10,000.00*

S. No.	*Name of the process*	*Name of the firm*	*Premium received (Rs.)*
19.	Vitamin B_6	M/s. Indian Drugs & Pharmaceuticals Ltd., New Delhi.	10,000.00*
		Total	2,39,500.00

* Part payment

Royalties

S. No.	*Name of the process*	*Name of the firm*	*Royalty received (Rs.)*
1.	Anion exchange resins from melamine	M/s. Tulsi Industries, Poona.	54.45
2.	Antipriming compositions	Research, Design & Standards Organisation, M & C, Chittaranjan, (Min. of Railways).	659.50
3.	Can sealing composition	M/s. Arya Chemical Works, Calcutta.	8093.07
4.	CDV Pigment base	M/s. Square Chemicals, Bombay.	30.00
5.	*ter*-Butyl catechol	M/s. Percynic Chemicals, Bombay.	2278.00
6.	Catechol	-do-	1550.75
7.	Ferrites (hard)	M/s. Semiconductors, Ltd., Poona.	2315.94
8.	Hexylresorcinol	M/s. Unichem Laboratories Ltd., Bombay.	42.50
9.	β-Ionone	M/s. Industrial Perfumes, Ltd., Bombay.	16321.93

S. No.	Name of the process	Name of the firm	Royalty received (Rs.)
10.	Nicotine sulphate	M/s. Urvakunj Nicotine Industries, Dharmaj.	18318.00
11.	D. C. Recording Polarograph	M/s. Elico Pvt. Ltd., Hyderabad.	300.00
12.	Polyurethane printing rollers	M/s. Sree Saraswaty Printing Press Ltd., Calcutta.	3616.43
13.	Sachets-hot and cold	(i) M/s. Thermochem Laboratories, Poona.	14.95
		(ii) M/s. Vasant Industrial Corporation, Nagpur.	104.70
14.	Sisal wax	M/s. Aphali Pharmaceuticals Ltd., Ahmednagar.	3.44
15.	Direct reading spectrophotometer/ colorimeter	M/s. Neotronics Corporation, Bombay.	1059.25
16.	Vapour phase Chromatograph	M/s. Associated Instruments Manufacturers (India) Pvt. Ltd., New Delhi.	19000.00
			73762.91
		Total Premia and Royalty	3,13,262.91

4. SEMINARS AND LECTURES

4.1 *Lectures*

The following visiting scientists delivered lectures in the laboratory:

1.	Dr. R. M. Acheson, Deptt. of Biochemistry, Queens College, Oxford, U. K.	Novel reactions of acetylenic esters and new applications of an NMR shift reagent.
2.	Dr. K. H. Froming Dean, Faculty of Pharmacy and Prof. Pharmaceutical Technology, Free Univ. Berlin.	New developments in the field of pharmaceutical technology.

3.	Prof. M. Haake, Philipps University, Marburg, Germany.	Aza-analogous sulphones (sulphoximides) Sigma sulphoranes Methylene iminium salts.
4.	Dr. H. Knozinger, Physikalisch-Chemisches, Institut der Universitat, Munich, West Germany	Catalytic dehydration of alcohols on alumina-an interpretation of *trans*-elimination and *cis*-preference. Double-bond isomerization of olefins on alumina. Spectroscopic studies of surface properties of alumina.
5.	Prof. F. A. Long, Director, Program on Science, Technology & Society, Cornell Univ. 632, Clark Hall, Ithaca, New York.	Science and Technology in the development process.
6.	Dr. M. M. Mehra, Dept. of Chemistry, University of Chicago, USA.	Synthesis of 7-oxa and thia—prostaglandins.
7.	Dr. P. G. Menon, Manager, Catalysis Group, Indian Petrochemicals Corpn. Ltd., Baroda.	Catalysis—its applications in the petrochemical industry.
8.	Dr. L. M. Mulay, Prof. & Chairman, Solid State Science Divn., Pennsylvania State University, U S A.	Amorphous magnetic materials and catalysis.
9.	Prof. P. T. Narasimhan, I. I. T., Kanpur.	Studies on Nuclear spin couplings. Electron spin resonance, Spin Densities, Line widths and internal motions in free radicals. Nuclear quadruple resonance studies on compounds containing Spin 3/2 Nuclei.

10.	Dr. H. Plieninger, Prof. of Organic Chemistry, Heidelburg University, West Germany.	Synthesis of indole derivatives with isoprenoic side chains including precursors or ergot alkaloids biosynthesis. New results of organic reactions at very high pressure (10 k bar) Modern synthetic methods with transition metal compounds.
11.	Dr. J. W. Rowe, Forest Products Laboratory, U. S. Dept. of Agriculture, Madison, Wisconsin, U.S.A.	Recent progress in Naval Stores Chemistry. Research on tree extractives at the Forest Products Laboratory.
12.	Prof. (Mrs.) B. Serafin Technical Univ, Warsaw, Poland.	Some aspects of organoboron Chemistry.
13.	Prof. Y. T. Shah, University of Pittsburg, U.S.A.	Stability in isothermal flow channels.
14.	Prof. M. M. Sharma, Bombay University, Department of Chemical Technology, Bombay.	Some aspects of fluid-fluid reactor design.
15.	Dr. V. Sperling, Czechoslovakia.	Scientific work being carried out in the Institute of Chemical Process Fundamentals of the Czechoslovak Academy of Sciences. Terephthalic acid.
16.	Prof. E. J. Staba Dept. of Pharmacognosy University of Minnesota, U.S.A.	Medicinal plant tissue culture.
17.	Dr. A. V. Subbaratnam, Phoenix Laboratories, Inc. Omaha, Nebraska, U.S.A.	Industrial research in USA and management goals in various areas; difficulties in acquiring know-how and the use and abuse of chemists.
18.	Dr. P. R. Venkatachalam, Indian Institute of Science, Bangalore.	An improved design for tubular reactors with rapidly poisoning catalyst.

19.	Prof. W. Von Philipsborn, Univ. of Zurich, Institute of Organic Chemistry. Switzerland.	Carbon magnetic resonance in structural organic chemistry.
20.	Prof. G. Wegner, University of Mainz, West Germany.	Kinetics of Trioxane polymerization.
		Polymer reactions inside organic crystals.
		Melting and crystallization of copolymers.

4.2 The following NCL scientists delivered lectures at various institutes, universities, colleges, etc.

Name of the Scientist	*Subject*	*Venue*
1. Dr. A. Goswami	Physics of thin films.	Poona University, Poona.
	Semiconducting, structural, optical and dielectric properties of thin films.	Sardar Patel University. Anand.
	Electron miscroscopy in Chemistry	Poona University, Poona.
	Structure and Physics of thin films.	Dibrugarh University, Dibrugarh, Assam.
	Studies on vacuum-deposited films.	Poona University, Poona.
2. Dr. A. V. Rama Rao	Chemistry and Technology of dyes.	Yeshwant College, Nanded.
	Biogenetic concepts in structural elucidation in some phenolic compounds.	Osmania University, Hyderabad.

Name of the Scientist	*Subject*	*Venue*
	Phenolics of Garcinia species.	Osmania University, Hyderabad.
	Applications of NMR Spectroscopy and mass spectrometry to some problems concerning synthetic dyes.	-do-
3. Dr. P. N. Rangachari	Industrial fermentations	Abbasaheb Garware College, Poona.
	Microbial assays	-do-
	Enzymes	Maharashtra Association for the Cultivation of Science, Poona.
	Industrial fermentations	-do-
	Prostaglandins	Poona University, Poona.
4. Dr. M. C. Srinivasan	Some aspects of phytopathogenic bacteria.	Hindustan Antibiotics Limited, Pimpri, Poona.
	Seed transmission of phytopathogenic bacteria and technique for their detection in seed samples.	Central Food Technological Res. Institute, Mysore.
5. Dr. S. S. Tavale	The physical methods of molecular structure determination.	Wadia College, Poona.

