

REVIEW  
OF  
PROGRESS  
1950-70

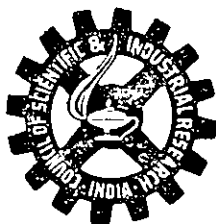
ANNUAL  
REPORT

1969-70

National Chemical Laboratory



**NATIONAL CHEMICAL LABORATORY  
POONA  
1969 - 1970**



**COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH**

# CONTENTS

## INTRODUCTION

## SPONSORED PROJECTS

## PRIVATE PARTIES

## INTERMEDIATES AND DYES

1. Quinacridone pigments .. 1

## TERPENOIDS

2. Development of perfumery products based on longifolene and isolongifolene .. 1

3. Development of perfumery products based on  $\Delta^3$ -carene .. 1

## DRUGS AND PHARMACEUTICALS

4. Carbimazole .. 2

5. 8-Hydroxyquinoline .. 2

## PERFUMERY CHEMICALS

6.  $\beta$ -Phenethyl alcohol .. 2

7. Coumarin .. 2

## INDUSTRIAL CHEMICALS

8. Production of rutile titania from Indian ilmenite .. 3

9. Maleic anhydride by the oxidation of benzene .. 3

## MISCELLANEOUS

10. Analysis of organic compositions of industrial importance .. 3

11. Screening of NCL compounds by Bristol Laboratories, Syracuse (N.Y.) for pharmacological activity	..	3
12. Technical preparation of liquid stabilizer for PVC	..	3
13. Technical preparation of butylated hydroxyanisole (BHA)	..	4
14. Development of infrared spectrophotometer	..	4

#### CENTRAL AND STATE GOVERNMENT INSTITUTES AND INDUSTRIES

15. Composite drug research scheme on Indian medicinal plants	..	4
16. Fabrication of Electron Diffraction Camera	..	5
17. Constitution of lac	..	5
18. Pulping of Kashmir soft-woods	..	5
19. Pulping of Kerala hard-woods	..	6
20. Chlorobenzenes	..	6

#### PL-480 SCHEMES

21. Chemical and thermodynamic properties of refractory materials at high temperatures	..	7
22. Wood phenolics with special reference to their use in chemotaxonomy and their biosynthesis by tissue culture studies	..	8

#### PILOT PLANTS

1. Industrially useful products from polysaccharides	..	11
2. Aniline	..	11

3. Technical preparation of organic intermediates	..	12
4. Fine chemicals project	..	12
5. Bulk organic chemicals	..	13
<b>PRODUCT ORIENTED RESEARCH</b>		
1. Cadmium sulphide photoconductive cells	..	14
2. Tin oxide resistor	..	14
3. Ferrite compositions	..	14
4. Thick film materials	..	15
5. Potential catalyst materials	..	15
6. Magnetic properties of acicular $\gamma$ -iron oxide	..	16
7. Polycrystalline silicon	..	16
8. Silicones and silicone intermediates	..	16
9. Ethyl silicate	..	17
10. Aluminium silicate as rubber filler	..	18
11. Aluminium hydroxide gel	..	18
12. Utilization of some mineral concentrates	..	18
13. Polytetrafluoroethylene (PTFE)	..	19
14. Fluorine chemicals	..	19
15. Superphosphate from low grade indigenous rock phosphates	..	19
16. Vitamin B <sub>6</sub> (Pyridoxine hydrochloride)	..	20
17. N, N-Diethyl- <i>meta</i> -toluamide	..	20
18. Colchicine	..	20
19. Terpin hydrate and terpineol	..	21

20. Camphene to camphor	..	21
21. <i>p</i> -Menthane hydroperoxide	..	21
22. Utilization of $\beta$ -himachalene	..	22
23. <i>Celastrus paniculatus</i>	..	22
24. Utilization of the by-products of opium alkaloid industry	..	22
25. Alkaloids from lanced poppy capsules	..	23
26. Hydroxyethyl starch	..	23
27. Cashewnut shell gum	..	24
28. Synthetic glycosides and other synthetic carbohydrate materials	..	24
29. Cellulose powder	..	25
30. Saccharification of cellulose	..	25
31. N-Acetylneuraminic acid	..	25
32. Starch phosphate	..	25
33. Foundry core binder	..	25
34. Substitute for gum Arabic	..	26
35. Chemical stabilizer for desert and sea-beach sands	..	26
36. Tamarind kernel powder	..	26
37. Low temperature fluxes	..	26
38. Stabilized gel for hot and cold sachets	..	27
39. Dyes for synthetic fibres	...	27
40. Organophosphorus compounds	..	27
40.1 Trioctyl phosphate	..	28

40·2 Triphenyl phosphate	..	28
40·3 Tris-nonylphenyl phosphite	..	28
41. Simazine and Atrazine	..	28
42. Fraudulently altered bank cheques	..	29
43. 'Sonobuoy' fluorescent dye marker	..	29
44. Shark repellent compositions	..	29
45. Photosensitive chemicals	..	29
46. Theophylline-caffeine	..	30
47. <i>p</i> -tert-Butyl catechol	..	30
48. Catechol	..	31
49. <i>p</i> -Nitrophenol	..	31
50. Purification of crude anthracene	..	31
51. Carbazole Dioxazine Violet	..	32
52. Vitamin D <sub>3</sub>	..	32
53. Utilization of methyl benzoate	..	32
54. Semi-synthetic penicillins—ampicillin and cloxacillin	..	33
55. Phenylacetic acid	..	33
56. Stationary liquid phases and solid support for GLC instruments	...	33
57. Nitrile rubber	..	34
58. Polystyrene foam	..	35
59. Synthetic polymers for cane juice clarification	..	35
60. Microfilters	..	36

61. Synthetic polymers for oil well drilling	..	36
62. Can lining composition	..	36
63. Linseed oil emulsions	..	37
64. Coating for nylon fabric	..	37
65. Polyisocyanates for coatings and adhesives	..	37
66. Rubber base adhesive	..	37
67. Composite propellant	..	38
68. Materials for artificial limbs	..	38
69. Polysulphide rubber	..	38
70. Sulphochlorinated polyolefin elastomers (SCPE)	..	38
71. Sorbitol and mannitol from invert sugar	..	39
72. Chlorination of methane gas	..	40
73. Plasticizers	..	40
74. Ethylenediamine	..	41
75. Self-balancing strip chart recorder	..	41
76. ESR spectrometer	..	41
77. Preparative VPC	..	42
78. Ultrasonic interferometer	..	42
79. Recording polarograph	..	42
80. Osmotic microbalance	..	43

## RESEARCH PROJECTS

1. Studies in solid state	..	44
1·1 Ligand field theory	..	44



1·2	Studies on spinels	..	44
1·3	Photoconducting materials	..	45
1·4	Dielectric properties and molecular structure of organic compounds	..	45
1·5	Diffusion in solid state	..	45
2.	Nuclear and radiation chemistry	..	46
2·1	Studies on Mossbauer effect	..	46
2·2	Radiolysis of aromatic hydrocarbons	..	46
3.	Thermodynamic studies	..	47
3·1	Thermodynamic properties of solutions	...	47
3·2	Thermodynamics of metal ion complex formation in mixed dioxane-water solutions	..	48
4.	Crystal and molecular studies	..	49
4·1	Crystallography	..	49
5.	Thin films	..	50
5·1	Structure of thin films	..	50
5·2	Physics of thin films	..	50
6.	Synthetic inorganic chemistry	..	52
6·1	Titanium and tin organics	..	52
6·2	Coordination compounds	..	53
6·3	Fluorine chemistry	..	54
7.	Physico analytical chemistry	..	54
7·1	New analytical reactions	...	54
8.	Natural organic products	..	56

8·1	Oleoresins	..	56
8·2	Functionalization of saturated aliphatic and alicyclic compounds	..	57
8·3	Photochemistry of organic compounds	..	57
8·4	Compounds of vegetable origin for insect control	..	57
8·5	<i>Shouwia arabica</i>	..	58
8·6	<i>Vetiveria zizanoides</i>	..	58
9.	Synthetic organic chemistry	..	59
9·1	Transformation products of costunolide	..	59
9·2	Chemical transformations of dehydrocostus lactone	..	60
9·3	Compounds related to selinane, elemene and <i>p</i> -menthane	..	60
9·4	Sarkomycin	..	60
9·5	Flavonoids	..	60
9·6	Naturally occurring anthraquinone pigments	..	60
9·7	Marschalk reaction	..	61
9·8	Synthetic dyes	..	61
9·9	Studies in heterocyclic compounds	..	63
9·10	Antifertility drugs	..	65
10.	Physical organic chemistry	..	66
10·1	Studies on conjugated systems	..	66
10·2	Carbonium salts	..	67

10·3	Structural and stereochemical studies	..	67
10·4	Electronic spectra and electronic structure	..	67
10·5	Conformational studies	..	67
10·6	Mass spectrometry	..	68
11.	National collection of industrial microorganisms	..	69
12.	Growth of plant and animal cells	..	69
13.	Enzymes	..	70
13·1	Hexokinase	..	70
13·2	Phytase	..	71
13·3	DPNase	..	71
13·4	Acylphosphatase	..	71
13·5	Enzymes and the metabolism of organic acids	..	72
13·6	Metabolism of nitrate by <i>A. fischeri</i>	..	72
13·7	Citrate-oxaloacetate lyase (Citrase)	..	72
13·8	Screening of microorganisms for the preparation of industrially important enzymes	..	73
13·9	DFP-Susceptible enzymes and antidotes	..	74
14·	Physico chemical studies in polymers	..	74
14·1	Stereospecific polymerization	..	74
14·2	Oxetane polymers	..	75
14·3	Degradation of long chain molecules by ultrasonics	..	76
14·4	Polymer characterization	..	76

15·	Chemical engineering studies	..	7
15·1	Fluidization	..	76
15·2	Mass transfer	..	77
15·3	Direct liquid-liquid heat transfer	..	77
15·4	Diffusion in solid catalysts	..	77
15·5	Reaction models and reactor design	..	77
15·6	Distillation	..	78

## APPENDICES

A-I	Service projects	..	79
A-II	Services rendered to outside parties	..	83
A-III	Demonstrations	..	85
A-IV	Processes leased out during 1969-70	..	86
A-V	Sponsored projects completed during 1969-70	..	86
A-VI	Receipts through sponsored projects	..	87
A-VII	Training	..	87
A-VIII	Seminars and lectures	..	88
A-IX	Staff news	..	90
A-X	Publications	..	96
A-XI	Patents in force	..	109
A-XII	Products manufactured on the basis of NCL know-how	..	116
A-XIII	Processes released and awaiting production	..	125
A-XIV	Processes approved for release to industry by 'Process Release Committee', NCL	..	133

A-XV	Know-how available	..	138
A-XVI	Staff strength (1969-70)	..	141
A-XVII	Cost-benefit analysis for 1969-70	..	142
A-XVIII	NCL-Executive Council Members	..	144
A-XIX	Our Advisors	..	145
REVIEW OF PROGRESS-1950-70			.. 149

## INTRODUCTION

The Report covers the period April 1969-March 1970. The activities and achievements of R & D work are presented under four sections: sponsored projects, pilot plant projects, product oriented research, and research projects.

During the year, work on 11 sponsored projects has been successfully completed and work on 8 new projects was undertaken. Currently (August 1970), work on 21 projects is in progress. Of these, 13 are sponsored by industry, 6 by State and Central Government Agencies, and 2 are being supported by PL-480 grants. The receipts on account of sponsored projects for 1969-70 amounted to Rs. 7.17 lakhs (as against Rs. 6.22 lakhs for 1968-69).

Five pilot plant projects were operated during 1969-70. The Fine Chemicals Unit has, for the first time, made a profit of Rs. 0.22 lakhs on the total sales of Rs. 2.71 lakhs. Bulk organic chemicals project was commenced in early 1969 and is based on raw materials from the petrochemical industry.

Of the 80 product oriented projects, 6 relate to solid state materials, 9 to inorganic and organo-metallic products, 41 to natural and synthetic organic chemicals, 18 to rubber and polymeric materials and 6 to development of instruments.

The Laboratory has also been actively engaged in basic research in various disciplines of chemistry and chemical engineering. This work is described in the fourth section of the Report. As a result, 97 research papers have been published in Indian and foreign journals and 23 scientists (7 Research Fellows, 2 guest workers and 14 staff members) received post-graduate (M.Sc. and Ph.D. etc.) degrees.

In the Appendices, Table I lists the NCL processes under commercial exploitation. As against the value of production of Rs. 70 lakhs from 47 processes in 1968-69, the value of production based on NCL processes in 1969-70 showed nearly a two fold rise, the production value being over Rs. 130 lakhs from 53 processes. 40% of the above (Rs. 48.8 lakhs) may be taken as the foreign exchange saved by the above indigenous production.

Table II gives information on 35 processes which are released and expected to go into production in the near future.

Table III enlists 39 processes approved for release so far by the NCL Process Release Committee (functioning since September 1967); 11 of which have already been released to the industry and negotiations for the others are in progress.

The following highlights of technology transfers which have fructified during 1969-70 may be mentioned:—

1. The acetanilide plant has now been taken over by the Hindustan Organic Chemical Ltd., Rasayani, management from the technical control of the Project Engineers and NCL chemical engineers. Subject to availability of aniline the plant can now work to full capacity and indeed the production which will be available from the plant will be more than earlier anticipated. Even during the discontinuous operations during 1969-70, 247 tonnes of acetanilide valued at Rs. 15.5 lakhs was sold by the company.
2. M/s Alta Laboratories, Khopoli, have commenced production of DOP based on NCL know-how and the production during the year 1969-70 was 197 tonnes valued at Rs. 14.6 lakhs. The production is expected to be much more this year if raw materials are freely available.
3. M/s Hypophosphite and Co's (Bombay) calcium hypophosphite plant based on know-how developed by the NCL on sponsorship by this firm has become fully operational this year and the production during 1969-70 was 9 tonnes valued at Rs. 4.50 lakhs. The plant is also capable of producing more of the product if phosphorous is available more freely.

Photographs of the above three plants are included in the Report.

In addition, information on sponsored projects completed and processes released during the year, patents and publications, members of the NCL advisory bodies, services rendered to outside parties, technical aid to the industry etc., is documented in the appendices.

#### **COST/BENEFIT ANALYSIS :**

We are aware that cost/benefit analysis of an industrial research laboratory, such as the NCL, which is functioning independently of an industrial enterprise is exceedingly difficult since the work of the laboratory forms only one of the components of a total system comprising the various links in the

technology transfer chain. The Laboratory has no control over the other components of this total system as against applied research conducted by industries. However since the objective of this laboratory is not only research but also development, an attempt is being made to be as useful to industry as possible in spite of the above formidable handicap.

Starting with this year's Annual Report an attempt is therefore being made at carrying out a cost/benefit analysis of the NCL for the year under review. This analysis is described in Appendix (XVII). As against the total inputs of Rs. 71.57 lakhs, direct benefits amounted to Rs. 10.41 lakhs. The ratio of receipts to recurring expenditure which was 16.6% in 1968-69 has now risen to 18.9% during 1969-70. In addition to the above direct benefits, the laboratory can claim credit for indirect benefits which have accrued to the nation on account of contribution towards establishment/increase in production/productivity, saving in foreign exchange on account of development of indigenous know-how and increase in scientific and technological capability of the country. These benefits have been highlighted by the following factual data:-

Total number of processes in production, total value of production, estimated saving in foreign exchange on account of above production, total cumulative value of production upto March 1970, research papers published during 1969-70, Doctorate and Masters degrees awarded during 1969-70, Patents— (a) New Patents filed (b) Total patents in force (c) Patents which are being commercially exploited and total number of technical enquiries which were answered.

#### REVIEW OF PROGRESS-1950-70 :

In January 1970, the NCL has completed twenty years of service to the nation. In this year's report we have therefore presented a review of progress made during the last 20 years. With the help of 9 tables and 11 graphs, an overall cost/benefit analysis of the work of the laboratory during 1950-70 has been made. The results of this analysis seem to indicate that scientific and technological output of the laboratory has increased considerably in recent years.

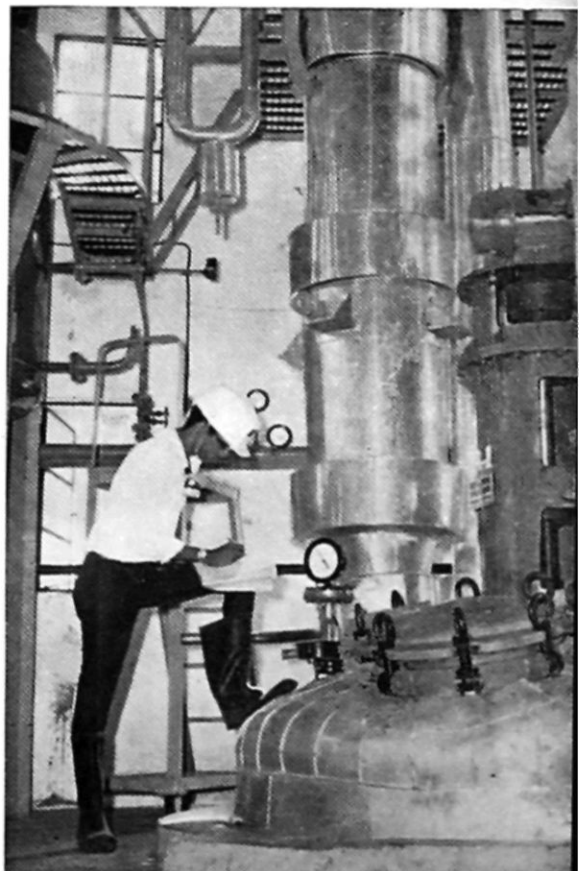
Modifications in administrative and research management policies introduced in the last few years which have been discussed in this review have brought rich dividends by way of making the laboratory much more productive as regards its contributions to the Indian chemical and allied industries.

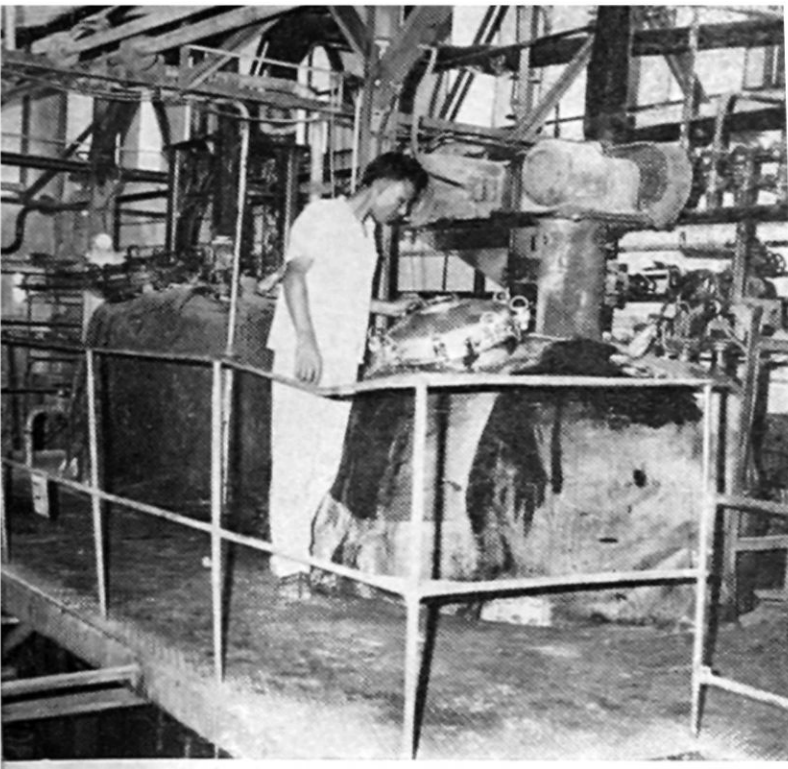


Quantification of benefits accruing from R & D investments has been cited as a Gordian knot of science policy by an expert analyst. Nevertheless since inputs are measurable in monetary terms, we consider it worthwhile to make an attempt to also quantify in monetary terms, wherever possible, the direct as well as the indirect benefits which may be expected from R & D investments in the NCL. The present report is our first attempt in this direction.

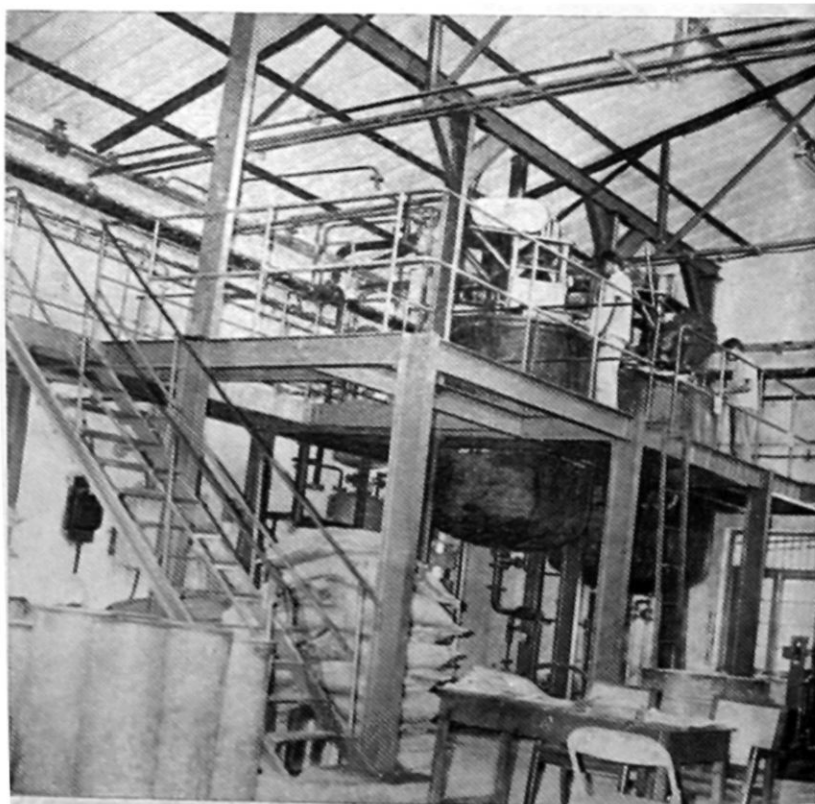


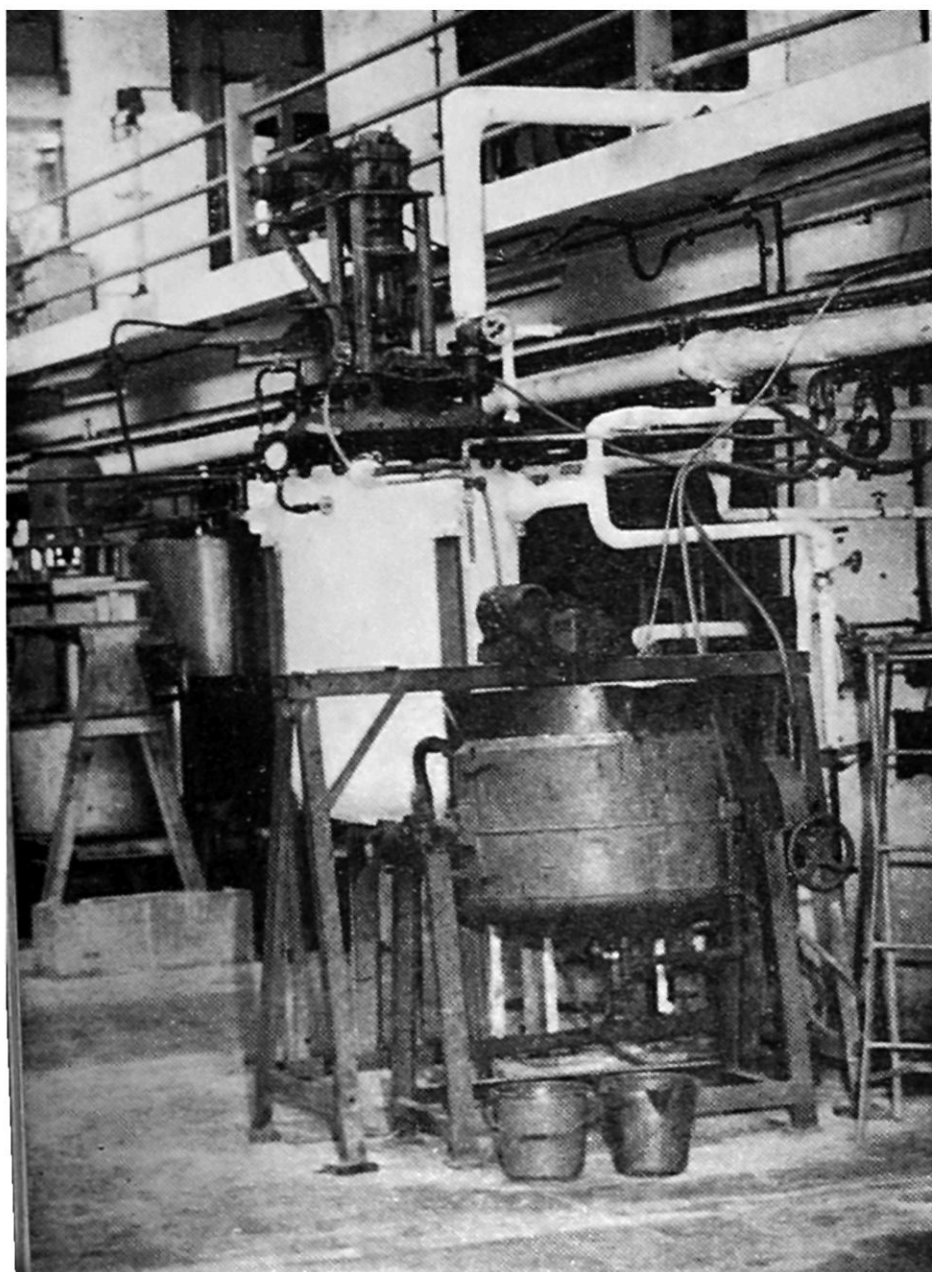
ACETANILIDE PLANT – HOC, RASAYANI





DIOCTYL PHTHALATE PLANT-M/s ALTA LABORATORIES, KHOPOLI





*p*-NITROPHENOL PILOT PLANT

# SPONSORED PROJECTS

## PRIVATE PARTIES

### INTERMEDIATES AND DYES

1. *Quinacridone pigments* : (SP-49/69)

Quinacridone pigments, with shades ranging from bright orange-red to purple, are characterized by very high allround fastness properties comparable to the phthalocyanines. These pigments are not manufactured in the country.

Work on this project has been undertaken since September, 1969.

### TERPENOIDS

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

Indian turpentine oil obtained from *Pinus longifolia* contains 2-10% of longifolene which has no utility. During the period under review longifolene has been converted into some derivatives having perfumery value and the details have been supplied to the sponsor.

The preparation of these derivatives was accompanied by undesirable isomerization of longifolene to isolongifolene and considerable formation of undistillable residue. These side reactions were prevented appreciably by using various catalysts under different conditions. Further work to improve the yields of the required products is currently in progress.

Attempts to obtain a useful perfumery product from isolongifolene (obtained as a major by-product in certain reactions of longifolene) were successful. This product was accepted as a perfumery chemical. The preparation of this product was optimized on a 1 kg. scale. The process details and the product were supplied to the sponsor. Further work is being carried out to get other useful derivatives.

3. *Development of perfumery products based on  $\Delta^3$ -carene* : (SP-54/70)

During the period 1966-68 four derivatives of  $\Delta^3$ -carene were prepared which were found suitable as perfumery compounds. Processes for preparation of these compounds were optimized for commercial exploitation.

In addition to this work, structure elucidation of these four compounds has also been initiated.

#### DRUGS AND PHARMACEUTICALS

##### 4. *Carbimazole* : (SP-11/65)

This drug is used in the treatment of hyperthyroidism and *angina pectoris*. It is considerably less toxic than most of the drugs used in the field.

Although this project was completed last year, it became necessary to modify the process with a view to avoiding the usage of one of the raw materials. Developmental work on the modified process for the manufacture of this drug is still under way. Of the four steps involved, two were standardized and the project would be completed in the coming year.

##### 5. *8-Hydroxyquinoline* : (SP-43/68)

This is an important intermediate for the manufacture of anti-dysentery drugs, and is largely imported at present.

After successful optimization, pilot plant trials (10 kg/batch) are now being conducted to demonstrate the process to the sponsor.

#### PERFUMERY CHEMICALS

##### 6. *$\beta$ -Phenethyl alcohol* : (SP-31/67)

As reported last year, necessary process data were collected and chemical engineering designs of a plant of 75 kg./batch capacity were supplied to the sponsor.

The firm has reported good progress towards implementation of the know-how and this important perfumery chemical would be commercially produced by the firm during 1970 (150 TPA valued at Rs. 45 lakhs).

##### 7. *Coumarin* : (SP-51/69)

This is an important perfumery chemical. Work on the standardization of an economical process for the manufacture of coumarin was initiated in March, 1970.

#### INDUSTRIAL CHEMICALS

##### 8. *Production of rutile titania from Indian ilmenite* : (SP-52/70)

Experiments on vapour phase oxidation of anhydrous titanium tetrachloride in the temperature range 1200° to above 1700° were successfully carried out and formation of rutile phase and its correlation to the quench-

ing temperature and retention time in the reactor were studied. Particle size (less than  $1 \mu$ ), bulk density and oil absorption of the titania obtained were determined.

The laboratory work carried out so far attracted attention of a firm which has now sponsored this programme.

9. *Maleic anhydride by the oxidation of benzene* : (SP-46/69)

Maleic anhydride is the key intermediate in the manufacture of ester type resins, extensively used for lacquer and varnish compoundings. The chemical is not produced at present in the country and nearly 350 tonnes valued at Rs. 5-6 lakhs are being imported every year. Work on this project was started on a sponsored basis in June, 1969.

A detailed assessment of several catalysts was carried out on a laboratory unit, and a pilot plant reactor was set up for more intensive and continued assessment of the catalysts. The results obtained were encouraging which would facilitate the selection of a suitable catalyst and the design of a maleic anhydride reactor that could be operated economically.

#### MISCELLANEOUS

10. *Analysis of organic compositions of industrial importance* : (SP-33/66)

A number of special organic compositions available under different trade names are used in various industries. Many of these are imported in substantial quantities and are listed in import statistics under the NES category. Analysis of these compositions to separate their components and identify them was undertaken at the instance of sponsors with a view to explore the feasibility of manufacture of similar compositions from indigenous raw materials.

During the period under report, work on a textile auxiliary sample and a dispersing agent was initiated.

11. *Screening of NCL compounds by Bristol Laboratories, Syracuse (N. Y.) for pharmacological activity* : (SP-35/67)

Under this scheme some 28 selected organic compounds isolated or synthesized in the NCL were sent for biological testing and evaluation during the period under review.

12. *Technical preparation of liquid stabilizer for PVC* : (SP-47/68)

A liquid Ba-Cd stabilizer for PVC using indigenous chemicals was

successfully developed on a laboratory scale, which was found satisfactory by the users. The scheme is now concluded.

13. *Technical preparation of butylated hydroxyanisole (BHA) :*  
(SP-50/69)

This is an anti-oxidant additive used in various fuel and lubricating oils. Optimum laboratory scale runs for the preparation of BHA were standardized. Large scale batches will now be taken up.

14. *Development of infrared spectrophotometer :* (SP-45/68)

Infrared spectrophotometer, a versatile instrument in research and analytical laboratories, is not produced in the country. The demand for a low priced (in the range of Rs. 40,000 to 50,000) instrument is likely to be around 100 units per year.

Two units of a working model of I. R. spectrophotometer were fabricated and the alignment procedures established. Work on improvisation of these models is in progress.

#### CENTRAL AND STATE GOVERNMENT INSTITUTES AND INDUSTRIES

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

Under this scheme, the chemistry of pharmacologically active extracts of some of the well known Indian medicinal plants is being studied. The isolated individual components were sent to another unit for pharmacological testing.

At present *Boerhavia diffusa* Linn (Punarnava) and *Asparagus racemosus* Willd (Shatavari) are being studied.

From the petroleum ether extract of Punarnava, for which diuretic activity was established earlier,  $\beta$ -sitosteryl palmitate, tetracosanoic acid, stearic acid, and palmitic acid were isolated. Ethylacetate extract obtained from the acetone extract of Punarnava was found to possess anti-inflammatory activity. Allantoin was also isolated from the alcohol extract of Punarnava.

From the alcohol extract of Shatavari four saponins were isolated. Amongst these, the saponin present in major amounts was found to have antioxytocin activity. The compound was found to contain the aglycone sarsapogenin and 5 to 6 sugar units of glucose and rhamnose. Two more glycosides obtained from the extract were purified by crystallization. Further work on these compounds is in progress.



16. *Fabrication of Electron Diffraction Camera* : (SP-21/65)

The fabrication of electron diffraction cameras was undertaken at NCL on special requests from Gauhati University, Gauhati; Fertilizer Corporation of India, Sindri and Defence Science Laboratory, Delhi. Sophisticated equipments of this type have to be imported at a high cost. In spite of difficulties in the import of components, and in the alteration of our design to suit indigenous production, three units were fabricated, assembled and tested to the satisfaction of the parties, of which two were dispatched and the third would be sent shortly.

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

After establishing the structures for most of the lac acids resulting from base hydrolysis of lac, useful information regarding the points of linkage of the component acids in pure lac molecule is being obtained by oxidative degradation of the pure fractions of lac.

By careful chromatography of the soft resin from lac, smaller basic units of lac were isolated, two of such units were characterized as : (1) jalaryl aleuritate ester formed through the secondary hydroxyl group of jalaric acid and the carboxyl group of aleuritic acid; (2) a similar ester from laccijalaric acid and aleuritic acid, laccijalaryl aleuritate. Experiments are in progress to synthesize these two naturally occurring esters.

Preliminary work was undertaken to find out the reasons for the poor keeping quality of shellac at higher temperatures, which results in the blocking of shellac. The main difficulty experienced during the export of this commodity is the poor keeping quality.

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

The scheme was undertaken on behalf of Government of Jammu and Kashmir to determine the suitability of some of the Kashmir soft-woods for the manufacture of tyre cord grade dissolving pulp.

After establishing conditions for rayon grade pulp from Kashmir soft-woods, work was further extended to obtain high purity tyre cord grade pulp. Pulps of tyre cord grade specifications could be obtained in high viscosity and low viscosity ranges.

Reproducibility of the established conditions was confirmed by taking a number of trials.

Conditions were established for getting  $\alpha$ -cellulose of 96 to 97% purity. The average pulp sample was tested by the sponsor in other laboratories and the results were satisfactory.

Larger pulp samples (25 kg.) were sent to parties in India and abroad for further evaluation.

Similar study of Kashmir soft-woods was carried out by acid-sulfite process and acid-prehydrolysis sulphate process. All these pulps were tested in the NCL for polydispersity and compared with commercial tyre cord grade pulps.

19. *Pulping of Kerala hard-woods* : (SP-38/67)

At the instance of the Pre-investment Survey of Forest Resources, Ministry of Food and Agriculture, Government of India, the suitability of several wood species for the production of dissolving pulps is being studied.

Accordingly, studies on twenty-two hard-wood species from Kerala forest were undertaken. These woods were separately chipped and then the chips were mixed in the proportion given by the sponsor. Pulping was carried out by water prehydrolysis sulphate process under different conditions. Optimum conditions were established which could be consistently repeated.

Yields :

Unbleached pulp yield on O. D. chips	.....	36.60%
After centricleaning on O. D. chips	.....	34.75%
On multistage bleaching, final yield on O. D. chips	.....	31.25%

Analysis of the final pulp was as follows :

α-Cellulose	95.04%
Pentosan	1.4%
Ash	0.07%
Fe	33 ppm
SiO <sub>2</sub>	22 ppm
Ca	26 ppm
D.P.	1000
Alc. ben. solubles	0.1561%
Viscose filterability-F <sub>s</sub>	25

The above results indicated that even a mixture of different hard-wood species could be used for the manufacture of rayon grade pulp.

20. *Chlorobenzenes* : (SP-48/69)

It was reported last year that on the basis of the developmental work carried out in the NCL, a 1 tonne/day pilot plant for the production of

monochlorobenzene (80%) and dichlorobenzenes (PDCB & ODCB 20 %) was installed and operated by a firm in Bombay. A turn-key proposal has now been submitted to the HOC, Rasayani.

Further, a new project was sponsored by HOC for the development of a continuous process yielding monochlorobenzene as the predominant product. For this purpose the earlier pilot plant of 10 kg./hr. capacity was suitably modified and conditions were standardized for the desired product composition. The necessary design and engineering data were collected for setting up of an industrial unit.

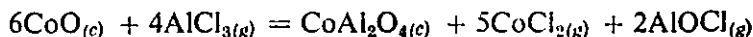
## PL-480 SCHEMES

### 21. *Chemical and thermodynamic properties of refractory materials at high temperatures : (SP-27/64)*

High temperature reactions of anhydrous metal halides (viz: halides of Al, Ni, Mn, etc.) with the corresponding metals and also with metal oxides (viz: NiO, Mn<sub>3</sub>O<sub>4</sub>, MgO) were studied employing the transpiration technique. These investigations helped to determine the thermodynamic properties (heats of formation, entropy, etc.,) of the unstable species viz: MnCl<sub>(g)</sub>, NiCl<sub>(g)</sub> and also solid spinels such as NiAl<sub>2</sub>O<sub>4</sub>, MgAl<sub>2</sub>O<sub>4</sub>. During the period 1969-70, some more systems involving AlF<sub>(g)</sub>, AlOCl<sub>(g)</sub> and CoAl<sub>2</sub>O<sub>4(s)</sub> were also studied.

The high temperature vacuum microbalance fabricated in the laboratory functioned normally. Vaporization of molten silver and nickel powder was studied with the help of Knudsen effusion cell. Studies on Langmuir free evaporation were also carried out employing Nickel foil.

- (A) The reaction between gaseous AlF<sub>3</sub> and molten aluminium metal Al<sub>(l)</sub> + AlF<sub>3(g)</sub> = AlF<sub>(g)</sub> was studied between 1245° and 1375° K. From the above reaction  $\Delta H_{f298}$  for AlF was found to be - 118 Kcal./mole.
- (B) The equilibrium of the reaction of Al<sub>2</sub>Cl<sub>6(g)</sub> with light-lanthanum sesquioxides (La<sub>2</sub>O<sub>3</sub>, Nd<sub>2</sub>O<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub>) was investigated and the standard heat of formation H<sub>f298</sub> of the rare earth metaloxchlorides calculated.
- (C) The reaction of cobaltous oxide with AlCl<sub>3(g)</sub> was studied between temperatures 1120° - 1360° K. The experimental data show the following stoichiometry for the reaction :



The standard heat of formation  $H_{f298}$  for  $AlOCl$  was calculated from the experimental results. The value obtained was  $H_{f298} = -80.4 \pm 1.4$  Kcal./mole.

- (D) Studies on molten silver using graphite Knudsen cell were carried out between  $1253^\circ$  and  $1433^\circ$  K and it was observed that the vaporization of the metal depends on the surface condition of the molten metal i.e., the accommodation coefficient.

Experiments on vaporization of nickel were also carried out both by the Knudsen effusion and the Langmuir free evaporation techniques in the temperature range  $1303^\circ$  to  $1713^\circ$  K. The vapour pressures were found to depend on the orifice areas of the Knudsen cells employed. The accommodation coefficient at  $1523^\circ$  K has been calculated as follows :

$$P \text{ (Langmuir)} = 3.514 \times 10^{-4} \text{ mm. of H}_g \\ \text{(Orifice area} = 1.363 \times 10^{-3} \text{ sq. cm.)}$$

$$P \text{ (Knudsen)} = 1.874 \times 10^{-2} \text{ mm. of H}_g$$

$$\text{Accommodation Coeff.} = \frac{PL}{P_k} = 1.876 \times 10^{-2}$$

22. *Wood phenolics with special reference to their use in chemotaxonomy and their biosynthesis by tissue culture studies : (SP-28/66)*

Work on the heartwood and bark constituents of four Indian species of *Morus* (*alba*, *serrata*, *indica* and *laevigata*) was continued, and work was undertaken on *M. rubra*, a North American species. An efficient method of separation of the phenolics was established. From the heartwoods of *M. alba*, *M. serrata* and *M. indica*,  $\beta$ -resorcyaldehyde, resorcinol, quercetin, morin, dihydromorin, dihydrokaempferol, resveratrol, oxyresveratrol, and dihydroxyresveratrol were isolated. The heartwood of *M. laevigata* did not contain dihydromorin, dihydrokaempferol and resveratrol, but afforded two new compounds, 3, 4'-dihydroxydihydrostilbene and 6, 3', 5'-trihydroxy-2-phenylbenzofuran; it also contained dihydroresveratrol, synthesized earlier, but unknown as a natural product. The structure of the benzofuran was confirmed by synthesis from oxyresveratrol. From the heartwood of *M. rubra*,  $\beta$ -resorcyaldehyde, resorcinol, morin, dihydromorin, quercetin, kaempferol, dihydrokaempferol, oxyresveratrol, piceatannol, and a new flavanone, norartocarpanone (5, 7, 2', 4'-tetrahydroxyflavone) were isolated.

An attempt to synthesize cyclomulberrochromene by cyclodehydrogenation of mulberrochromene and mulberrin with dichlorodicyanoquinone (DDQ) gave a novel 7-membered oxygen heterocycle.

From the heartwood of *Artocarpus lakoocha*, artocarpin, cycloartocarpin, and a new flavone, norcycloartocarpin, were isolated, besides oxyresveratrol. From the heartwood of *A. chaplasha*, in addition to the phenolics reported earlier, a novel 7-membered ring compound was also isolated. A new sample of the heartwood of *A. heterophyllus* from Kerala contained norartocarpin, not encountered earlier. An attempt to convert dihydroartocarpin to dihydrocycloartocarpin by cyclodehydrogenation with DDQ gave a 7-membered oxygen heterocycle. When artocarpin was treated with *p*-toluene sulphonic acid in refluxing benzene a novel 8-membered oxygen-containing heterocyclic compound was obtained. A benzene extract of the bark of *A. heterophyllus* gave betulinic acid, cycloartocarpin, artocarpin, and a new flavone, cycloheterophyllin, m. p. 204° — 206°, whose structure was established by spectroscopic methods as a derivative of cyclomulberrochromene with a dimethylallyl group in the 8-position and an additional hydroxyl group in the 5'-position.