5. STAFF STRENGTH* AS ON 31-3-1974.

1. Scientific

(i)	Director	1
(ii)	Scientist F	6
(iii)	Scientist E	18
(iv)	Scientist C	60
(v)	Scientist B	38
(vi)	Scientist A	39
(vii)	S. S. A.	81
(viii)	J. S. A.	34
(ix)	S. L. A.**	68
	Total	345

2.	Technical	192
3.	Administration	102
4.	Class IV technical	138
5.	Class IV non-technical	83
	Total	860

6. Research Fellows, Pool Officers and Guest Workers

(a)	JRF & SRF	64
(b)	CSIR Pool Officers	6
(c)	Guest Workers	29
	Total	99

7. Scientific staff working on sponsored projects

(a)	NCL staff	9
(b)	Staff deputed by sponsors	18
	Total	27

* This denotes actual working staff.

** Senior Laboratory Assistants (S.L.A.s.) are included under scientific category since majority of them have postgraduate qualification and are engaged on scientific work.

6. STAFF NEWS

6.1 *Deputations/training etc.*

1. Dr. N. D. Ghatge visited Romania under the agreement on Scientific and Technological Cooperation between Govt. of India and the Socialist Republic of Romania for studies in "Polymer and elastomer chemistry and technology" (May 1973).

2. Dr, M. Goswami, Mr. A. M. Lele, Dr. H. B. Mathur and Dr. R. B. Mitra attended a course on "Management for Research" at Administrative Staff College of India, Hyderabad (August, 1973).

3. Mr. S. S. Joshi, attended a course on "Process Planning" at NITIE, Bombay (September, 1973).

4. Mr. D. D. Kelkar attended a course on "Quantitative Methods for Decision Making" at NITIE, Bombay (Aug. 1973).

5. Dr. A. F. Mascarenhas was deputed to U. K. for three months under a British Council Award to study "the techniques of plant tissue culture and plant cell hybridization" (Oct. 1973).

6. Dr. C. A. Menezes was appointed to the Faculty of Stanford University, Dept. of Material Science and Electrical Engineering USA from July 1972 to September, 1973.

7. Mr. S. A. Tambe attended (i) "Management Information Systems" course conducted by NITIE, Bombay (April 1973) and (ii) "Management of Motivation" course conducted by SIET, Hyderabad (October, 1973).

8. Dr. B. D. Tilak visited U.K. as a Member of Indo-British Technological Sub-Group (July 1973).

9. Dr. B. D. Tilak attended the seminar and workshop on "Technology Forecasting" organized by the Industrial Management Centre Inc. at Castine, Maine, USA (June 1973)

6.2 *Participation of NCL scientists in seminars and symposia.*

1.	The scope of chemical industries in Vidarbha region (Seminar) Nagpur.	Dr. S. M. Abhyankar
2.	Golden Jubilee Celebrations of the Indian Chemical Society and the Annual Convention of Chemists, Calcutta.	Dr. J. L. Bose Dr. J. C. Sadana Mr. M. Husain
3.	Indo-German Seminar on Advance Solid State Physics and Conference on defects in Non-metallic solids, Madras.	Dr. A. Goswami
4.	Guha Research Conference, Mahabaleshwar.	Dr. V. Jagannathan Dr. J. C. Sadana

5.	Joint Seminar organized by ICMA-IMO-CPMA on NCST Science and Technology Plan, Bombay.	Mr. A. M. Lele Mr. J. V. Rajan
6.	Annual Meeting of the Society of Biological Chemists, Mysore.	Miss K. S. Meenakshi
7.	Meeting of the American Physical Society, San Diego, California, U S A.	Dr. C. A. Menezes
8.	Seminar on Mathematics in Biology and Medicine, Bangalore.	Dr. P. N. Rangachari
9.	Meeting on Bacterial Leaching of Ores, Dhanbad.	Dr. P. N. Rangachari Mr. V. S. Krishnamachar
10.	All India Summer Institute for Advanced Microbiology, Pimpri, Poona.	Dr. M. C. Srinivasan
11.	Summer School on seed pathology, quarantine and storage, Mysore.	Dr. M. C. Srinivasan
12.	14th Seminar on Electrochemistry, Karaikudi.	Dr. P. R. Subbaraman

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

Sr. No.	*Name*	*Degree*	*University*	*Subject of thesis*	*Guide*
1.	Chaudhari, R. V.	Ph.D.	Bombay	Studies in gas-liquid reactions	Dr. L. K. Doraiswamy
2.	Deshpande (Mrs.), V. V.	Ph.D.	Poona	Acylphosphatase: Studies on acylphosphatase of *Vigna catjang*	Dr. V. Jagannathan
3.	Dhaneshwar, N. N.	Ph.D.	Poona	The crystal and molecular structures of *o*-dimethyl-aminobenzoic acid and related compounds by X-ray diffraction	Dr. L. M. Pant
*4.	Goswami, A. P.	Ph. D.	Poona	Dielectric and other properties of thin films	Dr. A. Goswami
5.	Kulwant Singh	Ph.D.	Bombay	Studies in disperse dyes	Dr. B. D. Tilak
6.	More, K. M.	Ph.D.	Shivaji	Synthesis of heterocyclic compounds	Dr. V. N. Gogte

Sr. No.	*Name*	*Degree*	*University*	*Subject of thesis*	*Guide*
7.	Mujumdar, R. B.	Ph.D.	Poona	Isolation and structural elucidation of wood phenolics	Dr. K. Venkataraman
8.	Nayar, M. S. B.	Ph.D.	Kerala	Mass spectrometry of organic ions	Dr. K. G. Das
9.	Patil, J. N.	Ph.D.	Poona	Physico-chemical studies on some metal complexes	Dr. D. N. Sen
10.	Sathaye, S. D.	Ph.D.	Poona	Study of chemically deposited thin films by Zr0 and CdS	Dr. A. P. B. Sinha
11.	Yemul, S. S.	Ph.D.	Shivaji	Isolation and structure elucidation of wood phenolics	Dr. K. Venkataraman

6.4 *NCL scientists recognized by different universities as research guides*

1.	Dr. Ayyangar, N. R.	Poona
2.	Dr. Bose, J. L.	Bombay, Poona, Nagpur. Shivaji
3.	Dr.Chakravarti, K. K.	Poona, Shivaji, Karnatak, Bombay
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.	Bombay, Poona, Punjab, Madras
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, Poona
*17.	Dr. Kelkar, G. R.	Poona, Bombay, Shivaji
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, Banaras
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. Nayak, U. R.	Poona
26.	Dr. Pai, M. U.	Bombay

27.	Dr. Pant, L. M.	Poona
*28.	Dr. Ramachandran, B. V.	Shivaji, Poona
29.	Dr. Rama Rao, A. V.	Shivaji, Poona
30.	Dr. Rangachari, P. N.	Poona, Shivaji
31.	Dr. Rao, A. S.	Poona, Shivaji
32.	Dr. Roy Chowdury, P.	Marathawada, Poona, Shivaji
33.	Dr. Sadana, J. C.	Poona, Aligarh
34.	Dr. Sen, D. N.	Poona, Bombay
35.	Dr. Sethi, S. C.	Poona
36.	Dr. Sinha, A. P. B.	Poona, Banaras, Vikram, Bombay, Karnatak
37.	Dr. Siva Raman, C.	Poona
38.	Dr. Subbraman, P. R.	Poona, Bombay, Kerala, Gujarat
39.	Dr. Tilak, B. D.	Bombay, Poona
*40.	Dr. Venkataraman, K.	Poona, Bombay, Madras, Banaras, Karnatak

* Retired/Emeritus scientists

6.5 *Consultancy*

During the year consultancy was offered to the following firms either through individual scientists or groups of scientists, wherein 14 scientists were involved:

1. Hindustan Organic Chemicals Ltd., Rasayani
2. Hindustan Antibiotics Ltd., Pimpri, Poona.
3. Indian Petrochemicals Corporation Ltd., Baroda.
4. Vidarbha Organic Chemical Industries Pvt. Ltd., Nagpur.
5. Hindustan Ferodo Ltd., Bombay.
6. National Rifles Ltd., Ahmedabad.
7. Poona Synthetic Company, Poona.
8. Indian Dyestuffs Industries Ltd., Bombay.
9. Aniline Dyestuffs and Pharmaceuticals (P) Ltd., Bombay.
10. Industrial Oxygen Co. (P) Ltd., Poona.
11. Camphor and Allied Products, Baroda.