Work on *Garcinia* species was continued. From the bark of *Garcinia morella*, isomorellic acid, morellinol, morelloflavone, dihydromorelloflavone, and a new compound, m.p. 260° — 265° (decomp), were isolated. From the bark of *G. xanthochymus*, morelloflavone and dihydromorelloflavone were isolated. Dihydromorelloflavone gave a heptamethyl ether, oxidation of which with selenium dioxide gave a heptamethyl ether of morelloflavone. Alkaline hydrolysis of *o*-heptamethyl dihydromorelloflavone led to products, which indicated a steric effect resulting in the greater stability of the flavanone ring substituted in the 3-position.

The isolation of the lignans, secoisolariciresinol and isotaxiresinol, from the heartwood of *Taxus baccata* was reported earlier. From the benzene extract two more phenols were isolated. From the acetone extract a new lignan, demethyl-lariciresinol, m.p. 158°, was isolated and its structure established.

In continuation of the work on the pines, the bark of *Pinus longifolia* was examined, and taxifolin, quercetin, and (+) catechin were isolated. Taxifolin and (+) catechin were identified in the bark of *P. excelsa*, from which four flavandiols were also isolated, but not yet in a pure crystalline state. An improved procedure for the isolation of pinoquercetin and pinomyricetin from the bark of *P. ponderosa* was developed.

From the bark of *Picea morinda*, quercetin, taxifolin, piceatannol, *p*-hydroxycinnamic acid, and two flavone glycosides under examination were isolated.

The bark of *Populus ciliata* gave  $\beta$ -sitosterol, quercetin, taxifolin, eriodictyol, isosakuranetin and naringenin. The heartwood of *P. ciliata* and

*P. nigra* contained very little extractable material, and the phenolic content was negligible.

The acetone extract of teak tissue showed the presence of four pigments. The major constituent was identified as 2, 5-dihydroxy-1-methoxy-3-methylanthraquinone by spectroscopic methods. Confirmation by synthesis is necessary in view of the biosynthetic aberration of the structure, and is in progress.

Investigation of the heartwood and bark constituents of *Fraxinus pennsylvanica*, *Juniperus macropoda*, *Nyssa aquatica* and *Adina cardifolia* is in progress.

#### *Tissue culture*

On chemical examination, the callus cultures of *Populus nigra*, *Morus alba*, *Artocarpus heterophyllus* and *Tectona grandis* did not show the presence of any known phenolics found in the respective woods. A new triterpene ( $C_{32}H_{50}O_4$ ) from *P. nigra*,  $\beta$ -sitosterol (about 0.001% on the dry weight of the tissue) and a flavan-3,4-diol from *M. alba*, and several related quinones from *T. grandis* tissues grown *in vitro*, were isolated. The characterization of some of these compounds is in progress. A quantitative variation in the pigment production of *M. alba* callus grown in media supplemented with different auxins is under investigation.

□   □   □

## PILOT PLANTS

### 1. *Industrially useful products from polysaccharides* : (PP-2/58)

The project includes investigations on indigenous cellulosic raw materials for the manufacture of dissolving pulps of various grades and manufacture of industrially useful polysaccharide materials.

#### *Cellulose pulp as a filler in plastics*

It is reported that about 800 tonnes of cellulose pulp are imported every year for use as a filler in plastics. Also about 300 tonnes of cellulose powder are used in different industries. Cotton linters were studied for this type of pulp.

In all, two commercial samples of second-cut cotton linters were processed by alkaline purification under pressure, both with and without addition of surface active agents. Bleaching of the linter pulp was carried out under different sequences including chlorine dioxide.

The pulp sample was then evaluated by industry which reported satisfactory results.

#### *Flowered bamboo pulp*

Flowered bamboo chips obtained from M/s Central Pulp Mills, Fort Songad, were tested by two-stage alkali pulping process and a pulp which corresponded to the desired specifications was obtained.

#### *Eucalyptus hybrid pulp*

Work on *eucalyptus hybrid* by prehydrolysis sulphate methods was completed. Experimental work on improving quality of *eucalyptus hybrid* pulps for tyre-cord resulted in acceptable chemical analysis of pulps with  $\alpha$ -cellulose upto a maximum of 96%.

### 2. *Aniline* : (PP-4/63)

The development of a continuous pilot plant of 5 kg./hr. of aniline and the performance of the NCL catalyst were reported earlier. It was also reported that the process was released to a project engineering firm, who had offered a turn-key plant of 600 TPA of aniline to Hindustan Organic Chemicals, Rasayani.

During the year under report, regeneration aspects of the deactivated catalyst were studied further with reference to the rate of oxidation of the carbon deposit by air in a single tube reactor. Operation regeneration-cycles were studied in considerable detail. After two successive regenerations it was observed that the catalyst did not deactivate even after a period of about 50 days. All the required data were obtained for the design of a commercial unit and handed over to the project engineering firm.

Kinetic experiments were conducted with previously poisoned catalyst at three temperatures and the necessary data obtained at different levels of poisoning.

This project has now been concluded.

### 3. *Technical preparation of organic intermediates* : (PP-6/63)

This is a multipurpose pilot plant project for scale-up of batch type processes developed in the laboratory. Processes for pilot plant work in this project are selected on the basis of import and demand pattern of a chemical in the country and interest shown by industry for its commercial exploitation.

A few more large scale batches (50 kg.) of *o*-tolylbiguanide were carried out and the process was licensed to M/s. Industrial Perfumes Ltd., Bombay. Tris-nonyl phenyl phosphite was prepared on 150 kg. per batch scale. Pilot plant batches on *o*-nitrophenol (10 kg.), *p*-nitrophenol (25 kg.), *o*-aminophenol (1 kg.), catechol (25 kg.), *p*-tert-butyl catechol (12.5 kg.) and chloroacetic acid (30 kg.) were carried out. Negotiations are in progress to sell these processes to industry. Work on *o* nitrophenol and *o*-aminophenol forms a part of the sponsored project on 8-hydroxyquinoline.

### 4. *Fine chemicals project* : (PP-7/64)

For the first time since its inception in 1964, the Fine Chemicals Unit has now made a profit of Rs. 0.22 lakhs on the total sales of Rs. 2.71 lakhs. During the current year no new compounds were introduced and the emphasis was on making the project economically viable.

Comparative figures of production and sales (in lakhs of rupees) for the past four years are given below :

<i>Year</i>	<i>Production</i>	<i>Sales</i>
1966—67	2.83	1.87
1967—68	2.08	1.46
1968—69	2.43	2.42
1969—70	2.35	2.71



5. *Bulk organic chemicals : (PP-9/69)*

The project deals with the development of indigenous technology for the preparation of basic organic chemicals required in large quantities. Since the raw materials for these chemicals will be available from the petrochemical industry which is being set up in the country, it can be reasonably assumed that these processes will be economically viable provided the optimization is vigorously carried out.

Work on this pilot plant project was commenced in early 1969 and developmental work on chlorobenzene, ethylenediamine and terephthalic acid was continued. Work on the first two items has now been reported separately.

Terephthalic acid is an extremely important chemical used in the production of polyethylene terephthalate. Techno-economic data on the requirements of polyethylene terephthalate and terephthalic acid etc., are being collected. There are three commercial methods of manufacturing terephthalic acid. Whereas all these methods will be examined for their comparative merit, the method involving disproportionation of benzoic acid appears to more attractive and will be studied in detail.

To start with, experiments were carried out on the disproportionation of potassium benzoate using different diluent and catalyst systems, pressures and temperatures. All these runs were taken in a 5 ltr. autoclave wherein the maximum yield obtained was 56%. In order to improve stirring and avoiding charring and lump formation, a ball mill type reactor was designed and fabricated and runs carried out. Methods for the analysis and processing of the reaction products have also been established and efforts are being made to improve the performance of the reactor to optimize yields.

□ □ □

## PRODUCT ORIENTED RESEARCH

### 1. *Cadmium sulphide photoconductive cells* : (ATT-2/66)

The NCL process for the preparation of cadmium sulphide photoconductive cells is now released to one firm for commercial exploitation.

During the period under review, improvements in encapsulation technique were introduced by carrying out moulding in two stages. It was found that this increases the lead strength considerably.

Controlled preheating of the charge, before crystals are grown, was found to have a beneficial effect on the yield of crystals. Pulse technique, in which high voltage, short duration electrical pulses are applied to the crystal, was successfully used to improve contact characteristics of the photocell.

A demonstration of the process was given to the licensee.

### 2. *Tin oxide resistor* : (ATT-61/68)

In view of similar work which is in progress at the CECRI, Karaikudi, work on this project is suspended. However, some basic investigations which were already initiated will be continued for some time.

### 3. *Ferrite compositions* : (ATT-10/59)

#### *Hard ferrites*

Barium ferrite is used to prepare the permanent magnets by usual ceramic techniques. The composition usually gives an energy product between  $0.9$  to  $1.0 \times 10^6$  gauss/oersteds. Improvements were made by making use of various additives. Thus the energy product at  $\sim 1.3$  to  $1.5 \times 10^6$  gauss/oersted was achieved. These improved magnetic materials are better than other imported ferrite magnets.

The BH product has been further increased by orientation of the pre-fired magnetic particles. The BH product achieved so far is about  $3.3 \times 10^6$  gauss/oersteds. Further improvements relating to the orientation are in progress.

The NCL process for hard ferrites is released to a firm, and successful trial runs at the factory were taken. The firm is awaiting the arrival of the machinery before going in for full production.

### *Soft ferrites*

A Ni-Zn-Co ferrite composition containing less nickel than conventional products suitable for short-wave antenna rods was developed. The process is now ready for commercial exploitation.

#### 4. *Thick film materials* : (A-B75/69)

Thick film technology is gaining in importance in the microelectronic industry, particularly in the field of integrated hybrid circuitry, where the combination of screened glaze elements like conductors, resistors, capacitors etc., with other discrete devices offers many advantages. In view of their increasing applications, there is likely to be a good demand for thick film devices in the country.

The object of the work is to develop formulations of thick film materials, screen print on ceramic substrates, and measure the physical properties of the glazes.

It is reported that thallium oxide resistive glazes have several advantages over those of Pd-Ag. Work on the former to standardize the conditions of preparation, printing and measurements was started and several such compositions were prepared. Resistivity of these glazes ranged from 20 ohms per square centimeter to one megohm per square centimeter. Several conductive pastes and glass frit with the required properties were prepared. Screen printing facilities are now being explored and once these are standardized, different oxide mixtures will be tried.

#### 5. *Potential catalyst materials* : (AB-8/67)

##### *Molecular sieves:*

Synthetic zeolites (molecular sieves) are alkali metal aluminosilicates which are widely used as adsorbents and catalysts in petrochemical and related industries. These are also used for drying and purification of gases in a number of industrial processes.

The purpose of this project is to prepare the zeolites and to evaluate their adsorption and catalytic properties.

Starting from indigenously available materials, crystalline sodium aluminosilicate was prepared. The X-ray pattern indicated that the product is similar to an imported variety. The D.T.A. and T.G.A. studies were also carried out. The extent of sorption of nitrogen, argon and water vapour on the zeolite is under investigation.

#### 6. *Magnetic properties of acicular $\gamma$ -iron oxide* : (AB-45/68)

$\gamma$ -Ferric oxide of this crystal habit is the type of magnetic material which is particularly suitable for coating magnetic recording tapes. It is estimated that 6.5 TPA of magnetic materials costing about Rs. 1 lakh are presently imported in the country. With the growth of recording tape industry, the demand is likely to rise to about 30 TPA in the next few years. The present work is undertaken to prepare and study acicular  $\gamma$ -iron oxide in relation to its particle size and shape.

$\gamma$ -Ferric oxide was prepared earlier through controlled decomposition of ferrous oxalate. Another process for its manufacture was also developed through the conversion of scrap-iron into hydrated ferric oxide which is first reduced to magnetite and then oxidised to  $\gamma$ -ferric oxide. The magnetic properties (coercivity = 434 Oe., remanence = 1669 gauss) of this product is found to be within the range required for materials used for coating magnetic recording tapes. Particle size and shape studies are in progress.

#### 7. *Polycrystalline silicon* : (ATT-9/67)

Silicon is a material of utmost importance in modern electronics industry and will remain indispensable for many years to come. From pure polycrystalline silicon single crystals are grown for semiconductor industry, and are used for making rectifiers, transistors, integrated circuits etc. Several firms have established production of silicon devices from imported silicon. The present demand of polycrystalline silicon in the country is estimated to be about 500 kg./year valued at Rs. 15 lakhs.

Work on the preparation of hyper-pure silicon is being carried out at several places in India. These are the (1) Solid State Physics Laboratory, Delhi; (2) National Physical Laboratory, New Delhi, and (3) Bhabha Atomic Research Centre, Bombay (BARC). The first two laboratories are working on the iodide process whereas BARC is working on the hydrogen reduction of trichlorosilane. In the NCL, therefore, it has been decided to work on the preparation and thermal decomposition of silane ( $\text{SiH}_4$ ). The method, though more hazardous, gives a product better in purity and in higher overall yield. Two runs were successfully completed on 25 gm. scale. The run takes only two hours and the yield is about 95% of the theoretical value.

A special room designed and equipped for extreme purity control is under construction.

#### 8. *Silicones and silicone intermediates* : (ATT-13/66)

Essential techno-economic data and scope of this five-year research

project were presented in the last year's Report. The scheduled programme for the year suffered a setback on account of non-availability of pure methyl chloride gas for the greater part of the year. Esterification studies were, however, carried out from synthetic chlorosilane mixtures and from small samples of Rochow mixtures obtained from a few experiments on direct synthesis.

The Rochow liquid, a complex mixture of methyl chlorosilanes, can be fractionally distilled without much difficulty to give a major fraction consisting principally of dimethyl dichloro and methyl trichloro silanes. A method was first standardized for converting this fraction into a mixture of the corresponding methoxy/ethoxy esters, which have much larger differences in boiling points than between the methyl chlorosilanes themselves, and are non-corrosive liquids fairly stable in air. Fractionation of these ester mixtures in simple packed columns gave good separation of the constituents (80% overall recovery of D from synthetic binary mixtures). In contrast with the dichlorosilane, subsequent hydrolysis of the diester to the low polymer siloxane intermediate  $(\text{Me}_2\text{SiO})_n$  (n may vary from 3 to 9) was mild and easily controlled. The methyl triesters, in their turn, were found suitable for reaction with a phenyl Grignard to give methyl phenyl diesters which form the corresponding methyl phenyl siloxane low polymer on hydrolysis.

Since the beginning of 1970, crude Rochow liquid mix is being produced routinely in the laboratory at the rate of 60-100 gm./hr. in a specially designed reactor. The dichloro dimethyl silane content of the liquid mix, based on the recovery of the siloxane low polymer, appears to have reached 50% (actual dichloro content in the chlorosilane mixture will be higher). Further work is designed to increase the dichloro dimethyl silane content in the Rochow mix, and to improve the recovery value of the dimethyl constituent (in the form of siloxane low polymers) to above 80% from the mix.

The progress of work on this project in the NCL and two other research institutions in the country is periodically under review by the Planning Commission.

9: *Ethyl silicate* : (ATT-120/68)

Samples of ethyl silicate-40 were sent to two places for evaluation tests; the results of the tests are awaited. Parties interested in exploitation of the NCL process may now obtain free samples on request.

On the use of ethyl silicate-40 in investment castings, M/s. Mysore Kirloskar Ltd., Harihar, have expressed the view that the new technology of investment castings is likely to develop fast in the next few years and that ethyl silicate-40 has a bright future.

The laboratory details for the preparation of ethyl silicate-40 can be made available to interested parties. Further laboratory runs are not contemplated but developmental work can be undertaken on a sponsored basis if industry shows interest.

10. *Aluminium silicate as rubber filler* : (ATT-121/69)

Aluminium silicate of uniform particle size is much valued as a rubber filler, in particular, in soleing materials of all types, since it specially toughens the raw mix with remarkably small need for accelerators and gives the vulcanizate a high hardness, possessing good physical properties. The demand for this material is entirely met by import. A firm in Poona alone uses about 36 TPA.

Comparative study of several samples of aluminium silicate indigenously produced and an imported sample showed that the Indian samples were inferior. Attempts are therefore being made to prepare a product comparable in properties to the imported sample.

11. *Aluminium hydroxide gel* : (ATT-122/69)

The demand for 'Alhydrogel' (100 TPA valued at about Rs. 10 lakhs) by the Indian Veterinary Research Institute, Mukteshwar, Kumaon, (UP) is entirely met by import. At the instance of the Institute, work was undertaken to develop methods for the manufacture of this item indigenously.

The problem of the preparation of a comparable sample of  $\text{Al}(\text{OH})_3$  to 'Alhydrogel' was attacked from various angles. In the preliminary investigations,  $\text{Al}(\text{OH})_3$  gel was prepared from alum or aluminium sulphate, in presence of ammonium chloride. In another experiment, alum or aluminum sulphate alone was used. Ammonium alum was also used but the  $\text{Al}(\text{OH})_3$  gel obtained was impure. After numerous trials a 2 kg. product was prepared which nearly matches with an imported sample. The product has been sent to IVRI for evaluation.

12. *Utilization of some mineral concentrates* : (ATT-66/68)

At the instance of the Sikkim Mining Corporation, Sikkim, work on the recoveries of zinc, copper and lead from their concentrates was undertaken last year. The pressure leaching of the zinc concentrate with dilute sulphuric acid was tried and a mild steel reactor fabricated in the workshop was used for the purpose. The analyses of the solution obtained, the residual pellets and the other residue showed that the leaching at 40 psi was optimum at which the solution contained about 35% Zn (82% extraction based on zinc concentrate) and 8% Fe. Attempts to carry out this experiment on a 2 kg.

scale in a 40 litre capacity lead lined steam-jacketed mild steel reactor with stirring arrangement, was not successful. Meanwhile it was gathered that the Sikkim concentrates are now fetching good price, as such by export to Japan and further work on the project is, therefore, suspended.

13. *Polytetrafluoroethylene (PTFE) : (AB-46/68)*

Laboratory experiments on the polymerization of tetrafluoroethylene (TFE) were carried out under different conditions of pressure, temperature, TFE/water and TFE/catalyst ratios. Ivory white PTFE not wetted by water and with desired bulk density was obtained, but in uneconomic yields.

In view of the heavy capital outlay on imported highly sophisticated equipments and also due to limitations of carrying out this work through laboratory and later pilot plant stages at the NCL, it was decided to discontinue this project. Work on the project may be resumed later only if sponsored by industry.

14. *Fluorine chemicals : (AB-15/63)*

Preliminary experiments on the preparation of  $\text{AlF}_3$  from fluosilicic acid were carried out using bauxite sludge as a raw material. Since M/s. Dharamsi Morarji Chemical Co. Ltd., Bombay, have already carried out similar work, further work on this project is discontinued. A write-up on the process for synthetic cryolite is being sent to interested parties.

15. *Superphosphate from low grade indigenous rock phosphates : (ATT-144/69)*

Since proven rock phosphate deposits of suitable quality are still very inadequate in the country, most of the requirements of phosphate rock are at present met by imports. A good part of the deposits available in the country are either of poor phosphate content or not sufficiently reactive. Examples are Maton rock phosphate and Jhamal Kotra rock phosphate which are not reactive enough to make available their phosphate content in a water-soluble form on conventional treatment with sulphuric acid.

Our investigation has indicated that the reactivity of the above rock phosphates can be significantly enhanced by the following simple procedure: Mixing the rock phosphate with ammonium nitrate followed by the usual reaction with sulphuric acid brings up the reactivity from 82 to 95%. In other words, 95% of the phosphate in the rock phosphate samples is solubilized. Since ammonium nitrate is an excellent nitrogenous fertilizer in its own right, and most of it is retained after reaction with sulphuric acid, the economy of

the process also does not suffer. Experiments to use nitrophosphate instead of ammonium nitrate in the above activation procedure are also under way.

It is proposed to investigate, whether other recently discovered rock phosphate deposits in the country also show enhanced solubilization by pre-treatment with minor additions of ammonium nitrate. The results promise to have a great significance on the problem of utilization of low grade phosphate rock deposits available in the country.

16. *Vitamin B<sub>6</sub> (Pyridoxine hydrochloride)* : (ATT-16/66)

Vitamin B<sub>6</sub> is not manufactured in India from basic raw materials. Total imports of this vitamin amount to Rs. 11 lakhs/year.

In the present work, *dl*-alanine is used as a starting material. Preparation of N-formyl derivative of ethyl *dl*-alaninate hydrochloride was optimized earlier. The following work was carried out during the period under review :

More attempts were made to optimize the preparation of the intermediate oxazole. Reproducibility upto 50% yield could be accomplished.

Conditions were optimized for the preparation of the title compound. 80-90% yields were obtained.

As a bye-pass to the stage of reduction of the cinchomerone, attempts were made to react the oxazole with a number of dienophiles viz., 2-butene-1, 4-diol, its diacetate and its cyclic acetal etc., to form the corresponding Diels-Alder adduct, which may yield the vitamin on hydrolysis. However, these attempts failed.

Conditions were optimized for the reduction of the free base from the cinchomerone by a metal hydride complex, and more than 90% yield of the vitamin was obtained. The process is being patented. Cost estimates of the process are being worked out.