7. PUBLICATIONS

7.1 *Research papers*

Solid state materials

1. Suseela (Mrs.), S. and Sinha, A.P.B.
   Electrical and magnetic properties of some Cu-Mn spinels.
   Ind. J. of Pure and Applied Physics, **11**, 112 (1973).

2. Suseela (Mrs.), S. and Sinha, A.P.B.
   A study of 90° B-B interaction in spinels containing Cr^{3+} and Mn^{4+} ions.
   Ind. J. of Pure and Applied Physics, **11**, 116 (1973).

3. Rao, V. J.
   Optical absorption spectra of Yb^{3+} in alkali halide single crystals.
   Ind. J. of Pure and Applied Physics, **11**, 441 (1973).

4. Rao, V. J. and Bakare, P. P.
   Conversion of Infrared to visible emission in Yb^{3+} sensitized rare earth phosphors.
   Ind. J. of Pure and Applied Physics, **11**, 442 (1973).

5. Pol, P. G. and Sinha, A.P.B.
   Structural and luminescence properties of (Ca, Cd) S phosphors.
   Ind. J. of Pure and Applied Physics, **11**, 504 (1973).

6. Rao, V. J.
   Absorption and fluorescence emission spectra of Nd^{3+} in NaCl and KCl single crystals.
   Ind. J. of Pure and Applied Physics, **11**, 833 (1973).

7. Pol, P. G. and Sinha, A.P.B.
   Fluorescence studies on Europium doped sodium-yttrium fluoride phosphors.
   Ind. J. of Pure and Applied Physics, **11**, 886 (1973).

8. Goswami, A. and Goswami, A. P.
   Dielectric and optical properties of Zns films.
   Thin Solid Films, **16**, 173 (1973).

9. Goswami, A. and Ojha, S. M.
   Semiconducting properties of tellurium films.
   Thin Solid Films, **16**, 189 (1973).

10. Goswami, A. and Prabhat Singh.
    Epitaxial growth of Co-Te films.
    Ind. J. of Pure and Applied Physics, **11**, 373 (1973).

11. Goswami, A. and Goswami, N. N.
Vapour phase deposits of GaP on single crystals.
Ind. J. of Pure and Applied Physics, **11**, 435 (1973).

12. Goswami, A. and Ojha, S. M.
Studies on electrical properties of bismuth oxide films.
Thin Solid Films, **20**, 307 (1974).

13. Goswami, A. and Goswami, A. P.
A.C. behaviour of vacuum deposited praseodymium oxide films.
Thin Solid Films, **20**, S3 (1974).

14. Goswami, A. and Rao, B. V.
Optical properties of cuprous sulphide films.
Ind. J. of Pure and Applied Physics, **12**, 4 (1974).

15. Goswami, A., Rao, B. V. and Prabhat Singh.
Structural and optical properties of indium arsenide films.
Ind. J. of Pure and Applied Physics, **12**, 26 (1974).

16. Goswami, A. and Goswami, A. P.
Optical properties of Sb_2O_3 films.
Thin Solid Films, **20**, S33 (1974).

17. Goswami, A. P. and Goswami, A.
Dielectric and structural properties of vacuum deposited niobium oxide films.
Ind. J. of Pure and Applied Physics, **12**, 26 (1974).

Physico chemical studies

18. Bhasin, S. K. and Sen, D. N.
Mass spectrometric identification of hydrazones of an aromatic aldehyde.
J. Indian Chem. Soc., **50**, 155 (1973).

19. Das, K. G. and Madhusudanan, K. P.
Substituent effects on retro-Diels Alder reaction in some 3,6-diphenyl-5-aryl-4-amino- $\triangle'$-cyclohexenes.
Org. Mass Spectrom, **7**, 619 (1973).

20. Das, K. G. and Kulkarni, P. S.
Substituent effect on rearrangement in aldoximes.
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7.2 *Publications in collaboration with outside scientists*

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7.3 *Chapters in Books*

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7.4 *Papers presented at symposia, seminars, etc.*

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   Structure of the Kostanecki-Robinson acetylation product of β-orcacetophenone.
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2. Sivaramakrishnan, V. and Nair, P. M.
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3. Vartak, H. G., Paranjpe, S. V., Bodhe, A. M., and Jagannathan, V.
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   Annual Meeting of the Society of Biological Chemists (India), Mysore, October/November, 1973.

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   Characterization of papain inhibitors of *Vigna catjang*.
   Annual Meeting of the Society of Biological Chemists (India), Mysore October/November, 1973.

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Electrical properties of vacuum deposited bismuth films.
Seminar on Advanced Solid State Physics, Madras, February/March, (1974).

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Seminar of the Advanced Centre of Bombay University Dept. of Chem. Technology, Bombay, (1974).

8. PATENTS IN FORCE

8.1 *Indian patents sealed*

1. **64958***
Improvements in or relating to polishing compositions.
Shah, S. M., Hinge, V. K., Mhaskar, V. V. and Shah, R. C.

2. **65440***
A process for the extraction of wax from sisal waste.
Shah, S. M., Hinge, V. K., Mhaskar, V. V. and Shah, R. C.

3. **65976***
Improvements in or relating to suspension polymerization of vinyl monomers.
Joshi, R. M., and Kapur, S. L.

4. **65977***
Rubber base adhesive.
Uma Shankar.

5. **66096***
A process for the production of bacterial diastase by submerged culture.
Babbar, I. J., Bekhi, R. M. and Srinivasan, M. C.

6. **66194***
Improvements in or relating to can sealing composition.
Raghunath, D. and Kapur, S. L.

7. **71063**
Production of bacterial protease by submerged culture.
Babbar, I. J., Powar, V. K. and Jagannathan, V.

8. **71190***
Preparation of anion exchange resins.
Krishnaswamy, N., Govindan, K. P. and Dasare, B. D.

9. **73702**
A process for the preparation of cylcopentadecanolide (exaltolide).
Dhekne, V. V., Ghatge, B. B. and Bhattacharyya, S. C.

10. **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.

11. **77225***
A process for the preparation of β-ionone from pseudoionone.
Joshi, B. N., Chakravarti, K. K., Shah, R. C. and Bhattacharyya, S. C.

12. **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.

13. **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.

14. **86991***
Preparation of polyurethane printing rollers.
Ghatge, N. D. and Kapur, S. L.

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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.

16. **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.

17. **98156***
Preparation of solvent modified copolymers of vinyl monomers in bead form.
Kapur, S. L. and Ramakrishnan, K.

18. **109489**
Production of aryl-glycosides.
Ingle, T. R. and Bose, J. L.

19. **113406**
A new thermistor composition exhibiting high positive temperature coefficient of resistivity (posistor).
Brahmecha, B. G. and Sinha, K. P.

20. **116453**
Disperse dyes for polyesters with good affinity and sublimation fastness.
Raman, S. K. and Tilak, B. D.

21. **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.

22. **117210**
A direct process for the manufacture of dibutyltin diiodide and of the oxide therefrom.
Gupta, J., Gopinathan, C., Gopinathan, (Mrs.) S. and Awasarkar, P. A.

23. **118476**
Methods of preparing isocyanates, mono and polyureas from pentadecenyl phenol (anacardol).
Ghatge, N. D. and Yadav, S. D.

24. **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.

25. **123638**
Inprovements in or relating to the manufacture of cashewnut shell gum (CNS-gum).
Ingle, T. R., Vaidya, S. H. and Pai, M. U.

26. **125138**
Method of preparing polyisocyanates and polyureas from pentadecenyl phenol (anacardol).
Ghatge, N. D. and Mahajan, S. S.

27. **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.

*These patents (14) have been released for exploitation.

8.2 *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. **113825**
   Nickel zinc ferrites.
   Krishna Rao, V. V., Kanade, (Miss) S. B. and Sinha A. P. B.

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. **131606**
Improvements in or related to a process for the preparation of gamma ferric oxide.
Lakhbir Singh, Chavan, A. M. and Kotasthane, A. N.

12. **134641**
A device for warming and cooling.
Lakhbir Singh and Kotasthane, A. N.

13. **221/72**
New dyes containing azido groups for cellulosic protein and synthetic fibres.
Ayyangar, N. R., Badami N. V., Daruwalla, E. H. and Tilak, B. D.

14. **328/72**
A new process for the halogenation of copper phthalocyanine.
Ayyangar, N. R., Moghe, P. P. and Tilak, B. D.

15. **1894/72**
A new method for beneficiation of minerals, ores and industrial waste products through direct high temperature chlorination in absence of a reducing agent.
Neurgaonkar, V. G., Dadape, V. V. and Kuruvila, J.

16. **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.

17. **2331/72**
Improvement in or relating to the preparation of new reactive dyes.
Ayyangar, N. R., Badami, N. V., Tilak, B. D. and Daruwalla, E. H.

18. **2232/72**
Process for the preparation of D-Galactose from cashewnut shells.
Ingle, T. R., Vaidya, S. H. and Pai, M. U.

19. **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.

20. **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.

21. **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.

22. **1107/Cal/73****
Improvements in or relating to the preparation of new reactive dyes containing azido acetylamide groups.
Ayyangar, N. R., Badami, N. V., Tilak, B. D. and Daruwalla, E. H.

23. **2642/Cal/73****
Improvements in or relating to the preparation of 1, 3, 3-trimethyl-2-methylene indoline.
Ayyangar, N. R., Pandit S. K. and Tilak B. D.

24. **2687/Cal/73****
Method of preparing phosphites metal salts from 3-pentadecyl phenol for use as stabilizers for polymers such as PVC.
Vernekar, S. P. and Ghatge, N. D.

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

8.3 *Foreign patents in force*

1. **Belgium Patent No. 751806.**
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. 2946783**
Preparation of costus root oil and the production thereof (corr. to Indian patent No. 59853).
Kelkar, G. R. and Bhattacharyya, S. C.

4. **Cuba Patent No. 149082**
Manufacture of nicotine sulphate from tobacco or tobacco waste (corr. to Indian patent No. 45666).
Gedeon, J. and Goswami, M.

5. **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.

6. **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

T-Metric tons

S. No.	Name of the process/product & Indian Patent No.	Field of utilization	Name of the manufacturer (year of commencement of production)	Production 1973-74 Qty/Value Rs. in lakhs	Production Upto Mar. 73 Qty/Value Rs. in lakhs	Terms of release and Remarks
1	2	3	4	5	6	7
1.	Acetanilide	Intermediates	M/s. Hindustan Organic Chemicals Ltd., Rasayani, through project engineers M/s. R. L. Dalal & Co., Bombay-18(1969).	1108.8 T 113.17	4605.75 334.37	Non-exclusive
2.	Acriflavine	Pharmaceuticals	M/s. Western India Fine Chemicals, 83, Lalbahadur Shastri Marg, Bombay-80 (1969).	919 kg. 3.98	3629 kg. 15.78	Sponsored 470 kgs. worth Rs. 2.13 lakhs were exported.

1	2	3	4	5	6	7
3.	Adhesives for decorative laminates	Laminates	M/s. Swastik Rubber Products, Ltd. Poona-3 (1969).	11 T 1.16	64.75T 6.53	Sponsored
4.	Anion exchange resin from melamine (71190)	Demineralization of liquids	M/s. Tulsi Industries, Bhosari Industrial Area, Poona-26 (1963).	— 0.018 (estimated)	845 Cu. ft. 2.05	Non-exclusive
5.	Antipriming compositions (77081)	Antipriming in locomotives	Research, Design and Standard Organisation, M & C Wing, Chittaranjan (1964).	10.3 T 0.33	108.30 2.90	Non-exclusive
6.	Bacterial diastase (66096)	Textile desizing	M/s. Chemaux (P) Ltd., Sitaladevi Temple Rd., Mahim, Bombay-16 (1967).	— —	341.58 T 19.00	Exclusive
7.	Berberine hydrochloride	Pharmaceuticals	M/s. Nitin Pharmaceuticals, 180/82, Samuel Street, Bombay-9 (1965).	4.0 T 8.00	20.30 T 40.36	Sponsored
8.	tert-Butylcatechol	Synthetic rubber	M/s. Percynic Chemicals, United Bank of India Bldg., Sir P. M. Road, Bombay-1 (1972).	— 2.28 (estimated)	7.8 T 3.67	Non-exclusive

1	2	3	4	5	6	7
9.	Butyl titanate	Varnishes, enamels	M/s. Synthochem, 33-A, Laxmi Nagar, Indore-1 (1973).	19.85 T 6.95	16.6 T 5.00	Non-exclusive
10.	Cadmium sulphide photo-conductive cells	Electronics	M/s. Chinoy Electronics, 64, Koregaon Park, Poona-1 (1971).	659 Nos. 0.07	1071 Nos. 0.13	Non-exclusive
11.	Calcium hypophosphite	Pharmaceuticals	M/s. Hypophosphite & Co., 79-F, Princess Street, Bombay-2 (1967).	18 T 8.00	50.25 T 28.30	Sponsored
12.	Calcium silicate	Low density insulators	M/s. Newkem Products Corpn. Harganga Mahal, Bombay-14 (1968).	310 T 10.85	1022.21 T 36.00	Sponsored
13.	Can sealing composition (66194)	Metal can industry.	M/s. Arya Chemical Works, 114/2A, Dharamtala Street, Calcutta-13 (1962).	38.2 T 2.31	215.20 T 11.94	Non-exclusive
*14.	Carbazole Dioxazine Violet pigment	Organic pigment	M/s. Square Chemicals, 73, Sant Tukaram Road, Bombay-9 (1974).	5 kg. 0.03	— —	Non-exclusive

1	2	3	4	5	6	7
15.	Carbimazole	Pharmaceuticals	M/s. Indian Schering Ltd., Sion-Trombay Road, Chembur, Bombay-71 (1970).	180.6 kg. 14.94	49.4 kg. 4.09	Sponsored
16.	Catechol	Pharmaceuticals	M/s. Percynic Chemicals, Bombay-1 (1972).	— 0.77 (estimated)	9.15 T 2.14	Non-exclusive
17.	Chloral hydrate	Industrial chemicals	M/s. Hindustan Insecticides Ltd., Industrial Area, New Delhi-15 (1963).	—	20.78 T 2.14	Sponsored
18.	Clofibrate	Pharmaceuticals	M/s. Biological Evans, 18/1 & 3, Azamabad, Hyderabad-20 (1973).	516 kg. 0.88	100 kg. 0.17	Non-exclusive
19.	Diethyl-m-aminophenol	Dye-intermediate	M/s. Sahyadri Dyestuffs and Chemicals (P) Ltd., 117, Vithalwadi Road, Poona-9 (1973).	62.52 T 62.52	51.44 T 30.83	Sponsored
20.	Dihydroisojasmone and peach aldehyde	Perfumery	(i) M/s. S. H. Kelkar & Co. (P) Ltd., Bombay-Agra Road, Bombay-80 (1965).	0.6 T 1.28	6.96 T 13.97	Non-exclusive
			(ii) M/s. Sonebon Laboratories Kottayam-5 (1971).	0.15 T 0.35	0.07 T 0.18	Non-exclusive