17. *N, N-Diethyl-meta-toluamide* : (ATT-18/65)

This is one of the most effective mosquito repellents known. Even when applied in very low concentrations the mosquito repellency of this compound remains for several hours. It is not being manufactured in India at present.

A laboratory process has been worked out and will be shortly submitted to the Process Release Committee for approval for release.

18. *Colchicine* : (ATT-68/68)

Colchicine is used in pharmaceuticals and in plant breeding. The current



demand is approximately 2kg. per year, valued at Rs. 70,000/- and is expected to be almost doubled by next year.

Colchicine was extracted from the tubers of *Gloriosa superba* (Hindi-Kariheri; Langute) grown around Poona, Maharashtra and also from the *Colchicum luteum* corms (Hindi-Hirantutiya; Surinjan) grown in Jammu area. The yield of colchicine from both these sources is not satisfactory. A systematic study of other possible sources has led to the discovery of a very potent source for this chemical.

19. *Terpin hydrate and terpineol* : ( ATT-69/68 )

The total imports of terpineol and its esters amounted to 200 tonnes valued at Rs. 10 lakhs in 1968-69. Terpineol is used extensively in the perfumery industry. Terpin hydrate is used as an expectorant in cough mixtures.

The preparation of terpin hydrate from  $\alpha$ -pinene was optimized and scaled upto 2 kg./batch.

The effects of various catalysts and other reaction conditions were studied for the preparation of terpineol from terpin hydrate. A process for its preparation on 1 kg./batch scale was standardized. The laboratory process is being offered to industry.

20. *Camphene to camphor* : ( ATT-148/69 )

Camphor is used extensively as a plasticizer in the celluloid and other plastics apart from its uses as an incense etc. It is produced synthetically from  $\alpha$ -pinene (a constituent of turpentine oil) *via* camphene. The present work is undertaken to simplify the currently used three step route to camphor from camphene.

A single step direct oxidation of camphene to camphor was worked out on a laboratory scale (100 gm.). All raw materials were indigenously available and the method was quite simple. The overall efficiency of the process was 90%.

Since the above method was economically not viable, work on further optimization was undertaken and promising results have now been obtained. Attempts are now being made to try out the improvised process in batch sizes of one kg.

21. *p-Menthane hydroperoxide* : ( ATT-109/67 )

*p*-Menthane hydroperoxide is used as an initiator for vinyl polymerizations and also as a curing agent for thermoset polyesters and styrenated alkyds. At present, the country's requirements are met through imports.

The process developed in the laboratory for the preparation of *p*-menthane hydroperoxide has now been released to a firm. All the steps involved were demonstrated to the representatives of the firm.

22. *Utilization of  $\beta$ -himachalene* : (AB-17/67)

$\beta$ -Himachalene is the chief component of the essential oil from the *Cedrus deodara Loud* (Hindi-Deodar), which is commercially available. This project is undertaken with a view to utilize  $\beta$ -himachalene by its conversion into industrially useful products.

With a view to see if the derivatives of  $\beta$ -himachalene could have hormonal activity, the transformation of  $\beta$ -himachalene to  $\beta$ -himachalene oxide was carried out and hormonal activity of the latter is being studied.

Separation of the oxygenated compounds from the oil of *Cedrus deodara* has been achieved. These compounds are being studied for minimizing insect hormonal activity.

23. *Celastrus paniculatus* : (AB-18/67)

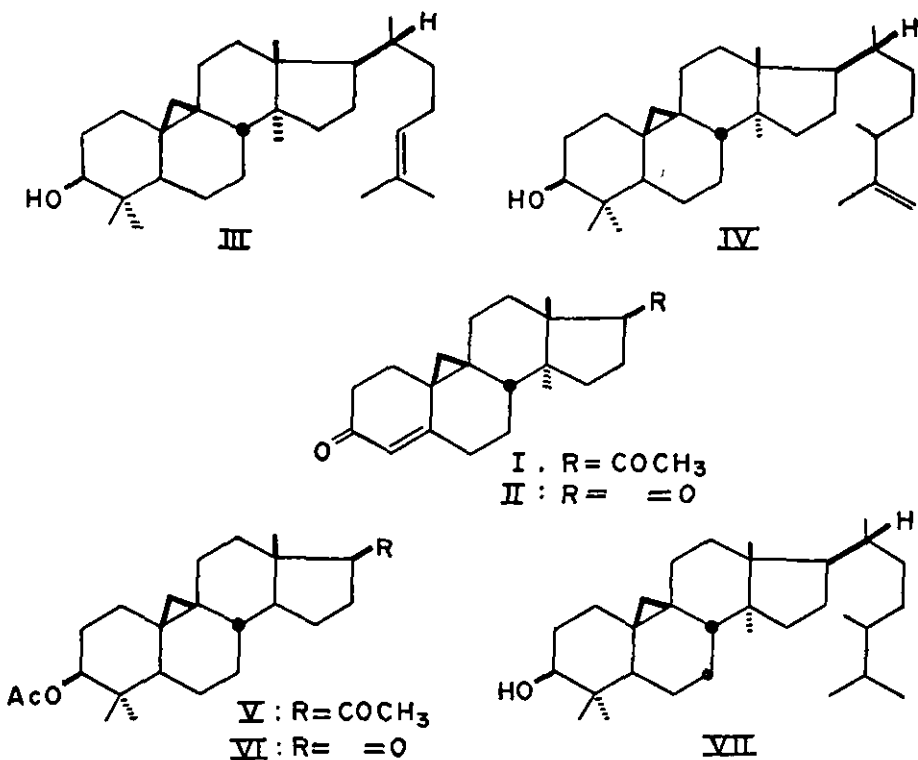
The sample of tetra-acetate of one of the parent polyalcohols obtained from the natural polyester extracted from *Celastrus paniculatus* (Hindi-Malkangini) is reported by the Reproductive Physiology Unit, G. S. Medical College, Bombay, to have valuable antifertility activity, similar to the parent polyester itself. A sample of natural polyester has been sent to Bristol Laboratories, Syracuse (N. Y.), U.S.A, for pharmacological activity. Work on the structure elucidation of the individual polyesters is being continued.

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

In order to evaluate the biological importance of 9,19 cyclosteroid hormones (I, II) cycloartenol (III) and cyclolaudenol (IV) seemed to be attractive starting materials. These triterpenoids (III and IV) are readily accessible from non-saponifiable fraction of opium marc, a bye-product of the Indian opium alkaloid industry.

A novel and efficient method was developed for the degradation of the side chain of (III) and (IV) to  $C_{17}$ -methyl ketone (V) and  $C_{17}$ -ketone (VI) in excellent yields.

For modification of ring A in (V) and (VI) to prepare the title compounds (I) and (II), the major obstacle is the removal of the geminal 4, 4' dimethyl groups. For model studies, modification of ring A in cyclolaudenol (VII) is under investigation. Since usual methods did not give good yields, a new method is being developed.



25. *Alkaloids from lanced poppy capsules* : (AB-48/68)

Lanced poppy capsules which still contain some residual alkaloids are not at present put to any significant use. It is estimated that nearly 4725 tonnes of lanced poppy capsules with a morphine content of about 0.2% are available for possible recovery of the alkaloid.

As a first step towards this end, a method was standardized for estimation of morphine in capsules. Utilizing this procedure, the morphine content of capsules, lanced as well as unlanced, obtained from different regions was estimated. In a preliminary laboratory experiment, some of the alkaloids have been actually isolated.

Further work is suspended pending the sponsorship of this project by the Narcotics Commissioner, Govt. of India, Gwalior.

26. *Hydroxyethyl starch* : (AB-60/68)

Hydroxyethyl starch (HES) which is widely used abroad in textile sizing and printing etc., is neither manufactured nor used in the country so far. It possesses many advantages over the conventional materials used at

present. Under this project, attempts are being made to develop specific varieties of HES suitable for textile industry.

Several samples of HES having different degree of substitution were prepared and sent to the Bombay University Department of Chemical Technology, (BUDCT) Bombay and Ahmedabad Textile Industry Research Association, (ATIRA) Ahmedabad for evaluation.

27. *Cashewnut shell gum* : (AB-76/69)

A water-soluble polysaccharide (CNS gum) was obtained from cashewnut shells to the extent of 10%. Experiments to obtain CNS gum from cashewnut shells on 1 kg. scale were successfully completed. A sample of CNS gum has been given to industry for exploring its possible industrial uses. The Oil and Natural Gas Commission (ONGC); the Institute of Petroleum Exploration, Dehra Dun; the National Metallurgical Laboratory, Jamshedpur, were also contacted for evaluation of the above gum as an aid in oil-well drilling mud and in ore floatation processes.

28. *Synthetic glycosides and other synthetic carbohydrate materials* :  
(ATT-19/66)

*Glycosides and thioglycosides* : During the period under review, eight different glycosides were sold to research institutes in U.S.A. and India.

1. Phenyl  $\beta$ -D-galactopyranoside
2. Phenyl  $\beta$ -D-glucopyranoside
3. *o*-nitrophenyl  $\beta$ -D-galactopyranoside
4. *p*-nitrophenyl  $\beta$ -D-galactopyranoside
5. *p*-nitrophenyl  $\beta$ -D-glucopyranoside
6. Phenyl  $\beta$ -D-thiogalactopyranoside
7. Phenyl  $\beta$ -D-thioglucopyranoside
8. *o*-nitrophenyl  $\beta$ -D-thiogalactopyranoside

*Phenolphthalein D-glucuronide* : Preparation of the title compound from phenolphthalein and methyl 1, 2, 3, 4-tetra-*o*-acetyl-D-glucopyranuronate using stannic chloride as a condensing agent did not meet with success. Hence the aceto-bromo-sugar was prepared and condensed with phenolphthalein in presence of silver carbonate using quinolene as a solvent. The glycoside was isolated as a cinchonidine salt in 5-8% yield.

Pending substantial orders for glycosides, work on this project has been discontinued.

29. *Cellulose powder* : (ATT-112/68)

Conditions to obtain cellulose powder (300 mesh) from cotton and ground nut shell (GNS) pulp in good yield were standardized. Several batches of 1 kg. have been prepared. The product was found suitable for chromatographic purpose. The project is concluded and the process is ready for release.

30. *Saccharification of cellulose* : ( AB-22/67)

It has been observed that the saccharification of cotton cellulose and GNS pulp could be carried out upto 80% with the help of gaseous hydrogen chloride and conc. hydrochloric acid; however, the residual hydrocellulose was obtained as a black powder and could not be utilized any further. Hence experiments have been undertaken to find out the possibility of using sulphuric acid of different concentrations and at different temperatures for saccharification of cellulose.

31. *N-Acetylneuraminic acid* : (ATT-113/68)

The synthesis of the key intermediate 1-nitro-2-acetamide-1,2-di-deoxy-D-mannitol has been standardized.

32. *Starch phosphate* : (ATT-54/64)

Cold water soluble starch phosphate is not manufactured at present in India. It is used as a thickener in food preparations, and as a core binder in foundry practice.

A sample of starch phosphate prepared from tapioca starch was tested by CFTRI, Mysore and reported to form a useful component of weaning food formulations with advantage. Complete analysis of the sample has shown absence of any harmful trace elements beyond the accepted limits. Attempts are being made to get this product cleared through Food Authorities so that further steps for releasing this process to industry could be taken.

33. *Foundry core binder* : (ATT-145/69)

NCL has been working for different types of foundry core binders. Development of sinol core binder, foundry core oil and a substitute for double boiled linseed oil was reported earlier.

After studying the evaluation reports on these products, it was thought to develop a cheaper product, which may possess all the desired properties of a good core binder and for which easily available cheap, indigenous raw materials are used.

Using modified tamarind kernel powder (TKP) as the main ingredient some compositions have been made and sent for testing to local foundries.

34. *Substitute for gum Arabic* : (ATT-107/70)

Based on the modification of indigenous gum Ghatti, some work for developing a substitute for gum Arabic was reported earlier. Due to non-availability and high prices of gum Ghatti and the low prices of imported gum Arabic, the work could not be implemented for commercial production.

A new approach is being made to the problem and a product based on modified starch, TKP and Guar gum is being prepared as a substitute for gum Arabic. The samples have shown comparable characteristics and they are being sent to industry for evaluation.

35. *Chemical stabilizer for desert and sea-beach sands* : (ATT-136/69)

The project was taken at the the instance of DRDO, Dighi, Poona, who wanted a substitute compound for the imported product now in use to stabilize desert sands to make helipads with necessary compressive strength.

Specially prepared starch phosphate samples were sent to the organization for testing and one of them was found to be satisfactory. Larger quantities of the material are now being supplied to Defence authorities for further tests and evaluation.

Another important and useful application of these chemicals may be in hardening sea-beach sand.

36. *Tamarind kernel powder* : (ATT-137/69)

Tamarind kernel powder (TKP) is produced in the country to the extent of 50,000 TPA and the production is likely to increase substantially in the future. At present the TKP is used in cotton and jute sizes and is also exported.

Work on phosphorylation and boration of TKP is undertaken with a view to prepare modified products from TKP which may be useful in the textile industry. One of the NCL samples tested at ATIRA, Ahmedabad, is reported to resemble hydrolysed corn starch with the added advantage that film extensibility is enhanced to six times that of unmodified TKP and 5 times that of corn starch. More samples are being made which will be tested at ATIRA, Ahmedabad and BTRA, Bombay. Samples are also sent abroad for testing and evaluation.

37. *Low temperature fluxes* : (ATT-85/68)

Good quality low temperature organic fluxes used for soldering electrical

contacts in electronic circuits are not manufactured in India. Technoeconomic data on these products are not readily available. These products are reported to be glutamic acid activated rosin compositions.

Attempts are under way to prepare similar compositions using cheaper and indigenously available materials. The samples are being sent to industry and further work will be done on the basis of the reports received from industry.

#### 38. *Stabilized gel for hot and cold sachets* : (ATT-128/69)

Various grades of commercially available PVC sheets were tried for their suitability for making sachets for the stabilized gel. Soft and flexible sachets, made from one of these materials by electronic heat-sealing, can be boiled in water and these do not become hard at the freezing temperature of the gel. The stabilized gel prepared from indigenous carbohydrate materials when sealed in these plastic sheet sachets, can be used both as a hot water bottle as well as an ice bag for convenient application of heat or cold to the human body and also for keeping food and drinks warm or cool as desired.

These gel-filled sachets may be expected to find wide domestic use and in hospitals.

Samples of these hot and cold sachets have been supplied to several parties. The process is available for commercial exploitation.

#### 39. *Dyes for synthetic fibres* : (ATT-23/66)

A new series of synthetic dyes has been developed under this project which has been covered by Indian Patent No. 116453. The invention relates to a new group of dyes which have not been marketed yet. As such before these dyes can be put on the market, considerable developmental work, feasibility studies and market research will have to be carried out. The process is being offered to industry.

During the year under report, exploratory work on the synthesis of new intermediates for disperse dyes is in progress. A few interesting diazo components containing benzyl sulfone groups were synthesized and will be used for preparation of disperse dyes.

#### 40. *Organophosphorus compounds*

Number of organophosphorus compounds find variety of uses such as plasticizers, catalysts, non-corrosive solvents, additives to petroleum and lubricants etc., in different industries. The project includes technical prepara-

tion of such compounds of potential industrial importance of which the uses could be identified and the products could be tested for specified requirements.

40-1. *Trioctyl phosphate* : (ATT-119/68)

This is a low temperature plasticizer of polyvinyl chloride type resins. It is also used for other plastics such as nitrocellulose, ethyl cellulose, polystyrene and chlorinated rubber.

Pilot plant trials on 12 kg. per batch were reported earlier. However, the samples prepared on laboratory and pilot plant scale were both found to contain higher amount of acids than permissible limits. The acidity in the product arises from the presence of entrained hydrochloric acid and/or from the hydrolysed ester. In spite of large number of trials it has not been found possible to prepare samples equivalent to imported commercial samples especially in regard to acidity specifications. This project has been suspended since technical inquiries indicated that the indigenous demand for this chemical is not very significant.

40-2. *Triphenyl phosphate* : (ATT-125/68)

This is an important plasticizer used in photographic films. The laboratory scale process developed earlier was scaled up to 5 kg. per batch and optimum laboratory conditions arrived earlier were confirmed. The product has been sent to M/s. Hindustan Photo Films, Ootacamund, for evaluation and for consumer acceptance. The work is now concluded.

40-3. *Tris-nonylphenyl phosphite* : (ATT-71/67)

This is a superior stabilizer for G. R. S. polymer and white rubber. The chemical is at present imported (70 TPA valued at Rs. 10.5 lakhs).

The development of large scale batch process (150 kg. per batch) with high yields was reported earlier. Samples from pilot plant batches of tris-nonylphenyl phosphite were sent to commercial firms for evaluation. Their report, however, indicated higher phosphorus and hydrolysable chlorine content in our sample than the imported samples. Accordingly the defect was rectified in further pilot plant trials and fresh samples have again been sent for evaluation. The results are awaited.

41. *Simazine and Atrazine* : (ATT-126/68)

Simazine is a herbicide useful in controlling herbs associated with wheat, maize and other crops. On completion of the bench scale work (0.5 kg./batch) reported earlier, a sample was sent to a commercial firm for dilution and standardization to produce a wettable powder for field trials. Field trials



have shown satisfactory results. The process was scaled up (3 kg./batch) and some more information was collected from the larger runs. The process is approved for release to the industry by the Process Release Committee. Negotiations with a commercial firm for exploitation of the process are in progress.

Atrazine is another product related to Simazine which is also used as a herbicide for maize and sugarcane crops. Work on this product was started on a bench scale and later scaled up to produce a product matching with the imported commercial sample. The process is also approved for release to industry by the Process Release Committee.

42. *Fraudulently altered bank cheques* : (ATT-146/70)

At the request of the Bank of India, Poona, the problem of fraudulent alteration of writing on bank cheques by means of various chemicals was investigated. Several remedies for detection and prevention of such fraud were recommended and a report sent to the bank authorities.

43. *'Sonobuoy' fluorescent dye marker* : (ATT-147/70)

At the request of the Defence, complete analysis of the various components of the Dye Marker package which is a part of an electronic gadget called 'Sonobuoy' was carried out. This component is at present being imported. On the basis of the analytical data attempts will be made to construct a prototype model for testing by the Defence authorities.

44. *Shark repellent compositions* : (ATT-148/70)

At the request of Defence, development of shark repellent compositions was undertaken. Such shark repellent units are part of standard survival equipment for shipwrecked sailors and are at present being imported. The analysis of the imported material was carried out, and with the help of a local dye manufacturing concern know-how for a suitable indigenous shark repellent composition is being developed. The results are encouraging and further work is in progress.

45. *Photosensitive chemicals* : (ATT-76/68)

The project has been undertaken with the object of standardizing technical preparations of the following two photosensitive chemicals: (i) 2, 3-dihydroxynaphthalene-6-sulphonic acid, and (ii) light sensitive stabilized diazonium salts. Both these chemicals are used for the preparation of photosensitive papers and are not manufactured in the country. Compound (i) is imported to the tune of 3 TPA valued at Rs. 1.5 lakhs; Compound (ii) is also imported to the same extent.

(i) *2,3-dihydroxynaphthalene-6-sulphonic acid*

A sample of the sodium salt of this acid was sent to industry for testing. The test report indicated that the process needed certain modifications to get a product of the requisite purity.

Laboratory conditions have now been standardized to get such a product and large scale trials will soon be undertaken.

(ii) *Light sensitive stabilized diazonium salts*

The sample of the zinc chloride complex of N-(*p*-hydroxyethyl) N-ethylaniline *p*-diazonium chloride which was sent to industry for trials has been approved.

Conditions were standardized for the preparation of the subject material in one kg. batch scale.

Further work on this project has been kept in abeyance until a party interested in the commercial exploitation of the process is located.

46. *Theophylline-caffeine* : (ATT-77/68)

Theophylline is used as a myocardial stimulant and diuretic. Aminophylline, a derivative of theophylline, is useful in bronchial asthma. Caffeine is widely used in pharmaceuticals.

Theophylline and its derivatives are not manufactured in the country, and their imports are estimated at 30 TPA valued at about Rs. 6.5 lakhs. Synthetic caffeine is also not produced in the country, but a few firms are extracting natural caffeine from tea-waste.

A convenient method has been developed for the synthesis of theophylline IP (1 kg./batch) starting from 1, 3-dimethylurea. The process is being offered to industry.

47. *p-tert-Butyl catechol* : (ATT-79/68)

This chemical is used as a polymerization inhibitor in the synthetic rubber industry. Present requirements are 50 TPA valued at Rs. 20 lakhs. With the growth of synthetic rubber industry in the country the demand is likely to grow further.

Satisfactory laboratory scale process for the preparation of *p-tert* butyl catechol was standardized. However, during scale up of pilot plant batches, some problems were encountered and these have now been solved. Pilot plant work is in progress and will be completed shortly. The process is being offered to industry.

48. *Catechol* : (ATT-82/68)

Catechol is a relatively expensive intermediate used in the manufacture of *p*-tert-butyl catechol. It is also used in the preparation of certain drugs like adrenaline. Catechol is neither manufactured in the country, nor are the estimates for its demand or imports available.