1	2	3	4	5	6	7
21.	Dimethylaniline	Industrial chemicals	M/s. Sahyadri Dyestuffs and Chemicals P. Ltd., Poona (1972).	11.13 T 1.67	25 T 2.50	Non-exclusive
22.	Ethylene oxide condensates	Surface active agents	M/s. Hico Products (P) Ltd., Mogal Lane, Bombay-16 (1965).	841 T 105.00	2373 T 287.88	Sponsored
23.	Ferrites (Hard)	Electronics	M/s. Semiconductors Ltd., Nagar Road, Poona-14 (1968).	3.5 T 0.50	5.2 T 4.10	Non-exclusive
24.	Geraniol, citronellal and citronellol	Perfumery	M/s. Opal Fine Chemicals, Veer Savarkar Marg, Bombay-25 DD (1970).	3.2 T 4.80	9 T 10.50	Sponsored
25.	β-Ionone (77225)	Perfumery, Pharmaceuticals	M/s. Industrial Perfumes Ltd., Army & Navy Bldg., M. G. Road, Bombay-1 (1968).	10.25 T 17.43	76.56 T 89.33	Non-exclusive. 10 M. T. were exported to USSR through an export house.
26.	Nicotine sulphate	Insecticides	M/s. Urvakunj Nicotine Industries, Petlad-Cambay Road, Dharmaj, (Dist. Kaira) (1963).	86 T 18.30	87.81 T 15.44	Non-exclusive. Entire production exported.

1	2	3	4	5	6	7
*27.	Nitrile rubber	Oil resistant rubber formulations, adhesives	M/s. Synthetics & Chemicals Ltd., Bombay-20 (1974).	30 T 6.00 (approx).	—	Exclusive for 4 years
28.	Orthotolyl biguanide	Soap	M/s. Industrial Perfumes Ltd., Bombay-1 (1970).	2.99 0.45	6.47 1.75	Exclusive
29.	Perfumery products based on longifolene (Capinone)	Perfumery	M/s. Camphor and Allied Products, Bareilly (1968).	11.2 T 6.66	28.55 T 23.54	Sponsored
30.	Perfumery products based on $\triangle^3$-carene (Meracene)	-do-	-do-	5.9 T 1.87	9 T 3.69	Sponsored
31.	β-Phenethyl alcohol	Perfumery	M/s. Sunanda Aromatic Industries, K. R. S. Road, Mettagalli, Mysore (1970).	79.06 T 31.62	69 T 23.55	Sponsored

1	2	3	4	5	6	7
32.	Phthalates-dioctyl and dibutyl	Plasticizers	(i) M/s. Alta Laboratories Ltd., Giri Vihar, Khopoli, (Dist. Kolaba) (1969).	—	1834.10 T 123.43	Non-exclusive
			(ii) M/s. India Carbon Ltd., Dr. A. B. Road, Worli, Bombay-18(1971).	1297 T 149.00	4833 T 262.17	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).	193.55T(DEP) 26.13	201.02 T 14.20	Non-exclusive
*34.	D. C. Recording Polarograph	Polarographic analysis	M/s. Elico Pvt. Ltd., B-17, Industrial Estate, Sanatnagar, Hyderabad-18 (1974).	1 Unit 0.10	—	Non-exclusive
35.	Polyurethane printing rollers (86991)	Printing	M/s. Sree Saraswathy Printing Press Ltd., 32, Acharya P. C. Road, Calcutta-9 (1965).	629 pieces of rollers 0.81	303 pieces of rollers 0.50	Non-exclusive
36.	Rigid filters (59608, 66966)	Tube wells	M/s. Ashim Filters, 196, Defence Colony, New Delhi (1965).	1735 Mtrs. 2.03	9220 Mtrs. 19.02	Non-exclusive

1	2	3	4	5	6	7
37.	Rubber blowing agent	Rubber chemicals	M/s. Swastik Rubber Products Ltd., Poona-3 (1968).	27.63 T 3.85	175.23 T 24.56	Non-exclusive
38.	Rubberized cork sheet	Gaskets	M/s. Bharat Casements (P) Ltd., P. B. No. 89, Baroda (1966).	24.38 lakh pieces 9.46	71.23 lakh pieces 25.31	Non-exclusive
39.	Rubber re-claiming agent	Rubber chemicals	M/s. Swastik Rubber Products Ltd., Poona-3 (1968).	4.49 T 0.89	26.06 T 3.98	Non-exclusive
40.	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).	275 pads 0.04	6000 pads 1.00	Non-exclusive
			(ii) M/s. Vasant Industrial Corpn. 356, Great Nag Road, Nagpur-2. (1971).	— 0.21 (estimated)	800 pads 0.08	-do-
41.	Silica gel	Humidity control	M/s. Minco Products, 301/27, T. H. Road, Madras-21 (1963).	12.5 T 1.02	95 T 6.67	Sponsored
42.	Sorbide nitrate	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay, (1969).	103.5 kg. 1.43	503 kg. 9.05	Sponsored

1	2	3	4	5	6	7
*43.	Sorbitol	Pharmaceuticals, Vitamin C synthesis	M/s. Hindustan Antibiotics Ltd., Pimpri, Poona-18.	40 T 4.00	— —	Non-exclusive
*44.	Direct reading spectrophotometer/colorimeter	Biochemical research, spectroscopic analysis in visible range	(i) M/s. Neotronics Corporation P. B. No. 7776, Mulund, Bombay-80 (1974).	10 Units 5.24	— —	Non-exclusive
			(ii) The Scientific Instruments Manufacturing Co. Ltd., Allahabad-1 (1974).	4 Units —	— —	Non-exclusive
45.	Thermistors	Electronics	(i) M/s. Semiconductors Ltd., Poona-14 (1963).	6.5 lakhs Nos. 6.00	47.15 lakh Nos. 39.48	Non-exclusive
			(ii) M/s. Tempo Semiconductors Vile Parle, Bombay-57 (1963).	26469 Nos. 0.97	64600 Nos. 2.79	Non-exclusive
*46.	Thermosetting resins for industrial laminates	Industrial laminates	M/s. Formica India Ltd., Chinchwad, Poona-19 (1974).	3 T for captive consumption	— —	Sponsored

1	2	3	4	5	6	7
47.	Vapour phase chromatograph	Instruments	M/s. Associate Instruments Manufacturers (India) P. Ltd., 26-27, Asaf Ali Road, New Delhi (1969).	21 Units 3.99	92 Units 18.35	Exclusive
*48.	Vitamin C	Pharmaceuticals	M/s. Hindustan Antibiotics Ltd., Pimpri, Poona-18 (1974).	85 kgs. —	— —	Non-exclusive
49.	Laboratory Chemicals	—	Produced by NCL	— 1.04	— —	—

* During the period under review, production has been newly reported on these items (7)

VALUE OF PRODUCTION BASED ON NCL KNOW-HOW

Year	No. of items manufactured (including major fine chemical 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
1973—74	49	651.39
	Total	2256.51

Note: The following processes which were included in Table I of the Annual Report 1972-73 have now been dropped since no production was reported for two consecutive years.

(a) 4-Hydroxycoumarin

(b) Warfarin

(c) Cation exchange resin from CNSL

These are in addition to the processes on (a) Hexylresorcinol, (b) Liquid rubber (c) Radio opaque dyes (d) Rubber based contact adhesive and (e) Sisal wax which were dropped in the last report.