Although the project was initially aimed at the synthesis of catechol from *o*-dichlorobenzene or *o*-chlorophenol, it was decided to explore the possibility of isolating catechol from the polyvalent phenols which are available from the Neyveli Lignite Corporation, Neyveli.

From the catechol rich fraction (containing 65-70% catechol) obtained from the Neyveli Lignite Corporation, a process was developed to obtain pure catechol in 33% yield. Pilot plant work on 25 kg. catechol rich cut per batch was carried out.

49. *p*-Nitrophenol : (ATT-73/67)

*p*-Nitrophenol is mainly used for the production of pesticides. It is also used in the preparation of *p*-acetamol, an antipyretic drug. Estimated demand of *p*-nitrophenol for parathion manufacture alone is of the order of 300 TPA. Although it is manufactured by a few firms in the country, imports of *p*-nitrophenol during 1967-68 were of the order of 190 tonnes valued at Rs. 9.7 lakhs.

Laboratory scale work on 750 gm./batch was completed. Pilot plant work on 25 kg. scale is in progress. M/s. Hindustan Organic Chemicals, Rasayani, who are licensed for the manufacture of *p*-nitrophenol, are interested in the process and negotiations for release are in progress.

50. *Purification of crude anthracene* : (ATT-83/68)

Coal tar crude anthracene containing carbazole is available from steel plants in the country. Purified anthracene and carbazole are in demand by dyestuff and other industries. There appears to be some export potential also.

Crude mixture containing anthracene (18%) was enriched to a product containing 44% anthracene in 3 kg./batch. The product containing 44% anthracene was further enriched to 92% anthracene. The enrichment was carried out by solvents. Anthracene (92%) was supplied to the Explosives Research & Development Laboratory (ERDL), Poona.

Carbazole (96%) was also supplied to the ERDL, Poona. Several experiments were carried out to enrich anthracene (18%) by employing hot-

press method. Anthracene (18%) could be enriched to 40% under optimum conditions, recovery being 65%.

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

These pigments are not manufactured in the country. They give bright violet shades which possess excellent fastness properties comparable to the phthalocyanines.

The two main raw materials for this pigment are N-ethylcarbazole and chloranil. A process for the preparation of N-ethylcarbazole (yield 96% crude, 90% cryst.) was developed on 1 kg./batch scale and standardized by employing ethyl chloride and diethyl sulphate. Laboratory conditions for conversion of N-ethylcarbazole to pigment violet were standardized.

Further scale-up work is in progress.

52. *Vitamin D<sub>3</sub>* : (ATT-140/69)

Vitamin D<sub>3</sub> has anti-rachitic activity. During the period April 1968-March 1969, 1608 kg. of Vitamin D (D<sub>2</sub> and D<sub>3</sub>) valued at Rs. 6 lakhs were imported.

7-Dehydrocholesterol, a provitamin, was prepared in satisfactory yields starting from 7-ketocholesteryl acetate. Work is in progress to convert cholesterol to 7-ketocholesteryl acetate in high yields. Allylic bromination of cholesterylacetate and dehydrobromination of the resulting bromo compounds are also being investigated.

53. *Utilization of methyl benzoate* : (ATT-142/69)

Approximately 300 TPA of methyl benzoate will be produced as a by-product from the dimethyl terephthalate plant being commissioned at the Gujarat Aromatics Project of the Indian Petroleum Corporation, Baroda, (IPC). Work was undertaken to convert the ester into benzyl benzoate, benzoic acid, sodium benzoate etc.

*Sodium benzoate*

Direct hydrolysis of methyl benzoate, obtained from the IPC, did not yield sodium benzoate of BP quality due to the impurities present in the methyl benzoate and the yields were also not satisfactory. Further, due to difficulties during processing, such as concentration and filtration, the process of direct hydrolysis of methyl benzoate to get sodium benzoate was not found practical.

### *Benzyl benzoate*

Sodium benzoate obtained directly by hydrolysis of methyl benzoate was converted to benzyl benzoate (500 gm./ batch) in 81% yield. The experimental conditions for this process were standardized.

### *Benzoic acid*

Methyl benzoate obtained from the IPC was fractionated and a fraction (95%) was obtained as pure methyl benzoate. This fraction on hydrolysis followed by acidification gave pure benzoic acid (BP).

Pilot plant batches for the conversion of methyl benzoate to benzoic acid will be carried out when raw materials will be available.

### 54. *Semi-synthetic penicillins—ampicillin and cloxacillin* : (ATT-141/69)

These penicillins are gaining importance over the classical penicillins (such as G or V) because they overcome the main drawbacks of the classical penicillins.

A laboratory scale process for the preparation of *dl*-phenyl aminoacetic acid was standardized. Resolution of *dl*-phenyl aminoacetic acid with camphor sulfonic acid was carried out. The project was undertaken at the suggestion of the Hindustan Antibiotics Ltd., Pimpri (HAL). However, as the HAL did not sponsor the project, further work on this project has been stopped.

### 55. *Phenylacetic acid* : (ATT-149/70)

Phenylacetic acid is an important chemical used as a precursor in the manufacture of penicillin G. Its various esters are used in the perfumery industry.

At present, most of our country's requirements are met through imports. About 150 tonnes of phenylacetic acid worth Rs. 15.73 lakhs were imported during April - November 1969. The present the c.i.f. price is Rs. 11.20/kg. and the landed cost Rs. 18.00/kg.

A process was standardized on 1 kg. scale for the preparation of pure phenylacetic acid suitable for use for the manufacture of penicillin G. The starting material is benzyl chloride which is indigenously available. A few firms have shown interest in exploiting this know-how.

### 56. *Stationary liquid phases and solid support for GLC instruments* : (AB-33/63)

Preparation of a solid support from different qualities of fire-brick after special treatments was standardized, and the product could be successfully

used in GLC columns in place of imported Celite (UK) and Chromosorb (USA) supports.

#### *Stationary phases*

Standard procedures for the preparation of following polyesters were worked out :

1. Diethylene glycol succinic acid
2. Ethylene glycol succinic acid
3. Diethylene glycol adipic acid
4. Ethylene glycol adipic acid
5. Diethylene glycol sebacic acid
6. Ethylene glycol sebacic acid

Samples of some of these polyesters on fire-brick support were supplied to M/s. Associated Instruments Manufacturers (India) Pvt. Ltd., New Delhi, and found satisfactory.

Further work on standardization of procedures for the syntheses of novel polyesters and systematic studies of several other polyesters for aromatic compounds have been undertaken.

#### *57. 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 laboratory scale preparation of medium nitrile content rubber, standardized earlier to produce rubber in 70% yields and in the required mooney range, was scaled up to about 7 kg./batch of rubber. A distillation assembly for distilling 8 kg. butadiene/day was fabricated. Thirty batches of polymerization with 10 kg. monomer charge yielding 7 kg./batch of the polymer were carried out. The primary observation in the large scale runs was that the modifier efficiency was lower than the laboratory runs and differential addition of modifier did not improve the efficiency. Some samples obtained in large scale runs were evaluated by three different parties against imported nitrile rubbers. The processibility, physical properties and oil resistance were reported to be in the required ranges. Stabilization against storage deterioration needs improvement and recovery of the unreacted acrylonitrile needs further examination.

58. *Polystyrene foam* : (ATT-34/66)

Polystyrene foam, which finds exclusive use in low temperature insulation and packaging, is at present being manufactured with imported know-how. The demand in the country for polystyrene foam is estimated at 2000 TPA valued at about Rs. 2 crores.

Experiments to standardize the preparation of expandable polystyrene beads and obtaining low density foam sheets were continued. As earlier reported, the use of an indigenous suspending agent was effective in reducing agglomeration and obtaining glossy and transparent beads. A number of variables were studied to obtain required size beads in 70-80% yield. It was possible to obtain required size beads by modifying the technique of addition of the suspending agent. It was, however, observed that when proper size beads were obtained, the prefoaming characteristics were not very satisfactory and some portion of the beads had minor pinholes and tended to stick to each other. Efforts are being made to overcome these shortcomings by suitable changes in design of the equipment as well as in the process. Results so far obtained are encouraging.

A batch pre-expander fabricated for this purpose is working satisfactorily. Use of the continuous pre-expander designed and fabricated is being standardized. A newly fabricated large mould (1 m × 1 m ×  $\frac{1}{4}$  m) is being installed. As lower densities obtainable in small mouldings were not achieved in the big mould, a few modifications in the moulding technique are being made.

59. *Synthetic polymers for cane juice clarification* : (ATT-86/68)

Indian sugar mills have hitherto been using an imported product, namely, 'Separan AP-30' for clarification of cane juice. The demand for this product is estimated to be worth Rs. 10 lakhs per year.

Two sugar mills (based on laboratory experiments) reported that the synthetic polymer prepared at the NCL was comparable to the imported product. These mills later requested for larger quantities of the sample for factory trials.

The laboratory scale preparation of the polymer was first stepped up from 25 gm. to 250 gm. per batch. Conditions were standardized on 1.5 kg./batch scale. Two kilograms of the polymer were sent to each of three sugar mills for large scale evaluation. Three more sugar mills have recently asked for 2 kilogram samples and these are being supplied for trials during the current cane crushing season. Arrangements are under way to prepare the product on a larger scale for carrying out factory trials.

60. *Microfilters* : (ATT-87/68)

These filters are employed in a variety of filtration jobs in dyes, pharmaceuticals, food and other industries. These are very compact and easy to operate.

Using cotton linter pulp and melamine formaldehyde resin, a technique for preparing a uniform filter cartridge was developed. A few cartridges of 10" length were prepared and their use in filtration of crude saline water gave encouraging results.

The regulation of density and strength in the cartridge has to be standardized. An apparatus for porosity determination is being fabricated.

61. *Synthetic polymers for oil well drilling* : (ATT-115/68)

At the instance of the Oil and Natural Gas Commission (ONGC), New Delhi, work was undertaken for the technical preparation of a cement additive used in drilling oil wells. The product was found to be a water soluble polymer, which, when added in small quantities to cement, prevented quick water losses from cement slurry, decreased its viscosity and increased its setting time. At present, the import of this product is estimated to be worth Rs. 4 lakhs per year. Demand of this product is likely to rise in the near future.

Analysis of the imported product indicated it to be a mixture of a carbohydrate derivative and sulphur-containing chemicals. The carbohydrate portion was found to be a cellulose derivative containing a hydroxyethyl group to a degree of substitution (D. S.) of 0.8 to 0.9 and carboxymethyl group of D. S. of about 0.1. The sulphur portion appeared to be wholly comprising of sodium sulphate.

A sample of the polymer containing imported hydroxyethyl cellulose was found to be slightly better than the imported cement additive. This was sent to the ONGC for evaluation. The preparation of hydroxyethyl cellulose of the required D. S. is being undertaken.

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

Lining materials based on synthetic latex are chiefly meant for use in metal cans suitable for storing mineral oils, grease, food and the like. These are presently imported and their consumption in the country is around Rs. 10 lakhs per year.

Recipes based on nitrile rubber latex, vinyl resins, zinc oxide and other chemicals were prepared in 500 gm. lots. Preliminary tests indicated better properties of adhesion, storage stability and film formation compared to



compositions based on natural rubber latex. One kilogram samples of one of these formulations were sent to 4 parties for trial. In the light of the report received from one of them, certain improvements are being made and modified sample will be again sent for trials. Large quantity of the composition will be later prepared and its performance on automatic lining machine will be compared with imported product.

63. *Linseed oil emulsions* : (ATT-49/66)

The project is aimed at the development of a low cost, water thinnable emulsion paint in place of plastic based emulsion paints.

Evaluation reports, received from outside parties, suggested further grinding of pigments and reduction of drying time.

Improved drying properties were obtained when vacuum polymerized linseed oil was used for further polymerization in the presence of SO<sub>2</sub> and CO<sub>2</sub>. Larger batches will be prepared by this new technique and samples will be evaluated.

64. *Coating for nylon fabric* : (ATT-47/64)

Polyurethane coating compositions impart good water repellent properties to synthetic and natural fabrics employed for making raincoats, umbrellas and showerproof clothes. These are not at present manufactured in the country and an estimate of their annual requirement is not available.

Encouraged by the preliminary reports obtained from the Defence Laboratory, Kanpur, and the SASMIRA, Bombay, 400 metres of nylon fabric was coated with the polyurethane coating composition prepared in this Laboratory. This sample was also approved by the above laboratories. The process is being offered to industry.

65. *Polyisocyanates for coatings and adhesives* : (AB-77/69)

The isocyanate type adhesives are known to have superior bond strengths for various types of joints. It is estimated that the requirement of this type of adhesive in footwear industry alone is around 50 TPA valued at Rs. 20 lakhs.

Starting from anacardol (from CNSL), polyamines were prepared and used for making polyisocyanates on a laboratory scale. Large quantities of the product are being prepared for user trials.

66. *Rubber base adhesive* : (ATT-42/66)

Different types of rubber base special adhesives are required by the

Hindustan Aeronautics Ltd., (HAL), Bangalore. Their estimated requirement is nearly worth Rs. 50,000 per year.

Substitute samples for Bostik 1790, 1753 and 1752 made by the NCL were approved by the HAL, Bangalore, and a bulk order for the same has been received. The products are being prepared for supply to the party.

67. *Composite propellant* : (ATT-37/63)

As reported earlier, the Space Science Technology Centre (SSTC), Thumba, approved the NCL formulation of a composite propellant. On their request, about 50 kg. of the composition was supplied to the SSTC, Thumba, for their use. Since the SSTC, Thumba, would prepare the composite propellents needed by them, this project has been concluded.

68. *Materials for artificial limbs* : (ATT-88/67)

The Artificial Limbs Centre (ALC), Poona, uses a synthetic foam material for preparing artificial limbs. Large quantities of the material are required for this strategic need.

An imported sample received from the ALC was analysed and found to be based on rigid urethane foam. The main constituent of the polyol mixture was identified as a polyether of low viscosity. Small foam samples prepared from polyol and TDI made at the NCL were found to have properties comparable to the imported foam material used for making artificial limbs. The above polyol was, however, based on imported chemicals. A new polyol was, therefore, prepared from an indigenously available raw material. This polyol was then used to make rigid foam by reacting it with different isocyanates. For a proper evaluation of this material, large quantities of foam are required for which procurement of isocyanate is in progress.

69. *Polysulphide rubber* : (ATT-89/67)

Liquid thiokol rubbers (polysulphide) are required for the production of rocket propellents, oil and fuel resistant sealing materials, adhesives, etc. These are also needed by the Defence Services. Thiokol rubbers are not made in the country. The total requirement of these rubbers, at present, is estimated to be around 600 TPA valued at Rs. 90 lakhs.

Liquid polysulphide polymers having different SH numbers were prepared on laboratory scale and their vulcanizing properties studied. The rubber obtained was found comparable to an imported sample LP-2. Larger samples will be prepared for user trials.

70. *Sulphochlorinated polyolefin elastomers ( SCPE )* : (ATT-90/68)

The vulcanized SCPE-rubber has several outstanding properties such

as weather, heat and chemical resistance. Import figures are not available. Estimated demand is roughly assessed at 100 TPA. These rubbers are used for chemical resistant lining for storage tanks and similar plant equipment, acid hoses, hot conveyor belts and flexible steam pipes, protective coating for wood, masonry and fabrics, shoe soles and heels, cable jacketing, and for diaphragms, gasket seals and packings. If produced commercially in the country, demand may even exceed the above estimate.

Experimental conditions for obtaining a type of vulcanizable polyethylene, by making use of sulfuryl chloride as the sulphochlorinating agent, have been established on a small scale. Further work using gaseous chlorine is to commence soon, aimed at optimization of process variables, for preparation of general purpose synthetic rubber from polyethylene.

#### 71. *Sorbitol and mannitol from invert sugar* : (ATT-55/66)

Sorbitol is a versatile chemical, the bulk of which is consumed in the manufacture of Vitamin C and surfactants. It is also used in foods, pharmaceuticals and cosmetics. Sorbitol is presently produced in the country based on foreign know-how. However, the demand for this chemical is more than the production.

Mannitol, an isomer of sorbitol, finds uses in medicines, explosives, synthetic resins and fermentation industries. Estimates for the demand for mannitol are not available.

As part of the development of Vitamin C technology, a method for the preparation of sorbitol from glucose was developed. The economics of the process mainly depends on the glucose price. In view of the high price of glucose in the country, investigations were undertaken for the hydrogenation of invert sugar yielding sorbitol and mannitol.

It was reported earlier that conditions for inversion of sugar and subsequent hydrogenation of invert sugar to sorbitol and mannitol were established. A method was also reported for the separation of mannitol in good yield.

The process for the inversion of sugar was modified to reduce the quantity of acid required. This also reduced caramelization of the sugar and the load on the deionization equipment subsequently required.

The process for hydrogenation was also modified to increase catalyst life. The catalyst could be used twenty times instead of two to three times.

The process for the separation of mannitol was tried on polyol mixtures

obtained by hydrogenation on a 250 gm. polyols/batch scale. The yields were found to be a little lower than with pure polyol mixtures.

The process of inversion was tried out on 25 kg. /batch scale and that of hydrogenation on 4 kg./batch scale. The separation procedure is being modified to obtain a higher yield of mannitol.

72. *Chlorination of methane gas* : (ATT-92/67)

A process was developed for the chlorination of methane to produce chloroform and carbon tetrachloride. This project is being further undertaken jointly by the NCL and M/s. Standard Mills Co. Ltd., Bombay, at the premises of the latter at Thana. The experimental pilot plant (2.5 kg. /hr.) has already been installed and chlorination trials using the C-1 stream from naphtha cracker will soon be undertaken. The plant will be further scaled up after studying the results of the experimental pilot plant with the available methane gas.

73. *Plasticizers* : (ATT-135/69)

After concluding the major part of the pilot plant project on phthalate plasticizers, work was commenced on adipates and sebacates. These are also important plasticizers which are not at present manufactured in the country. Imports of plasticizers NES amounted to about 1000 TPA valued at Rs. 26 lakhs. Residual work yet to be done on different phthalates, and the proposed work on other types of plasticizers cited above, have been undertaken under this project.

The NCL process for the production of DOP, which was released to industry, meets the specifications of the firms to whom the process was released. Though the colour index of the product (30 APHA increasing to 60-80 APHA on heat treatment) was well within the specification, certain parties were interested in a product which did not deteriorate on heat treatment. Processes are known which produce DOP of such quality by using an amphoteric catalyst; but DOP so produced is relatively costly.

Development work was carried out to improve the existing NCL process in order to meet this requirement. A process was developed to give DOP of 30 APHA which does not change after heat treatment. The product also shows electrical properties of the same magnitude as obtained by using an amphoteric catalyst (an important quality sought by manufacturers of electrical cable industries).

One of the parties, to whom the DOP process was released, has commenced regular commercial production ( production 197.4 tonnes worth Rs. 14.6 lakhs in 1969-70 ). Erection of the DOP plant by the other party is in progress.

Preliminary work to develop a continuous process for DOP is under way and work on different plasticizers mentioned above is also being planned.

74. *Ethylenediamine* : (ATT-58/68)

Ethylenediamine is an important starting material for the manufacture of many industrial organic chemicals and is used in the manufacture of various organic fungicides, pesticides etc. The chemical is not produced at present in the country and the demand is expected to be around 1200 TPA valued at Rs. 90 lakhs.

As reported earlier, while collecting engineering design data on a continuous pilot plant for the production of 5 kg./hr. of ethylenediamine, several experimental difficulties were experienced. These were eliminated and the pilot plant was operated for several hours at a stretch and useful data were collected. The separation scheme was examined and a flow-sheet for producing pure ethylenediamine was prepared. The project will be completed shortly and will then be offered to industry.

75. *Self-balancing strip chart recorder* : (ATT-101/67)

Strip chart recorder, of the type developed at the NCL, is not manufactured in India and the entire requirement is met by imports. It is a versatile instrument which can be used for a variety of purposes and as an attachment to various instruments such as NMR, ESR, IR and UV spectrometers, polarographs, etc. It is estimated that there will be a demand for 1200 such units per year valued at Rs. 12,000 each. The development of a self-balancing strip chart recorder was completed and the process released to M/s. Associated Instruments Manufacturers (India) Pvt. Ltd., New Delhi, which is already producing VPC units based on the NCL know-how. A prototype unit was fabricated and sold to the party.

Specifications of the above recorder were reported in our Annual Report 1968-69.

76. *ESR spectrometer* : (ATT-64/68)

These spectrometers are used for analytical and research work. The instruments valued at Rs. 2-3 lakhs/unit are at present imported.

Following sub-assemblies and circuits were fabricated and tested: Solid state magnet power supply, Remote control for magnetic field, Klystron power supply, Modulators, Recorder amplifier, Microwave bridge.

The assembly of the various units of the ESR spectrometer has been completed and testing is in progress. A magnetometer has also been fabricated

which will be tested shortly. The design of the recording unit is in progress. In the mean time, the mechanical fabrication work connected with the recording system has been completed. The cabinets and consoles have also been constructed.

*77. Preparative VPC : (ATT-123/69)*

The preparative vapour phase chromatographs (VPC) are employed in research for separation and preparation of pure organic compounds. These are at present imported.

The preparative assembly consists of an automatic injection and collection system with pre-programming and is usually attached to a VPC. The assembly costs approximately Rs. 800/-. It is estimated that more than 200 such assemblies per year will be required for use along with VPC units.