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 Organic Chemicals (HOC), Rasayani (1973).	Non-exclusive	B
2.	Benzoic acid (IP) from crude methyl benzoate	Pharmaceuticals	M/s. Indian Petrochemicals Corpn. Ltd., P. O. Jawaharnagar, Baroda (1972).	-do-	B
3.	Substitutes for bostik sealants	Adhesives	M/s. Swastik Rubber Products Ltd., Poona-3 (1972).	-do-	B
4.	Calcium silicate from wollastonite	Industrial inorganic chemicals	M/s. Chemicals India, Chetak Bldg., P. B. No. 4, Udaipur (1971).	Sponsored	B
5.	Can lining composition (based on nitrile rubber latex)	Lining cans for storing mineral oils, greases, food, etc.	M/s. Arya Chemical Works, 141/2A, Dharamtala St., Calcutta-13 (1972).	Non-exclusive	B

1	2	3	4	5	6
6.	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 High temperature chlorination unit installed; trial runs in progress
7.	Chlorobenzenes	Industrial chemicals	M/s. HOC, Rasayani (1969).	-do-	B Installation of 4500 TPA plant completed.
8.	Conversion of bauxite into anhydrous aluminium chloride	Industrial inorganic chemicals	M/s. Tata Chemicals Ltd., Bombay, (1973).	-do-	B
9.	Chloromethanes (86541)	Industrial chemicals	M/s. Standard Alkali, Chem. Div. The Standard Mills, Co. Ltd., Mafatlal Centre, P. B. No. 1038, Bombay-1 (1969).	Exclusive for 2 years	B 3000 TPA Plant installed. Commissioning runs in progress

1	2	3	4	5	6
10.	Coating for oil filter papers	Oil filtration	M/s. White Cloud Paper Mills, 412, Gultekdi Road, Poona-9 (1968).	Non-exclusive	B
11.	Ethylene from alcohol	Organic intermediates	M/s. Industrial Oxygen Co. Ltd., Poona-1 (1972).	-do-	A
12.	Ethylene diamine	Bulk organic chemical	The Bharat Vijay Mills Ltd., Kalol, Gujarat (1973).	-do-	B
13.	Flexible magnets	Refrigeration gaskets, toys, educational kits	(i) M/s. Ajanta Enterprises, Mahalaxmi Ind. Estate, Gandhi Nagar, Bombay-13 (1973).	-do-	A
			(ii) M/s. Ferrites and Electronic Components Pvt. Ltd., Balmiki Marg, Lucknow (1974).	-do-	A
			(iii) M/s. V. P. Nijhawan, III-F/5, Lajpat Nagar, New Delhi-24 (1973).	-do-	A
14.	Foundry core binder	Core binder in steel foundries for high dimensional accuracy	M/s. Card-Chem Industries, Hyderabad (1973).	-do-	B

1	2	3	4	5	6
15.	Gaskets from coir pith	Gaskets	M/s. Oberoi Industries, 12/37, Tilak Nagar, New Delhi (1974).	-do-	A
16.	Gum arabic substitute	Adhesives	(i) M/s. Karnatak Adhesives, 19, Mysore Deviation Road, Gopalapuram, Bangalore-23 (1974).	-do-	B
			(ii) M/s. Bal Krishan Mital, Quarter No. CN-2/1, NRC Colony, Mohone, Dist. Thana (1973).	-do-	A
			(iii) M/s. Supreme Enterprises, Bhagwan Nivas, Kailash Cinema Chowk, Ludhiana (1973).	-do-	A
17.	Hexachloroethane	Industrial chemicals	M/s. Industrial Oxygen Pvt. Ltd., Off Nagar Road, Poona-14 (1971).	Non-exclusive	B Plant of 150 TPA installed.
18.	8-Hydroxyquinoline	Pharmaceuticals	M/s. Alta Laboratories Ltd., Kopoli (1970).	Sponsored	B

1	2	3	4	5	6
19.	Ketene from acetone	Intermediates	M/s. Sudarshan Chemical Industries, Poona (1971).	-do-	B
20.	Liquid stabilizer for PVC	Plastics	M/s. Coated Fabrics (P) Ltd., 426/1, Chatushringi Road, Poona-16 (1969).	-do-	B
21.	Megimide	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay-71 (1969).	-do-	B
22.	*p*-Menthane hydroperoxide	Synthetic rubber	M/s. Camphor & Allied Products, Bareilly (1969).	Exclusive	B
23.	l-Menthol from dementholized peppermint oil	Fine chemicals, drugs	M/s. Bhavana Chemicals, Ltd., Laxmi Insurance Building, Bombay-1 (1968).	Sponsored	B The firm is not using NCL technology.
24.	l-Menthol from citronella oil of Indian origin	Fine chemicals, drugs	M/s. Bhavana Chemials Ltd., Laxmi Insurance Building, Bombay-1 (1973).	Sponsored	A

1	2	3	4	5	6
25.	Microfilters	Industrial filtrations	M/s. Sona Microfilters, 'Joseph House', Padamjee Park, Poona-1 (1973).	Non-exclusive	A
26.	Monochloroacetic acid	Weedicides, CMC	M/s. HICO Products (P) Ltd., Bombay-16 (1972).	-do-	B Plant installed; trial runs in progress.
27.	Monoethylaniline	Intermediates for explosives	M/s. Atul Products Ltd., Atul, Dist. Bulsar (1973).	-do-	B Installation of plant completed
28.	Extraction of morphine and other alkaloids from lanced poppy straw	Pharmaceuticals	Ministry of Finance, Govt. of India, New Delhi (1972).	Sponsored	A
29.	Preparation of nitro musk compounds	Perfumery	M/s. Opal Fine Chemicals, Bombay (1973).	-do-	A
30.	*p*-Nitrophenol	Insecticides	M/s. HOC, Rasayani (1972).	Non-exclusive	B

1	2	3	4	5	6
31.	Nonylphenol	Surface active agents	M/s. Surfactants (P) Ltd., 28, Apollo Street, Bombay-1 (1969).	Sponsored	B Implementation expected soon.
32.	Opium alkaloids	Pharmaceuticals	Ministry of Finance, Govt. of India, New Delhi (1966).	Exclusive	B 4.7 TPA plant under installation.
33.	Oxalic acid from bark of Ain tree	Industrial chemicals	M/s. Vidarbha Organic Chemical Industries Ltd., Indian Mutual Building, Mount Road Extension, Nagpur (1972).	Sponsored	B Plant under installation
34.	Oxy-urea	Textile chemical	M/s. Calico Chemicals & Plastics Divn., Anik-Chembur, Bombay-74 (1971).	-do-	B
35.	*p*-Phenetidine	Pharmaceuticals	M/s. Kabbur Industries Ltd., 3, Bastion Road, Bombay-1 (1971).	-do-	B
36.	Phenylacetic acid	Perfumery, Penicillin G	(i) M/s. Laxmi Chemical Industries (P) Ltd., Hyderabad-1 (1972).	Non-exclusive	B

1	2	3	4	5	6
			(ii) M/s. Orient Aroma Chemical Industries Pvt. Ltd., 27, Chinchbunder Road, Bombay-9 (1973).	-do-	A
37.	Potentiometric strip chart recorder	Recording instruments	(i) M/s. Associated Instruments Manufacturers (India) Pvt. Ltd., New Delhi (1970).	-do-	B
			(ii) M/s. Bagga Electronics, Bombay-4 (1972).	-do-	B
38.	Radiosonde thermistors	Meteorology	The Bhagyanagar Laboratories, 1-1-523/8, Golkonda Cross Road, Hyderabad (1973).	Non-exclusive	B
39.	Removal of silica from black liquor	Paper industry	The Central Pulp Mills Ltd., 1183, Shivajinagar, Poona-4 (1971).	Sponsored	B Commercial plant under installation.
40.	Synthesis of resin for friction material	Friction materials	M/s. Hindustan Ferodo Ltd., Ghatkopar, Bombay-86 AS (1973).	-do-	A

1	2	3	4	5	6
41.	Rubberized cork sheets from waste cork granules	Gaskets	M/s. Cork Products (P) Ltd., 9, Jor Bagh, New Delhi (1974).	Non-exclusive	A
42.	Solvent extraction of sandalwood oil	Perfumery	Govt. Sandalwood Oil Factory, Bangalore (1973).	Sponsored	B
43.	Sulphacetamide and its sodium salt	Pharmaceuticals	M/s. Indian Schering Ltd., Bombay-71 (1969).	-do-	B
44.	Terpineol	Perfumery	M/s. Dujodwala Industries, 14-1 Mile, Mathura Road, Faridabad (1972).	Non-exclusive	B
45.	Fractionation of turpentine oil (thermodynamic studies)	Industrial solvent	M/s. J. & K. Industries Ltd., Srinagar (1973).	Sponsored	A
46.	*p*-Toluidine from *p*-nitro-toluene by vapour phase reduction	Organic intermediate	M/s. Sudarshan Chemical Industries Private Ltd., 162, Wellesley Road Poona (1973).	-do-	B Installation of 300 TPA plant in progress.

1	2	3	4	5	6
47.	Vitamin B_6 (127750)	Drugs	M/s. Indian Drugs & Pharmaceuticals Ltd., New Delhi (1974).	Non-exclusive	A
48.	Xylit	Pharmaceuticals, fine chemicals.	M/s. Unichem Laboratories Ltd., Bombay (1972).	Sponsored	B

A—Processes recently released.
B—Processes likely to be implemented soon.

The following processes which were included in Table II of Annual Report 1972-73 have now been dropped as the licencees have not shown any progress towards their implementation for a considerable period:—

1. Benzoic acid from toluene
2. Butylated hydroxyanisole
3. Cation exchange resin polystyrene base
4. Chlorohydroxyquinoline
5. Coumarin
6. *p*-Nitroacetophenone
7. Pentachlorophenol
8. Propoxyphene
9. Radio opaque dyes
10. Sisal wax
11. Styrene DVB base cation exchange resin
12. Vat golden yellow GK

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

TABLE II(A) : The following processes which have been mentioned in Table I are also licensed to the following firms and are awaiting production:

	Name of the process	Name of the party and year of release
1.	Carbazole dioxazine violet pigment	M/s. Vapson Products, 419, A, Arun Chambers Tardeo Road Bombay (1973)
2.	Ferrites—hard	M/s. Ajanta Enterprises, Mahalaxmi Industrial Estate, Gandhi Nagar Bombay 13 (1971)
3.	β-Ionone	M/s. S. H. Kelkar and Co., Bombay (1964)
4.	Nicotine sulphate	M/s. P. Jaipuria, 1A, S. N. Banerjea Road Calcutta 13, (1972)
5.	Phthalates -dioctyl and dibutyl	M/s. Synthetic Organics, Amar Hill, Sakivihar Road, Bombay 72 (1971)
6.	DC Recording Polarograph	(i) M/s. Laxsons Engineering and Electronics (P) Ltd., opp. Marol Bus Depot, Andheri (East) Bombay 59 (1973) (ii) M/s. Chromatography and Instruments Co., 121-122, Makarpura Industrial Estate, Baroda (1973)
7.	Polyurethane printing rollers	M/s. United Ink and Varnish Co., P. B. 6862, Vile Park, Bombay 57 (1965)
8.	Sachets-hot and cold	M/s. Biswanath Fatesaria, 5/1 Ramkumar Rakhit Lane Calcutta 7 (1973)
9.	70% Sorbitol	M/s. Maize Products, P. O. Kathwada, Maize Products Ahmedabad (1973)

TABLE II(B): 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 (excluding FCP processes).	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
1974	32	388.50	39	44	115	28	88

Break-up of 39 processes which have been released and are awaiting production (column 4) is as follows:—

A — 8 Processes recently released.

B — 7 Processes on which progress has been reported.

C — 24 Processes on which progress has not been reported or on which production has been discontinued for market/economical/technical reasons.

* Total cumulative 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(C) : 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 not in 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).	% 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	14	36	18
1969	13	7.11	48.06	16	2	8	29	45	30
1970	15	7.17	68.08	15	9	18	30	50	42
1971	19	6.68	92.72	15	13	20	34	56	46
1972	16	8.67	120.67	28	13	22	44	36	55
1973	15	4.54	154.39	36	13	22	51	29	61
1974	16	4.21	261.85	39	13	23	55	29	58

* This column includes projects where no industrial production was expected to be realized. These include PL-480 schemes, Lac Cess Committee Project, ICMR Schemes, Projects for development of analytical procedures, etc.

TABLE III : KNOW-HOW AVAILABLE

S. No.	Name of the process/product	Field of utilization	Remarks
1	2	3	4
1.	Acetanilide	Drugs and dye intermediate	Released to one party. In production. Turnkey plant available through Project Engineers.
2.	Aniline	Organic intermediate	Released to one party.
3.	Anion exchange resin from melamine (Ind. Pat. No. 71190)	Demineralization of liquids	Released to one party.
4.	Antipriming composition	Antipriming in locomotives	Released to one party; in production.
5.	L-Arabinose(CP)	Biochemical research	Export potential.
6.	Atrazine	Herbicide	—
7.	Benzoic acid from crude methyl benzoate	Pharmaceuticals	Released to one party.
8.	Bisphenol-A	Epoxy resins	—
9.	Substitute for Bostik sealant	Oil resistant adhesive for fuel tanks	Released to one party; in experimental production.
10.	tert-Butyl catechol	Stabilizer and polymerization inhibitor for synthetic rubber	Released to one party; in production.
11.	Butyl titanate	Insulating varnishes, special paints, catalyst	Released to one party; in production.
12.	Cadmium sulphide Photo-cells	Instruments, photoelectric devices	Released to two parties ; one in production.

1	2	3	4
13.	Can lining composition	Lining cans for storing mineral oils, greases, food	Released to one party.
14.	Carbazole Dioxazine Violet pigment	Organic pigment	Released to two parties; one in production.
15.	Cashewnut shell gum (Ind. Pat. No. 123638)	Binder, thickening agent in food and pharmaceuticals	—
16.	Catechol	Organic intermediate	Released to one party; in production.
17.	Cellulose powder	Chromatography, coating for electrodes, filter media	—
18.	Civetone and Dihydro-civetone	Perfumery	—
19.	Clofibrate	Drug	Released to one party; in production.
20.	Coating for oil filter papers	Oil filters	Released to one party; in experimental production.
21.	Costus root oil	Perfumery	Costus roots are available in Kashmir and Punjab.
	Fructose	Medicine	
	Chamazulene	Cosmetics	
22.	D. C. Recording polarograph	Polarographic analysis	Released to three parties; one in production.
23.	Dibutyl tin stabilizers for PVC.	PVC Industry	—
24.	Dihydroambrettolide & Isoambrettolide	Perfumery	—

1	2	3	4
25.	Dihydroisojasmone and Peach aldehyde	Perfumery chemicals	Released to two parties; both in production.
26.	Dimethylaniline	Dyestuff and explosives intermediates	Released to one party; in production.
27.	N,N-Dimethyl-biguanide-HCl (DMBG-HCl) Phenethylbiguanide -HCl(PEBG-HCl)	Anti-diabetic drugs	—
28.	Direct reading spectrophotometer/ colorimeter	Biochemical research spectroscopic analysis in visible range	Released to two parties. Both in production.
29.	Dissolving grade pulp (Ind. Pat. No. 82822)	Rayon, tyre cord	The process is offered on turn-key basis through Project Engineers.
30.	Ethylenediamine	Bulk organic chemical	Released to one party. Turn-key plant available through Project Engineers.
31.	Ethylenedichloride from ethyl alcohol	Solvent, organic intermediate	—
32.	Ethylene from ethyl alcohol	Organic intermediate	Released to one party as technical aid to industry.
33.	Exaltolide (Ind. Pat. No. 73702)	Perfumery	—
34.	Exaltone	Perfumery	—
35.	Ferrite-Hard	Electronics	Released to three Parties. One in production.
36.	Ferrites-Soft	Electronics	—