The automatic injection system was fabricated and tested on the VPC set up. The performance was found to be satisfactory. A stainless steel automatic injection system is under fabrication. A trial model of the automatic collection system has also been made and is being tested. A leak-proof collection system is being designed. A new column injection system for rapid evaporation has been made and is under test.

*78. Ultrasonic interferometer : (ATT-124/69)*

The development of this instrument has been reported earlier. A technical data sheet was prepared for the instrument and circulated to different research laboratories, universities and firms to ascertain the demand. The response is very encouraging and reveals that there is a potential demand for this instrument in educational and other institutions in the country. Details of the design and the prototype are being worked out. Attempts will then be made to release the designs and know-how to industry.

*79. Recording polarograph : (ATT-134/69)*

Polarography is a widely used technique for inorganic and organic analyses. Polarographs having desirable sensitivity are not produced in India. An instrument with a sensitivity range of about 0.003 to 1 microampere/scale division of the recorder is expected to have a good market in various research institutions and universities. The price of imported units of this type ranges from Rs. 12,000-24,000 per unit. Scientific instrument manufacturers in the country have shown interest in this project.

The instrument consists of two basic units, recording assembly and bridge assembly. The design, fabrication and testing of the recording assembly

was successfully completed. Bridge assembly has been designed and the prototype is under test.

Work on the fabrication of mercury dropping electrode and glass electrodes is also in progress.

80. *Osmotic microbalance* : (B-14·14/69)

The construction of an electronic osmotic microbalance, using the new design based on gravimetric principle of measuring osmosis, has been undertaken. An electronic system with inductive-type transducer, capable of measuring linearly very small displacement in the range: 0·1 to 1·0 micron, has been fabricated for the manually operated osmotic balance, in the first instance. Transistor circuitry likely to be used for the final automation of the balance is also being developed.

□ □ □

## RESEARCH PROJECTS

### 1. STUDIES IN SOLID STATE

#### 1.1 *Ligand field theory* : (B-1.9/68)

The ligand field theory of trigonal bipyramidal vanadous complexes was worked out. The concerned energy level diagrams were prepared and employed successfully to interpret reported absorption spectra.

#### 1.2 *Studies on spinels* : (B-1.10/68)

Manganites of the general formula (1)  $\text{ZnCr}_{2x}\text{Mn}_{2-2x}\text{O}_4$  (2)  $\text{NiCr}_{2x}\text{Mn}_{2-2x}\text{O}_4$  (3)  $\text{CuCr}_{2x}\text{Mn}_{2-2x}\text{O}_4$  (4)  $\text{Zn}_x\text{Ni}_y\text{Mn}_{3-x-y}\text{O}_4$  and (5)  $\text{ZnFe}_{2x}\text{Mn}_{2-2x}\text{O}_4$  were prepared by solid state reactions. Electrical conductivity of all these compounds were studied as a function of temperature. Paramagnetic susceptibility was studied by the Gouy method in the temperature range  $90^\circ$  to  $600^\circ\text{K}$ . The compounds in the system  $\text{NiCr}_{2x}\text{Mn}_{2-2x}\text{O}_4$  and  $\text{Zn}_x\text{Ni}_y\text{Mn}_{3-x-y}\text{O}_4$  were ferrimagnetic at low temperatures. The results indicated that in these compounds the A-B interaction was of comparable magnitude to the negative B-B interaction which gave rise to a non-linear spin arrangement.

A study of the magnetic properties of some specifically chosen compounds containing paramagnetic ions only in the octahedral sites was also undertaken to establish the nature of B-B interactions.

It was found that  $d^5 - d^5$ ,  $d^4 - d^4$ ,  $d^2 - d^2$  interactions were all negative. However, in the case of  $d^3 - d^3$ , in isoelectronic pairs  $\text{Mn}^{+4} - \text{Mn}^{+4}$ ,  $\text{Mn}^{+4} - \text{Cr}^{+3}$  and  $\text{Cr}^{+3} - \text{Cr}^{+3}$ , only  $\text{Mn}^{+4} - \text{Mn}^{+4}$  gave ferromagnetic compounds suggesting a positive  $d^3 - d^3$  interactions, whereas the other two gave antiferromagnetic compounds suggesting a negative  $d^3 - d^3$  interaction. A series of compounds were synthesized where  $\text{Mn}^{+4} - \text{Mn}^{+4}$  pair was progressively replaced by  $\text{Cr}^{+3} - \text{Cr}^{+3}$ , in order to study the change in the sign of interaction.

X-ray powder patterns were taken for the compounds and they were all found to have cubic spinel structure.

The effect of replacing  $\text{Mn}^{+3}$  ion in  $\text{ZnMn}_2\text{O}_4$  by  $\text{Cr}^{+3}$  or  $\text{Fe}^{+3}$  on the temperature independent antiferromagnetism was also studied.



### 1.3 *Photoconducting materials* : (AB-5/62)

The electrical conductivity of chemically deposited layers was studied in the temperature region 80° to 400° K. The effect of doping with copper was also studied. The donor levels were found to be ionised in the region 80° to 300° K.

The spectral response of doped and undoped films was studied at 300° K and 80° K, which showed a blue shift compared to values for CdS single crystals. This could be due to (1) exciton formation, or (2) effect of surface states. The slow photocurrent relaxations showed the presence of  $\alpha$  and  $\beta$  traps.

A new cryostat for measuring thermally stimulated currents (TSC) is under fabrication. This will be used to obtain TSC measurements for a number of photoconductive materials.

A number of samples of chemically deposited layers of CdS were prepared and studies are in progress for utilizing them in devices.

### 1.4 *Dielectric properties and molecular structure of organic compounds* : (B-14-15/69)

The dielectric constant and loss factor of long chain alcohols, alkoxy ethanols and propanols showed sharp increase below the melting points. This anomalous behaviour was attributed to the transition to an unstable rotating phase. The molecular rotation in the solid state in the  $\alpha$  - phase was also confirmed by the I. R. spectra.

### 1.5 *Diffusion in solid state* : (B-2-1/67)

In continuation of the previous work on the diffusion of the rare-earth elements in copper, studies were undertaken on the isotope effect in the diffusion of  $^{57}\text{Co}$  and  $^{60}\text{Co}$  in copper. The method of determining the relative concentration of these two isotopes in their mixture was standardized so that simultaneous diffusion of  $^{57}\text{Co}$  and  $^{60}\text{Co}$  in copper could be followed. This procedure would eliminate any differences in the errors involved in the determination of relative depths of diffusion of  $^{57}\text{Co}$  and  $^{60}\text{Co}$  if diffusion experiments were carried out on different samples.

This method of analysis was further checked by two further experiments where the concentration of one of the isotopes in mixtures was kept constant and that of the other increased in the ratio 1, 2, 3, 4 and 5. The plots of the counting rate in Channel II (the twin 1.16 MeV + 1.33 MeV photo peaks of  $^{60}\text{Co}$ ) and Channel I (123 KeV photo peak of  $^{57}\text{Co}$ ) corrected for  $^{60}\text{Co}$

background, versus the relative concentration of  $^{57}\text{Co}$  and  $^{60}\text{Co}$  gave straight lines, one parallel (corresponding to the fixed concentration) to the relative concentration axis and the other with an expected slope.

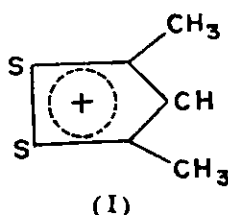
The analyses of the diffusion profiles of  $^{57}\text{Co}$  and  $^{60}\text{Co}$  in copper are in progress.

## 2. NUCLEAR AND RADIATION CHEMISTRY

### 2.1 *Studies on Mössbauer effect* : (B-3.1/67)

(a) The Mössbauer spectra in the temperature range  $-80^\circ$  to  $362^\circ$  of sources of  $^{57}\text{Co}$  in the form of oxidic spinels  $\text{Co}[\text{Cr}_2]\text{O}_4$  and  $\text{Co}[\text{Cr}_2]\text{S}_4$  matched against  $\text{K}_4[\text{Fe}(\text{CN})_6] \cdot 3\text{H}_2\text{O}$  absorbers were found to be complex. The spectrum of  $^{57}\text{Co}[\text{Cr}_2]\text{O}_4$  was analysed to consist of three separate Mössbauer spectra of ionic  $\text{Fe}^{+2}$ ,  $\text{Fe}^{+3}$  and  $\text{Fe}^{+4}$  states. The analysis of the spectrum of  $^{57}\text{Co}[\text{Cr}_2]\text{S}_4$  indicated the presence of ionic  $\text{Fe}^{+2}$  and  $\text{Fe}^{+3}$  states only. The stabilization of higher oxidation states viz.,  $\text{Fe}^{+4}$  and  $\text{Fe}^{+3}$ , produced due to Auger Effect in the electron capture decay of  $^{57}\text{Co}$  in addition to the expected  $\text{Fe}^{+2}$  state, may be attributed to the presence of cation vacancies in the source lattices of  $^{57}\text{Co}[\text{Cr}_2]\text{O}_4$ ,  $^{57}\text{Co}[\text{Cr}_2]\text{S}_4$ . Similar results were obtained from the Mössbauer spectrum of a source of  $\text{K}^{57}\text{CoF}_3$ .

(b) The crystals of *bis*-dithio-acetylacetonate tetrachloro ferrate (II) complex possess intense violet colour. It was, therefore, suggested by Knauer et al., [*Angew Chemie*, 6, 262 (1967)], that this complex contained a Fe-SS coordination since both (I)



and  $(\text{Fe}^{\text{II}}\text{Cl}_4)^{2-}$  ions were known to be colourless. The Mössbauer spectrum at  $78^\circ\text{K}$  of *bis*-dithio-acetylacetonate tetrachloroferrate (II) was shown to be similar to that of  $[(\text{CH}_3)_4\text{N}]_2[\text{FeCl}_4]$  indicating that  $\text{Fe}(\text{II})$  is tetrahedrally coordinated to 4  $\text{Cl}^-$  ions. The Mössbauer spectrum of  $[\text{C}_6\text{H}_7\text{S}_2]_2[\text{FeCl}_4]$  thus indicated that the Fe-SS coordination was absent in this complex. The intense violet colour of the complex could, therefore, arise only due to charge transfer absorption bands.

### 2.2 *Radiolysis of aromatic hydrocarbons* : (B-3.3/67)

#### 2.2.1 *Thermal hydroxylation of aromatic compounds using alcohol* :

A mixture of vapours of an aromatic compound, alcohol and oxygen at 300° and above yields a phenolic product whose rate of formation passes through a maximum as the concentration of alcohol is increased. Chemical kinetic analysis showed that the position of the maximum could be used to calculate the relative rates of reaction of a hydroxyl radical with various aromatics. The method was used for homolytic hydroxylation studies on benzene, naphthalene and bromobenzene and gave results consistent with existing theory.

*2.2.2 Temperature dependence of  $\gamma$ -radiolysis of the aqueous benzene system:* The hydroxyl radical attacks benzene in this system to yield phenol and  $\beta$ -hydroxymucondialdehyde. G values at room temperature are found to be 1.8 and 0.7 respectively. At 0° these values are retained, but the  $\beta$ -compound is replaced by a different product which has four carbonyl groups, a trihydroxy mucondialdehyde. From 0° upto  $\sim 50^\circ$  the G values of the two compounds remained constant. Thereafter, they rose significantly most probably because of thermal decomposition of the hydroperoxy radical, HO<sub>2</sub>. At 130°,  $G_{phenol} = 2.3$  and  $G_\beta = 1.5$ . Beyond this temperature the  $\beta$  compound was not stable.  $G_{phenol}$  continued to rise until, at 220°, it is  $\sim 5$ .

*2.2.3 Phenol formation in the  $\gamma$ -radiolysis of benzene vapour in presence of oxygen :* At 220° there was a chain reaction leading to phenol. This is presumably the chain reaction reported by M. A. Proskurnin et al. [Proc. First All-Union Conf. on Radiation Chem., Moscow, 11, 84 (1957)]. The yield, however, was not linear in dose, indicating a product effect. The highest G value obtained at 220° was 12. At 300° the product phenol was more active than benzene towards the intermediate species that the  $G_{phenol}$  dropped to a very low value. Some poly-hydroxy benzenes appeared to be the products of the decomposition of phenol.

### 3. THERMODYNAMIC STUDIES

#### 3.1 *Thermodynamic properties of solutions :* (B-13-2/63)

Linear polyelectrolytes possess both electrolytic as well as chain molecular properties and show peculiar characteristics in aqueous solution. Since ultrasonics is a powerful means for studying the properties of solutions and the nature of coulombic attraction, adiabatic compressibility measurements for polyelectrolyte solutions are expected to give interesting results.

It was observed that in polymethacrylic acid and in its partially or fully neutralized sodium salt solutions, the size of the molecules increased greatly upon dilution, but this chain elongation made no significant changes in the compressibility data. However, the compressibility was found to be influenced

appreciably by solvation of free counter ions and their concentration in dilute solution.

Further proof of this point was found in the study of a quaternized compound of polyvinyl pyridine i.e., Poly 4-vinyl N-n butyl pyridinium bromide. The apparent molal volume,  $\phi V_2$  and apparent molal compressibility,  $\phi K_2$  of this quaternized compound in aqueous solution was constant throughout the entire concentration range (0.1 — 2.0 gm./dl), as the free counter ions ( $\text{Br}^-$  ions) formed on dissociation were not solvated and made no significant contribution to the compressibility results. In presence of excess of KBr (1.0 M) solution, the  $\phi V_2$  and  $\phi K_2$  values showed a sharp decrease with increase of polyelectrolyte concentration and finally attained a constant value. This has been explained by the fact that due to the formation of a charge transfer complex with the bromide ions and the polycation molecule, more than one equivalent amount of bromide ions were bound in the molecule, leaving free an equal amount of  $\text{K}^+$  ions which eventually were solvated and caused the lowering of these values. It was concluded that the characteristics of the charged polymeric chain are responsible for the peculiar behaviour observed in viscosity and adiabatic compressibility data. The former was due to change of chain expansion, whereas the latter was due to variation of counter ions and their effective solvation in solution.

Further work on compressibility measurements of poly (acrylic acid) and poly (acrylamide) is being carried out.

### 3.2 *Thermodynamics of metal ion complex formation in mixed dioxane-water solutions* : (B-3-4/67)

The values of thermodynamic ionization constants of acetylacetone, benzoylacetone and dibenzoylmethane in 75 volume percent dioxane showed that the substitution of methyl group in acetylacetone brings about a significant increase in the value of the ionization constant, free energy and enthalpy of ionization. There was a small decrease in the entropy of ionization in the above series, but the increased negative contribution of entropy to the free energy of ionization was more than compensated by the changes in the enthalpy of ionization, leading to a net increase in free energy of ionization in the above series. These results showed that the presence of phenyl group in substituted acetylacetones has an electron donating effect which is possible only if the phenyl ring is coplanar with the hydrogen bonded enolic ring of the  $\beta$ -diketone. The presence of a strongly electropositive  $-\text{OCH}_3$  group in *p*-methoxy benzoylacetone further brings about an increase in the electron donor property of phenyl ring as shown by its increased free energy and heat of ionization as compared to that of the benzoylacetone.

It was found that in dioxane-water medium, the ionization of the  $\beta$ -diketone takes place with a higher free energy change and lower enthalpy change as compared to that in water and this could be attributed to the greater work done in the creation of ions in dioxane-water medium owing to its lower dielectric constant.

The experiments on the determination of the thermodynamic parameters for the formation of *bis*- $\beta$ -diketonato complexes of  $\text{Cu}^{+2}$  and  $\text{Ni}^{+2}$  were also completed in 75 volume percent dioxane solution. The results are being analysed on the CDC-3600-160A Computer. The preliminary results showed that for both  $\text{Cu}^{+2}$  and  $\text{Ni}^{+2}$  complexes the free energy as well as the enthalpy of formation increase as the methyl groups in acetylacetone are replaced by the phenyl group.

#### 4. CRYSTAL AND MOLECULAR STUDIES

##### 4.1 Crystallography : (B-2-5/59)

During this period, the precise structures of *p*-toluic acid and *p*-nitrotoluene were determined with the help of three dimensional data. These structure analyses were undertaken in order to study the influence of methyl group substitution in para position in monosubstituted benzenes containing electron withdrawing groups.

The crystals of *p*-toluic acid are found triclinic, space group with  $\bar{P}1$   $a = 8.88 \text{ \AA}$ ,  $b = 7.87 \text{ \AA}$ ,  $c = 7.59 \text{ \AA}$ ;  $\alpha = 121.4^\circ$ ,  $\beta = 118.7^\circ$ ,  $\gamma = 93.9^\circ$ ;  $\rho_o, 1.152 \text{ gm. cm}^{-3}$  and  $\rho_c$  for  $Z = 2, 1.256 \text{ gm. cm}^{-3}$ .

The central bonds in the benzene ring in *p*-toluic acid appeared to be a little shorter than the normal length ( $1.393 \text{ \AA}$ ). The carboxylic group had a pronounced torsional oscillation with an r.m.s. amplitude of about  $13^\circ$ ; the methyl group had a considerable intramolecular vibration normal to the C-C bond, the principle motion begin normal to the aromatic plane.

The crystals of *p*-nitrotoluene are found orthorhombic, space group  $Pcab$  with

$$a = 6.43 \text{ \AA}, b = 14.07 \text{ \AA}, c = 15.66 \text{ \AA};$$

$\rho_o, 1.294 \text{ gm. cm}^{-3}$  and  $\rho_c$  for  $Z = 8, 1.287 \text{ gm. cm}^{-3}$ . The molecular dimensions are found normal. The nitro group had pronounced torsional oscillation; the methyl group was wagging mainly normal to the aromatic plane.

The structure analyses of 2-nitro-4-methylbenzoic acid, 2-nitro-5-methylbenzoic acid and *o*-monomethylaminobenzoic acid are in progress.

## 5. THIN FILMS

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

A detailed study was made on the epitaxial growth, phase transition, crystal growth process etc., on vapour phase deposits of selenides and tellurides of copper, chromium, cobalt, germanium etc., on single crystal as well as polycrystalline materials by electron diffraction methods. It was generally observed that higher substrate temperature had a great influence in effecting epitaxial orientations on single crystal substrates. Despite the considerable difference in the structures of deposits as well as the substrates, the deposited films grew epitaxially on all single crystals at appropriate temperatures which eventually led to polycrystalline deposits with a preferred orientation. Both cuprous selenide and cuprous telluride showed a phase transition to hexagonal ones during the deposition process. Cupric selenide, however, decomposed to cuprous selenide during the deposition. Thin films of GeTe and GeSe formed below  $150^\circ$  were amorphous in nature whereas those formed above  $200^\circ$  were crystalline. GeTe at lower temperature had rhombohedral structure whereas at higher substrate temperature it developed both f.c.c. and h.c.p. structures, higher temperature, however, favouring epitaxial growth. In the case of GeSe, no change of structure was observed even at higher temperature. CoSe films at higher temperature not only grew epitaxially but also transformed two new phases, cubic ( $a_o = 5.78 \text{ \AA}$ ) and h.c.p. ( $a_o = 4.04 \text{ \AA}$   $c_o = 6.66 \text{ \AA}$ ) and varied from initial NiAs type of structure. The higher substrate temperature, again, favoured the formation of new phases. The vacuum deposited films of CrSe and CrTe developed new cubic and superstructure above  $300^\circ$  and  $400^\circ$  respectively along with their normal NiAs type of structure. The new cubic phase was often accompanied by (111) twinning.

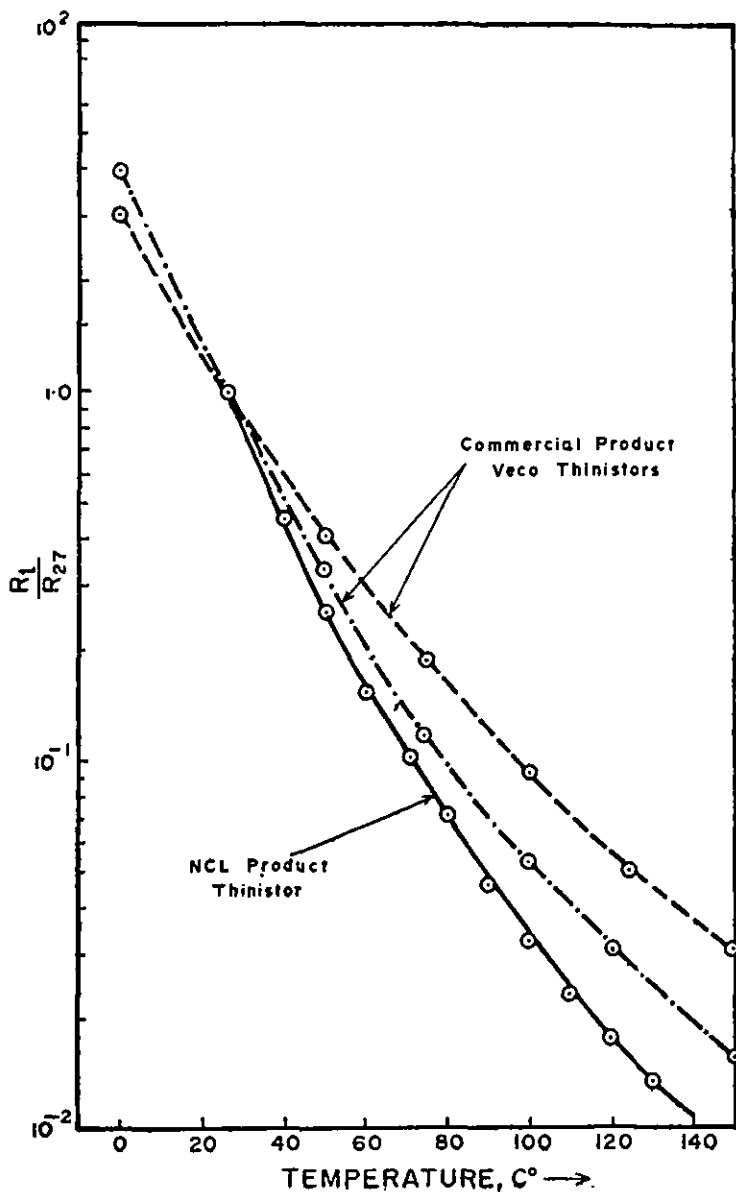
A detailed study was also made on the epitaxial growth, phase changes of alloys like In-Bi, In-Sn, In-Ga, In-Pb, In-Cd, Cd-Sb, Sb-Sn, etc. The orientation relationship, crystal growth process, phase transition etc., are now being evaluated.

### 5.2 *Physics of thin films* : (B-4.4/58)

Studies have been undertaken for the vacuum deposits of silver, aluminium, tellurium, bismuth etc., for their transmissions. It was found that the transmittance curves also pass through a minimum, then rising to a maximum again decreasing with increase of film thickness as observed in the case of selenium as reported earlier. Similar results were obtained for different wave lengths of light. Other films are also being studied for similar effects.

Preliminary investigations on thin film thermistors (thinistors) to be used for sensors for temperature measurements especially at upper atmosphere

during the flight of balloons or rockets have been undertaken. It was observed that films when prepared under suitable conditions of vacuum developed properties such as a high negative temperature coefficient. Typical films produced had a resistance ratio  $\left[ \frac{R_{\text{room}}}{R_{200^\circ}} \right]$  about 400, which is similar to imported products (see graph).



These films were, however, found to be slightly sensitive to long exposure to temperature and, hence, attempts are now being made to stabilize the temperature coefficient. Thin film resistors with low temperature coefficients and high stability are being used in numerous electronic circuits. Investigations have been undertaken to develop such thin film resistors with definite resistance/square. Several such resistors were made and tested. The TCR of these was found within the range of 50 to 100 p.p.m.

On the basis of the previous fundamental work done on Hall voltage, it has now been possible to find appropriate conditions of deposition so that a high Hall constant of the order of  $350 \text{ cm}^3/\text{C}$  could be obtained for some chalcogenide films. With such large value, it will now be possible to use these films as detectors for measuring low magnetic field. Work is in progress to make a suitable device for such measurements.

## 6. SYNTHETIC INORGANIC CHEMISTRY

### 6.1 *Titanium and tin organics* : (AB-14/63)

Organic oxo-titanium compounds (titanyl organics) of the type  $(\text{TiO})(\text{OR})_2$  are briefly mentioned in the literature as products of controlled hydrolysis of alkyl titanates or of oxidation of titanium (III) trialkoxides. It was observed that esterification of salicylaldehyde trichlorotitanium with the lower aliphatic alcohols (ethyl, propyl, butyl) gave rise to stable oxo-titanium organics of the type  $\text{L}(\text{TiO})(\text{OR})$  instead of the expected trialkoxy esters, LH being a molecule of salicylaldehyde. Products of the same composition were obtained by reacting salicylaldehyde with titanium (IV) tetra-alkoxides in benzene. The lone alkoxy group in these oxo-titanium compounds is found to be reactive and is easily replaceable by a large number of ligands operating through oxygen as well as through sulphur, the salicylaldehyde group remaining intact. These  $\text{L}_1\text{L}_2(\text{TiO})$  compounds are in general soluble and form dimers in boiling benzene.

Chelated titanium dipropoxides and cyclopentadiene titanium dichloride reacted in boiling toluene to give rise to new Ti-O-Ti organics, purified by recrystallization from methylene chloride. Using  $\beta$ -diketones as chelating reagents, identical compounds were obtained by reactions between (a) chelated titanium dichloride and cyclohexanol, (b) dicyclohexanoxy titanium dichloride and the  $\beta$ -diketone chelate, and (c) tetracyclohexanoxy titanium and the  $\beta$ -diketone.

New monomeric, organosoluble titano-stannoxanes have been isolated from reactions between chelated alkoxy-titaniums and aryl/alkyl tin (IV) hydroxides/oxides. Reactions were carried out usually in toluene in presence



of a fair excess of the tin compound and the crude product purified from benzene. Alcohol or ether was liberated in the course of the reaction. Some of these compounds may find practical application.

### 6.2 Coordination compounds : (B-6/63)

In continuation of our earlier work on quasiaromaticity of metal acetylacetonates, reactivities of beryllium chelates of a number of  $\beta$ -diketone containing ligands towards the electrophilic substitutions at the central carbon atom of the four membered ring were examined. Of the parent chelates used, *bis* (2,5-dimethoxy-4-chloro-acetoacetanilidato) beryllium (II), *bis* (*o*-chloroacetoacetanilidato) beryllium (II) and *bis* ( $\omega$ -bromoacetoacetanilidato) beryllium (II), as well as new substituted chelates like *bis* (2-bromo-1, 3-diphenyl propanediono) beryllium (II) *bis* (2-bromo-1-phenyl-1, 3-butanediano) beryllium (II), *bis* (3-bromo-2, 5-dimethoxy-4-chloroacetoacetanilidato) beryllium (II), *bis* (3-bromoorthoacetoacetanilidato) beryllium (II), *bis* (2-nitro-1, 3-diphenyl propanediono) beryllium (II), *bis* (2-nitro-1-phenyl-1, 3-butanediono) beryllium (II), *bis* (3-nitroorthoacetoacetanilidato) beryllium (II) were isolated for the first time. These new chelates are under study with respect to their IR, NMR and mass spectral data.

Investigations concerning the effect of coordination on the reactivity of aromatic ligands are of much current interest. Bromination, nitration, thiocyanation and mercuration carried on *bis* (salicylaldehydato) copper (II), -nickel (II) and -cobalt (II) showed that the pattern of aromatic reactivity exhibited by the chelated ligand differs somewhat from that exhibited by the free ligand. Careful acetylation of *bis* ( $\beta$ -resorcyldoximato) copper (II) and beryllium (II) gave *bis* (oximinoacetyl  $\beta$ -resorcyldoximato) copper (II) and beryllium (II). The NMR spectra of the parent and the acetylated chelates of beryllium (II) in DMSO confirmed the reactive nature of the oxime hydroxyl in comparison with the ring hydroxyl group.

Interest in the rare earth chelates has been stimulated by the fluorescence and laser properties exhibited by many of these and their possible use in the field of heterogeneous catalysis. Acetoacetanilidatochelates,  $ML_3 \cdot 2H_2O$  where  $M =$  neodymium, praseodymium, samarium and yttrium, were isolated in pure state. Thermogravimetric analysis (TGA) and Differential thermal analysis (DTA) studies for these hydrated chelates showed loss of one water molecule at  $120^\circ-60^\circ$ . IR spectra showed the coordination of the metal ions through the oxygen atom of the ligand. The chelated carbonyl frequency, however, showed no variation in contrast to the carbonyl frequencies of the acetoacetanilide chelates of other metals.

Mixed titanium complexes containing cyclopentadienyl group or

groups and another organic ligand were studied and part of the work is already published. *Bis- $\pi$ -cyclopentadienyl-bis* (thiophenolato) titanium (IV) a magenta coloured solid (m.p. 199°) and *bis- $\pi$ -cyclopentadienyl* (toluene-3, 4-dithiolato) titanium (IV), a deep green solid (m.p. 158-60°) were obtained by reacting the *bis- $\pi$ -cyclopentadienyl-titanium dichloride* with thiophenol and toluene-3, 4-dithiol in benzene or chloroform in presence of gaseous ammonia. Also by reacting the dichloride with *o*-aminothiophenol in chloroform with gaseous ammonia as base, a magenta coloured solid containing three molecules of the ligand per titanium atom was isolated. From its molecular weight, infrared data and TGA, this compound has been characterized as  $(\pi\text{-C}_5\text{H}_5)_2\text{Ti}(\text{HNC}_6\text{H}_4\text{S}) \cdot 2\text{H}_2\text{NC}_6\text{H}_4\text{SH}$ . Unstable compounds were obtained from the reactions of the dichloride with 2-mercaptopropionic acid, cysteine and 2-mercaptopyridine in chloroform.

### 6.3 Fluorine chemistry : (B-6.4/69)

Polarographic studies on cupferron and *m*-trifluoromethyl cupferron revealed that the aqueous acidic solutions of cupferron and its trifluoromethyl derivative could be preserved for many hours without any significant decomposition, provided air or any dissolved oxygen is completely excluded. Stability of *m*-trifluoromethyl cupferron was found to be quite comparable to that of cupferron.

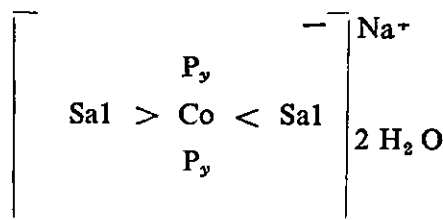
Reaction of fluoral hydrate with acetylacetone is being studied. The course of the reaction appears to be different from that involving chloral and acetylacetone.

## 7. PHYSICO ANALYTICAL CHEMISTRY

### 7.1 New analytical reactions : (AB-16/66)

7.1.1 *Sulphosalicylic acid in analytical chemistry*: Unlike  $\text{Mn}^{+2}$ ,  $\text{Co}^{+2}$  in sulphosalicylic acid does not readily undergo air-oxidation. But on treating such a solution with  $\text{H}_2\text{O}_2$ , the colour changed to green and the polarogram indicated trivalent cobalt. However, complete conversion to  $\text{Co}^{+3}$  was not effected, though addition of pyridine stabilized the trivalent state. The salt isolated from a solution containing cobalt (II) sulphate, salicylic acid, pyridine and hydrogen peroxide at an alkaline pH was greenish but not very crystalline. From elemental analysis, magnetic, IR, thermogravimetric and polarographic data, the salt is confirmed to be a mixed complex salt of trivalent cobalt which may be represented as ( Fig. on page 55 ).

The polarograms of copper (II) in sulphosalicylate indicated the presence of 1 : 1 and (at higher ligand concentration) 1 : 2 complex species. The 1 : 1 complex gave a well defined reversible two-electron cathodic step from which



the formation constant was computed as  $\log K = 9.6$ . The copper 1 : 2 complex as well as the  $\text{Co}^{3+}$  sulphosalicylate complex showed single minima in their polarograms. In other words, the current dropped at more negative potentials and rose again only at very high negative potentials. From the position of the minima in the potential spectrum and the attenuation of the minima at higher ionic strengths and/or in the presence of bulky positively charged ions such as cesium and quaternary ammonium, it is concluded that the fall in the current is primarily due to the electrostatic repulsion between the negatively charged electrode and the negatively charged depolariser in the electrical double layer.

**7.1.2 Redox behaviour of coordinated ligands:** Coordinated ligand reactivity in terms of nucleophilic reactions can be studied in an elegant manner through polarography, since electron addition is the simplest of nucleophilic reactions. It was indicated in the last year's report that ligands broadly fall into two categories, some whose  $E_{1/2}$  on coordination to a metal is displaced towards more positive potentials while others whose  $E_{1/2}$  on coordination is displaced towards more negative potentials. The  $\Delta E_{1/2}$  also appeared to be related in a qualitative way to the strength of the metal ligand bond as measured by their formation constants.

Interestingly enough, the product of electro-reduction as inferred by the number of electrons exchanged is also often different. In other words reduction is arrested at an intermediate stage. The following experiment will illustrate the point. While dimethyl glyoxime was reduced at the d.m.e. all the way to the corresponding amine, its cobalt (II) chelate was reduced to the corresponding hydroxylamine. The 1-nitroso-2-naphthols which, readily underwent reduction to the appropriate amine, when coordinated to  $\text{Fe}^{2+}$  got reduced to the corresponding hydroxylamine. The product of reduction of rhodizonic acid was normally the hexahydroxy benzene, but when it is chelated to  $\text{Co}^{2+}$  or  $\text{Ni}^{2+}$  reduction could be arrested at the tetrahydroxy benzoquinone stage. It was also confirmed by the controlled potential electrolysis of  $\text{Co}^{2+}$  chelate of dimethyl glyoxime at a mercury pool cathode, that the product of reduction is the corresponding hydroxylamine and not the amine. In favourable

situations, this electro-chemical approach of preparing oxidation reduction intermediates is likely to succeed where conventional methods fail.

The following analytical procedures were standardized in connection with the developmental work in progress in the Laboratory:

- (a) A new colorimetric method for aniline in acetanilide, based on diazotization of aniline followed by coupling with R-salt, was worked out.
- (b) A method was standardized for the estimation of nitrobenzene in aniline through polarography.
- (c) The potentiometric method for aniline based on titration with perchloric acid in glacial acetic acid, and the titrimetric method using nitrous acid were also standardized.

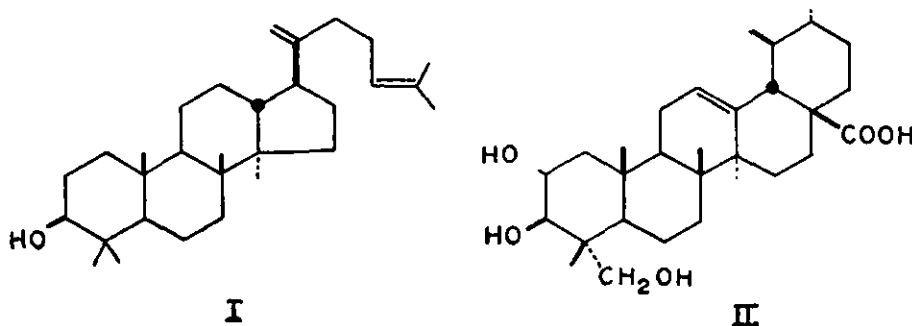
## 8. NATURAL ORGANIC PRODUCTS

### 8.1 *Oleoresins* : (AB-73/69)

8.1.1 *Veteria indica* Linn. (Hindi-Sufed dammar) —This is the commercially important white dammer resin of Kerala. The resin was first separated into neutral and acidic portions ( $\sim 75\%$  and  $\sim 25\%$  respectively on the weight of the resin taken).

The neutral portion was further fractionated by solvent extractions. The various fractions, thus obtained, were subjected to extensive chromatographic separations, whereby the neutral portion was found to contain large amounts ( $\sim 45\%$  on the resin taken) of higher ( $C_{60}$ ?) polyprenoids, small amounts of sesquiterpenes ( $\sim 0.5\%$ ), and triterpenes like dammardienol (I) and a related ketone.

The acidic portion of the resin was found to contain asiatic acid (II) and two other acids, whose structural investigation is under progress.



The possibility of utilizing these compounds commercially is being looked into.

8.1.2 *Gardenia lucida* : (Hindi, Marathi-Dikemali) — A systematic separation of the various constituents of the oleoresin from *Gardenia lucida* is in progress. The resin appears to contain  $C_{24}$ ,  $C_{26}$  and  $C_{28}$  saturated fatty alcohols, several flavonoids, some acids and small amounts of sesquiterpenes. Identification of the acidic portion is in progress.

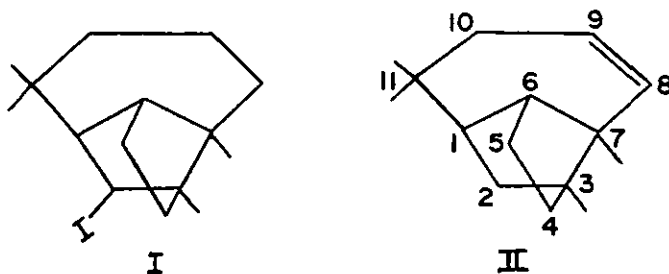
8.2. *Functionalization of saturated aliphatic and alicyclic compounds* :  
(B-8-46/69)

The functionalization of saturated hydrocarbons by photolysis is an attractive method to obtain commercially important products. For example, ( $\epsilon$ -caprolactum which is used in the production of Nylon-6 is being manufactured by photochemical nitrosation and subsequent Beckmann rearrangement. Similarly saturated hydrocarbons could be hydroxylated with peracids under UV irradiation.

Using *p*-menthane as the model compound some preliminary experiments were carried out by reacting it with peracid under UV irradiation, (*p*-menthane is easily obtainable by the hydrogenation of dipentene, a cheap raw material available from Indian turpentine oil). The material obtained seems to be mostly a mixture of isomeric *t*-alcohols. The separation and the characterization of these compounds is in progress.

8.3 *Photochemistry of organic compounds* : (B-8-47/69)

Photochemistry of longibornyl iodide (I) appeared attractive from a mechanistic view-point. Solution photolysis of (I) furnished longifolene (40%), longicyclene (44%) and a novel transannular hydrocarbon, longiborn-8-ene (II, 15%).



8.4 *Compounds of vegetable origin for insect control* : (AB-74/69)

A number of Indian plants are reputed as insect repellents and insecticides. It is proposed to investigate some of these plants to discover any new

structural types which may serve as a basis for developing new types of synthetic pesticides or insect repellents.

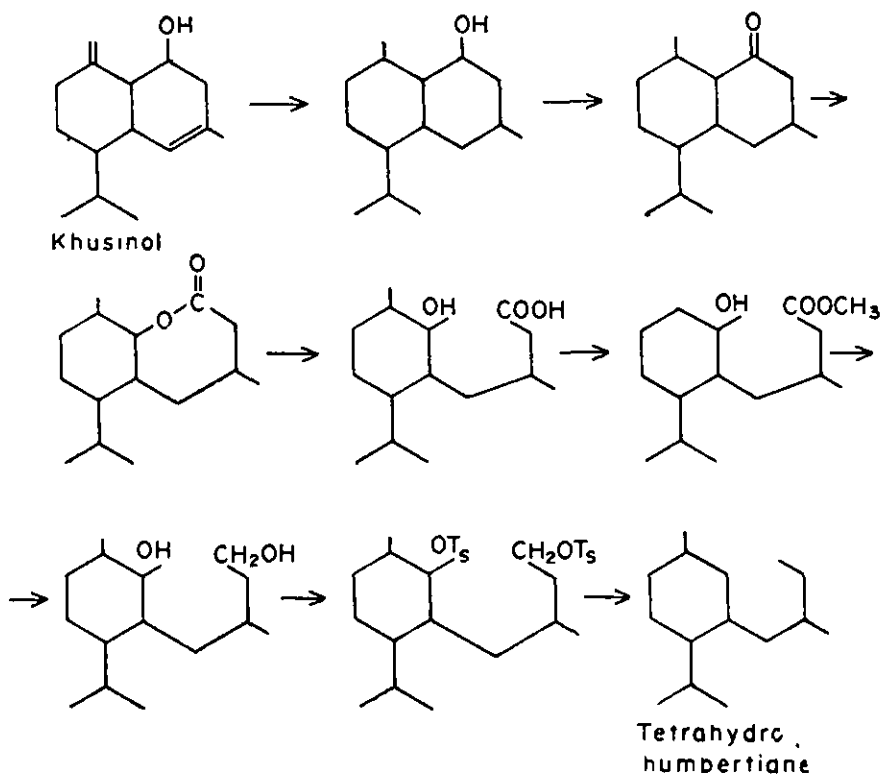
In the first instance *Spillanthus oleraceae* (Hindi-Akalkara) is chosen for this purpose. Various extracts were prepared and suitably emulsified for testing their insecticidal activity. The results are awaited. From the neutral portion of the petroleum ether extract, four crystalline compounds have been isolated by chromatography. Work on the characterization of these compounds as well as on their possible insecticidal properties is in progress.

#### 8.5 *Showwia arabica* : (AB-63/68)

The petroleum extract of the plant of *Showwia arabica* (stems and leaves) was found to consist essentially of fatty acids in which hexacosanoic acid (C<sub>26</sub>) predominates.

#### 8.6 *Vetiveria zizanoides* : (B-7-4/60)

A synthesis of humbertiane (tetrahydro humbertiene) was carried out by the following route from khusinol, a secondary alcohol obtained from Bharatpur vetiver oil.

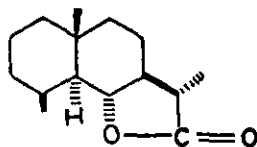


Further work on the transformation products of khusinol is in progress. Chemical examination of South Indian vetiver oil and Moosanagar vetiver oil is also in progress.

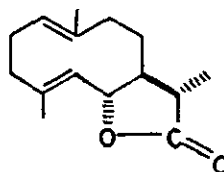
## 9. SYNTHETIC ORGANIC CHEMISTRY

### 9.1 Transformation products of costunolide : (B-8-15/66)

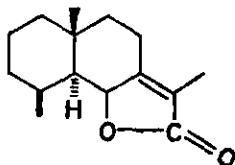
Starting from santanolide 'c' (I) obtainable from dihydrocostunolide, a method was developed for obtaining the conjugated  $\gamma$ -lactone (III) via the keto acid (IV).