1	2	3	4
37.	Fine chemicals	—	Know-how available for 200 laboratory chemicals
38.	Flexible magnets	Refrigerator gaskets, toys educational kits	Released to three parties.
39.	Foundry core binder (sinol core binder)	Core binder in steel foundries for high demensional accuracy	Released to one party.
40.	Gaskets from coir pith	Gaskets	Released to one party.
41.	D-Glucosamine hydrochloride (C. P.)	Biochemical research, Pharmaceuticals	—
42.	Glyceryl guaiacolate	Drug-expectorant, intestinal antiseptic	—
43.	Gum arabic substitute	Adhesives	Released to three parties.
44.	Hexachloroethane (Ind. Pat. No. 92997)	Pyrotechnics, smoke screen, veterinary medicine, fluxing agent in foundries	Released to one party.
45.	β-Ionone (Ind. Pat. No. 77225)	Perfumery chemical, intermediate for Vitamin A	Released to two parties; one in production.
46.	Linseed oil emulsion paint (Ind. Pat. No. 117403)	Emulsion paints	—
47.	Microfilters	Industrial filtration	Released to one party.
48.	Molecular sieves	Chemicals, Petrochemicals cryogenic industry	—

1	2	3	4
49.	Monochloroacetic acid	Organic intermediate for weedicides, CMC, etc.	Released to one party.
50.	Monoethylaniline	Intermediate for explosives	Released to one party.
51.	Morpholine	Intermediate for rubber chemicals, textile chemicals, optical brighteners, etc.	—
52.	Neo-Lavandulol	Perfumery	—
53.	Nicotine sulphate (40% nicotine)	Insecticide	Released to two parties; one in production.
54.	p-Nitrophenol	Intermediate for parathion and paracetamol	Released to one party
55.	Optical whitening agent for synthetic fibres	Whitening agent for synthetic fibres	—
56.	Phenacetin	Drugs	—
57.	Phenoxyacetic acid	Penicillin V	—
58.	Phenylacetic acid	Perfumery, Penicillin G	Released to two parties.
59.	Phthalate-butyl octyl	Plasticizer in non-electrical applications	—
60.	Phthalates-dibutyl/ dioctyl	Plasticizers	Released to three parties; two in production.
61.	Phthalates-dimethyl/ diethyl	Plasticizers	Released to one party; in production.
62.	Polyurethane coatings (Ind. Pat. No. 121538)	Leather, rubber wood, glass, Nylon fabrics	—

1	2	3	4
63.	Polyurethane printing rollers (Ind. Pat. No. 86991)	Printing rollers	Released to three parties; one in production.
64.	Potentiometric strip chart recorder	Instrument for use in research and industry	Released to three parties.
65.	Radiosonde thermistors	Meteorology	Released to one party
66.	Reactive dyes	Dyestuff industry	—
67.	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.	—
68.	Rubber blowing agent (Dinitrosopenta methylene tetramine)	Rubber chemical	Released to one party; in production.
69.	Rubberized cork sheets from cork waste	Gaskets	Released to one party.
70.	Rubber reclaiming agent	Rubber chemical	Released to one party; in production.
71.	Sachets—Hot and Cold	Substitute for Hot water bag and Ice bag	Released to three parties; two in production.
72.	Simazine	Herbicide	—
73.	Sisal wax (Ind. Pat. Nos. 64958 & 65440)	Cosmetics, polishes	Released to four parties.
74.	Sodium hydrosulphide	Reducing agent in Textiles, sugar and soap industries	Technology on reaction only is offered
75.	Mannitol/sorbitol from cane sugar	Pharmaceuticals (Mannitol) Pharmaceutical syrups, Humectant (Sorbitol)	—

1	2	3	4
76.	70% Sorbitol from dextrose monohydrate	Pharmaceuticals Vitamin C Synthesis	Released to two parties; one in production.
77.	Staple pin adhesive	Adhesive for staple pins	—
78.	Synthetic gemstones	Jewellery, electric meters	—
79.	Tamarind kernel powder—phosphate and borate	Textile sizing substitute for hydrolyzed maize starch	—
80.	Terpineol	Perfumery	Released to one party.
81.	Theophylline, Aminophylline and Caffeine	Drugs (Caffeine also used in beverage)	—
82.	Thermistors	Temperature measurement and control electronic devices, etc.	Released to three parties; two in production.
83.	Thioglycolic acid	Cosmetics, catalyst	—
84.	2, 2, 4-Trimethyl-6-ethoxy-1, 2-dihydroquinoline	Rubber antioxidant	—
85.	Vitamin B_6 (Indian Patent No. 127750)	Drugs	Released to one party.
86.	Vitamin C	Drugs	Released to one party, in production.
87.	Xanthates-Potassium ethyl (PEX) and Potassium amyl (PAX)	Froth-flotation	—

Note: Know-how on items, 18,24,33, 34 and 52 could be available as an integrated project.

COMPARATIVE COST-BENEFIT ANALYSIS FOR 1972-73 & 1973-74

	1972—73 (*Rs. in lakhs*)	1973—74 (*Rs. in lakhs*)
ACTUAL EXPENDITURE		
1. Recurring expenditure	69.52	79.51
2. Capital expenditure	7.87	11.32
3. Pilot plant expenditure	2.34	—
	79.73	90.83
BENEFITS		
Receipts		
Premia and royalty	1.07	0.58
Receipts on account of sponsored projects	4.53	4.21
Analytical/testing charges	0.30	0.22
Institutional consultancy (CSIR share)	0.36	0.23
Fine Chemicals Sales	2.40	1.74
Miscellaneous receipts including job work	5.99	4.52
	14.65	11.50
Indirect benefits		
1. Total number of processes in production	48	49*
2. Value of production based on NCL know-how	557.11	651.39
3. Estimated savings in foreign exchange on account of above production	222.84	260.40
4. Total number of NCL processes released and waiting production.		
(a) NCL processes	34	25
(b) Sponsored schemes	28	23

* Parties who have not reported production for two consecutive years are excluded from this total.

5.	Total number of parties who have taken up NCL processes for exploitation.	136	146
6.	Total number of processes which were not released but which were available for commercial exploitation	51	44
7.	Number of processes released. during the year		
	(a) NCL processes	17	8
	(b) Sponsored processes completed	6	5
8.	No. of processes newly added to the list of NCL processes available for exploitation	4	4
9.	Research papers published	71	100
10.	Doctorate and Masters degrees awarded	22	11
11.	No. of recognized guides for Doctorate and Masters degrees	39	40
12.	Patents		
	(a) Indian patents in force	58	51
	(b) Foreign patents in force	7	6

Premia and royalties received by NRDC through NCL processes

	(a) Premia	3,79,249.00	2,39,500.00
	(b) Royalties	97,290.00	73,762.91
	No. of processes assigned to NRDC	15	4

Cumulative Cost-Benefit Analysis (1950-74)

COST		(*Rs. in lakhs*)
1.	Recurring expenditure	896.24
2.	Capital expenditure	173.79**
3.	Pilot plant expenditure	71.47
	Total expenditure (1950-74)	1141.50

** This figure does not include capital expenditure on NCL buildings amounting to Rs. 30.76 lakhs incurred by CSIR during 1949-50.

BENEFITS

1.	*Total Money receipts*	
	(a) Total premia earned by NRDC based on NCL processes	11.54
	(b) Total royalty earned by NRDC based on NCL processes	4.45
	(c) Total receipts from sponsors	66.72
	(d) Miscellaneous receipts including CSIR share of consultancy, analytical and testing charges, Fine chemicals sales & other receipts including job work.	60.25
		142.96
2.	Total value of production based on NCL know-how	2256.51
3.	Total No. of research papers published	2745
4.	Total No. of degrees received	350

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8. Administrative Officer,
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9. Accounts Officer,
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2.	Govt. departments including DGTD	8
3.	Public sector	9
4.	Sister Laboratories	2
5.	Internal advisers (NCL)	5
6.	Research Institutes, Universities, etc.	11
7.	Bhabha Atomic Research Centre	1
	Total	55

CREDITS

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