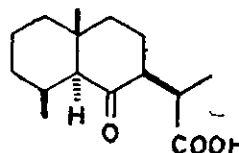
(I)



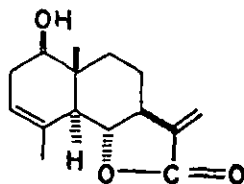
(II)



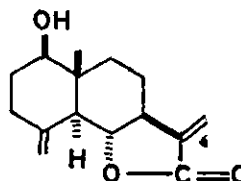
(III)



(IV)



(V)



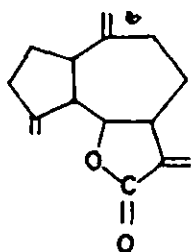
(VI)

The mass spectral fragmentation patterns of the two tetrahydrocostunolides and the ketolactones were studied with a view to correlate structures and fragmentation patterns in this series.

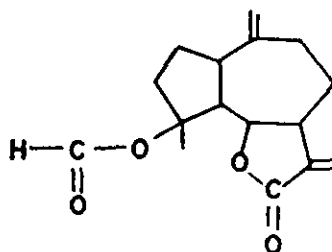
Costunolide was converted into santamarine (V) & Reynosin (VI).

#### 9.2 Chemical transformations of dehydrocostus lactone : (B-8-19/65)

The formate lactone obtained by the action of formic acid on dehydrocostus lactone (I) was shown to possess the structure (II) on the basis of chemical reactions and spectral data.



( I )



( II )

#### 9.3 Compounds related to selinane, elemene and p-menthane : (B-8-16/67)

Compounds related to selinane and elemene are widely distributed in nature and some of them as santonin are of medicinal value. Total syntheses of elemol,  $\beta$ -elemene, tauremisin and *Saussurea lactone* were carried out.

13-Noreudesm-4-en-3, 11-dione was synthesized starting from santonin. It is proposed to convert the diketone to carrissone and other interesting natural products.

#### 9.4 Sarkomycin : (B-8-26/68)

Sarkomycin causes destruction of tumour cells. With a view to synthesize sarkomycin and related compounds some substituted,  $\alpha$ ,  $\beta$ -epoxycyclohexanones were prepared through stereospecific reactions. The action of bases on these epoxyketones is under investigation.

#### 9.5 Flavonoids : (B-8-9/85)

The modified structure for gardenin A was confirmed by synthesis.

Eupatilin, isolated by Kupchan from a plant with cytotoxic properties was synthesized, and the synthesis of other related flavones is in progress.

#### 9.6 Naturally occurring anthraquinone pigments : (B-8-10/64)

Although the lac dye scheme sponsored by the Indian Lac Cess Com-



mittee, Ranchi, has been concluded, work is being continued on a few of the fundamental and applied aspects. The possibility of preparing a few useful pigments from crude lac dye is being investigated. In this connection, correspondence is in progress with M/s. Angelo Brothers, Calcutta, for developing a simple and inexpensive process for the isolation of lac dye from the water washings of stick lac. Attempts are being made to synthesize one of the derivatives of laccaic acid A or B with the object of obtaining further confirmation of the proposed structures. Work is also being continued on general methods for the arylation of anthraquinones.

#### 9·7 *Marschalk reaction* : (B-8·48/69)

The Marschalk reaction, which consists in the C-alkylation of hydroxy- and aminoanthraquinones by treatment with aqueous sodium dithionite, sodium hydroxide and an aldehyde, was used by us earlier for the synthesis of several naturally occurring anthraquinones. The reaction is also the subject of recent patents on disperse dyes. Using NMR spectra for following the progress of the reaction, the mechanism has now been elucidated, and a paper will shortly be communicated for publication. Carrying out the reaction with aromatic aldehyde including salicylaldehyde, the dyeing properties of the products and their derivatives are being examined.

#### 9·8 *Synthetic dyes*

9·8·1 *Mechanism of reactive dyeing* : (B-8·42/68)—The work reported in a preliminary note (*Ind. J. Chem.* 6, 397, 1968) is being extended. Preliminary work on the dyeing properties of Remazol Brilliant Blue R (CI Reactive Blue 19) and the corresponding vinyl sulfone has shown that the formation of the vinyl sulfone in the dye-bath is unfavourable for the dyeing process. Its formation on the fibre cannot be excluded at this stage, but an alternative mechanism of dyeing involving an SN2 reaction needs to be considered (paper under publication in *Text Res. J.*). However, much more experimental work using a variety of dyes of the Remazol type and a variety of dyeing conditions will have to be carried out.

Some of our work on reactive dyes, azoic coupling components, azophenol-quinone hydrazone tautomerism, vat dyes, and the possibilities of NMR spectroscopy in determining the structures of synthetic dyes, investigating steric effects, following reaction paths, and studying dye-fibre interactions were presented at a symposium in the Department of Chemical Technology, Bombay, in February 1969, the Proceedings of which have been published.

9·8·2 *Vat dyes* : (B-8·8/62)—Extensions of the work briefly reported derivatives earlier (*Chem. & Ind.* 1524, 1967) are in progress with the object of

preparing of polycyclic quinones possessing the requisite volatility for mass spectra and solubility for NMR spectra determination. Trimethylsilyl (TMS) ethers of anthrahydroquinone, xanthopurpurin, and the leuco derivative of a dibromoanthanthrone were prepared and their NMR spectra determined. Indanthrone gave the TMS ether of the leuco compound, but its NMR spectrum could not be determined because of its instability and inadequate solubility. The mass spectrum of the reductive methylation product of indanthrone has shown that it is the dimethyl ether of the tetrahydro derivative of the azine.

Treatment of 2-aminonthraquinone with potassium hydroxide in dimethyl sulphoxide under certain conditions yielded an isomer of indanthrone, as indicated by the mass and NMR spectra of the reductive methylation product.

9·8·3 *NMR spectra of anthrones* : (B-8·34/68)—The work reported earlier on the NMR spectra of anthrones was extended to carminic acid and laccaic acids A and B. The position of the C - glucosidic group in carminic acid and the aryl side-chain in laccaic acids A and B have been confirmed by determining the NMR spectra of the anthrones derived from the corresponding xanthopurpurin derivatives having an  $\alpha$ -hydroxyl group.

9·8·4 *Reduction of anthraquinones* : (B-8·35/68)—In connection with the structure of erythrolaccin, the migration of an  $\alpha$ -methyl group during zinc dust distillation was demonstrated earlier. Further work has shown that Clar reduction of l-hydroxy-4-methyl and l-chloro-4-methylanthraquinone yields a mixture of  $\alpha$ - and  $\beta$ -methylanthracenes; 1, 4-dimethylanthraquinone yields 1,3-dimethylanthracene. Reduction with zinc and aqueous sodium hydroxide gave 1, 4-dimethylanthracene, therefore involving no migration of  $\alpha$ -methyl groups.

9·8·5 *Reduction of quinones* : (B-8·20/68)—Three of the products which were isolated in a crystalline form from the exhaustive reduction of 1,1'-dianthrimide in alkaline dithionite were subjected to detailed spectroscopic (IR, UV and mass spectrum; NMR spectrum could not be determined for want of solubility in suitable solvents) and elementary analysis and tentative structures were assigned. The first step in the reduction is probably the formation of anthrone which eventually dimerises to bianthranyl type of compounds. The mechanism of formation of the reduction products is under study.

The study was extended to 1, 1'-dianthraquinonyl. Some of the side products formed during the Ullman condensation are also under investigation. The alkaline dithionite reduction of 1, 1'-dianthraquinonyl has indicated the formation of dibenzoperylenequinone.



CHART A

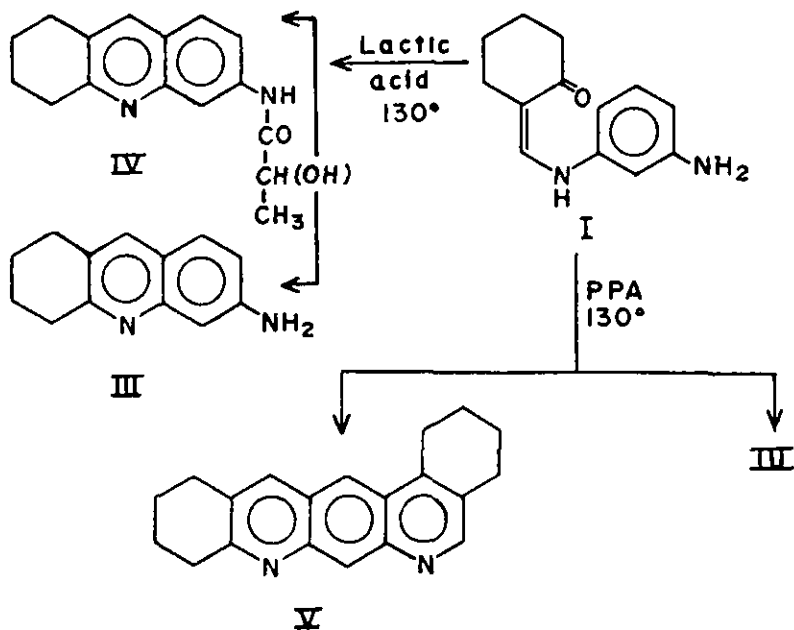
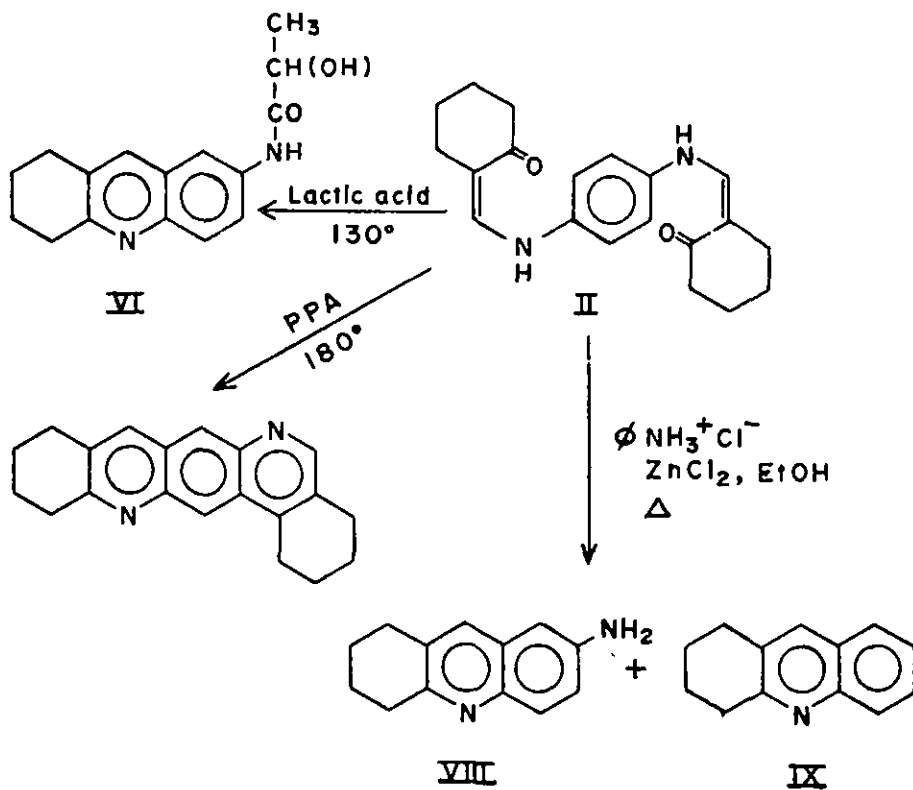
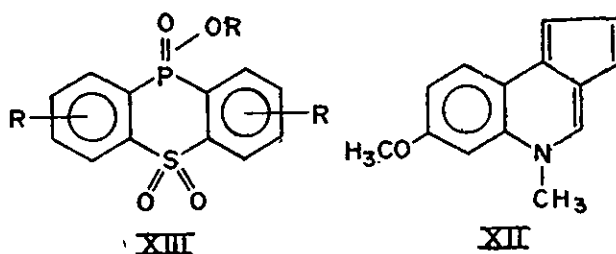


CHART B

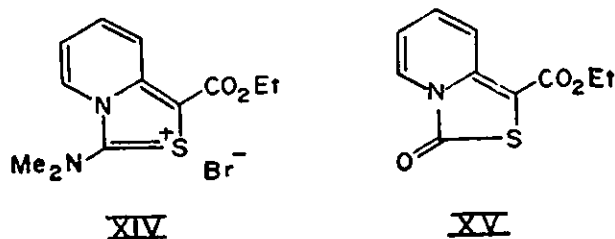


9·9·3 *Heterocyclic compounds containing two or more hetero-elements* : (B-8·52/69)—All attempts to aromatize 2, 3-dihydrofuro-(3,2-c)-quinolines proved futile. Similarly cyclodehydration of 2-arylaminomethylene-  $\gamma$ -butyrolactone involving rearrangement to yield linear furoquinolines could also not be effected.

A new class of phosphorus heterocyclics, phenothiaphospholanes (XIII) was synthesized. Biological activity of these interesting compounds will be studied.

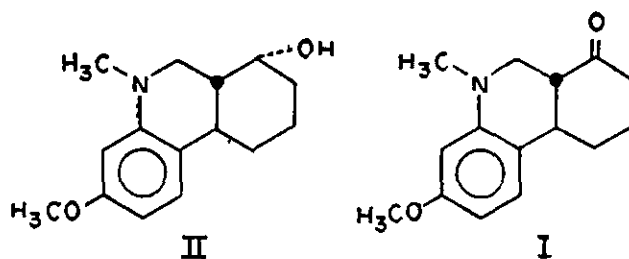


For preparation of 1-carbethoxy-3,4-phthaloyl-2-thiaquinolizine, it was necessary to synthesize ethylmercapto- $\alpha$ -pyridylacetate. Attempts to synthesize the latter by interaction of ethyl bromo- $\alpha$ -pyridylacetate with tetramethylthiourea, however, lead to 1-carbethoxy-3-dimethyl-aminothiazole-(3,4-a)-pyridinium bromide (XIV), which on boiling with ethanol yielded 1-carbethoxythiazolo-(3,4-a)-pyridine-3-one (XV). Both (XIV) and (XV) represent interesting new heterocyclic systems.



#### 9·10 *Antifertility drugs* : (AB-25/66)

For the synthesis of 11-aza steroids, the key intermediate 7-keto-3-methoxy-5-methyl-5,6,6a,7,8,9,10,10a-octahydrophenanthridine (I) was prepared earlier in low yield by the Oppenauer oxidation of the corresponding 7- $\alpha$ -hydroxy derivative (II). Experiments to improve the yield of (I) by microbiological oxidation of (II) are under way.



Synthesis of thiophene analogues of stilbestrol referred to in earlier reports failed at the final step.

## 10. PHYSICAL ORGANIC CHEMISTRY

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

#### 10.1.1 *Donor-acceptor interaction and hyperconjugation*

(a) *Proton chemical shifts in benzene derivatives* : In continuation of previous work, the approximate analyses of the NMR spectra of 4-substituted phthalic esters were refined using LAOCN-3. The matching between calculated and observed spectra was very good. The systematic variations in chemical shift reported earlier remain substantially unaltered by the refinement. The *t*-butyl group not only appears to be a weaker donor than the methyl group, but it also has a steric effect. Even the weak splitting of methoxyl signals could be used to obtain some useful information about electronic interaction in the system.

This type of analysis has been extended to *o*-dinitrobenzene and *o*-nitrobenzoic acid derivatives also. The results await interpretation.

(b) *Nucleophilic reactivity of o-dinitrobenzene derivatives* : The reactivities of these 4-substituted derivatives to sodium methoxide in methanol were measured at 30°, 35°, 40°, 45°, and 50° and Arrhenius parameters were evaluated. The *m*:*p* reactivity ratios for the methyl and *t*-butyl derivatives were determined at 50°. These ratios do not change very much with temperature. In order to estimate Arrhenius parameters for the *m* and *p* reactions separately for these derivatives, use was made of the approximate inverse relation that seems to obtain between log PZ values and the square roots of the activation energies. The analysis showed that the larger *para* reactivity of the *t*-butyl derivative as compared with the methyl derivative is in fact due to the lower activation energy for the latter as compared with the former. The conclusion is, again, that the methyl group is a stronger donor than the *t*-butyl group.

10.1.2 *α-Proton chemical shifts in cyclohexanones* : A 2,4,6-tri-substituted cyclohexanone was prepared and an attempt was made to prepare its stereo-isomers in pure condition by enolization and reketonization under selected conditions. The identification of the products obtained is being checked.

10.2 *Carbonium salts* : (B-5.3/64)

A series of internal salts from aromatic aldehydes and boron fluoride were prepared and their NMR spectra studied. They have been found to be similar to the corresponding acetoxy perchlorates previously studied.

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

It is proposed to take up for investigation biochemically interesting molecules where the primary chemical structures do not give any clue to their actual physiological functions. It is expected that study of secondary features is likely to furnish very useful information in this regard.

As part of this programme, a study of ascorbic acid derivatives has been initiated. Earlier work on the osazones of ascorbic acid suggested that such a study would be most valuable. Considerable progress has already been made.

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

A computer programme for all valence electron molecular orbital calculations in the CNDO and INDO approximations is being adapted for use with the CDC-3600 computer and this fairly refined method will soon be available for the study of not only aromatic but also saturated and partly unsaturated systems.

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

Esters of a number of steroid hydroxyl groups with the hydroxyls at adjacent positions were prepared and their infra red spectra determined in carbon disulphide, carbon tetrachloride, chloroform and dioxan solutions under different dilutions. Intermolecular hydrogen bonding between the carbonyl and the hydroxyl groups are observed in certain cases. The studies showed that the carbonyl group in these esters remain in such a conformation that these are nearest to the adjacent alkyl hydrogen atoms. This is with respect to the esters of both secondary and tertiary hydroxyl groups. The energy of stabilization of this conformation appears to be more than that of hydrogen bonding of the carbonyl and nearly hydroxyl group.

## 10·6 *Mass spectrometry* : (B-5·7/65)

10·6·1 *A structural probe* : A systematic study of the mass spectral fragmentation modes operating in some germacranolides of the costunolide type and some furanogermacranolides of the linderalactone type was made. An attempt was made to correlate structural features with the fragmentation reactions. The use of mass spectrometry as a structural tool in sesquiterpene lactone chemistry has been shown. Part of this work was published in the journal — *Organic Mass Spectrometry*. The structures of a new alkaloid and a new diterpene were established from physical methods.

10·6·2 *Molecular reorganisation processes induced by electron impact* : Many electron impact induced rearrangement processes were observed in the fragmentation of some bisazo compounds. Evidence in support of these reactions was obtained from the shift technique, high resolution data, metastable transitions and low ionization studies. The elimination of phenyl nitrene was observed in the mass spectra of some cyclosilazenes. This work has been accepted for publication in the *Journal of Organic Chemistry*.

10·6·3 *Characterization of organo-metallic compounds* : With a view to use mass spectrometry to characterize organophosphorus compounds a systematic study was completed on the behaviour of some  $\alpha$ -hydroxy phosphonates. A novel fragmentation mode resulting in the elimination of an oxygen atom from these hydroxy compounds was observed. The genesis of the major fragments was established.

10·6·4 *Stereochemistry and fragmentation* : Work on the behaviour of a number of *syn* and *anti*-7 substituted bicyclo-heptanes under electron impact was completed. This work was published in the journal — *Organic Mass Spectrometry*.

10·6·5 *Mass spectrometry of heterocyclic compounds* : In continuation of the work on cinnolines and quinoxalines mass spectra of cinnoline N-oxides were studied. A new type of rearrangement reaction resulting in remote group participation was observed. The structural requirements for this process to operate was established.

10·6·6 *Measurement of ionization and appearance potential* : Work is in progress on standardizing the technique involved in the accurate measurement of ionization and appearance potentials. This will lead to the work on the *Structures of intermediate ions*.

Efforts are being made to modify the instrument for the metastable defocussing and for field ionization work.



## 11. NATIONAL COLLECTION OF INDUSTRIAL MICROORGANISMS (AB-62/68)

Non-pathogenic microorganisms (yeast, bacteria and fungi) which are of importance in research and industry are maintained in this culture collection. Cultures are supplied free of charge on request to scientific institutions and industries. By supplying these cultures, which would otherwise have to be imported from abroad, a considerable saving in foreign exchange is effected. Identification of cultures obtained during screening programmes of microorganisms in the Laboratory is routinely undertaken. Research on the preservation of cultures especially under paraffin oil, which is relatively simple and requires no specialized equipment, is in progress as a long term project.

The routine maintenance and subculturing of the microorganisms were carried out and the cultures were tested for their biochemical performance. Cultures preserved under paraffin oil were tested twice during the year for their viability, morphology and biochemical performance.

Six hundred and eighty-three cultures were despatched to various industries and research organizations. Twenty-two cultures were added to the collection.

Phenol coefficients of six samples were determined and ten compounds from the Organic Division were tested for bactericidal and bacteriostatic and fungicidal and fungistatic action.

## 11. GROWTH OF PLANT AND ANIMAL CELLS (B-11/63)

The study of the growth and metabolism of different plant cells is of interest in connection with plant growth and the formation of useful plant products. The objectives of this long range project at present are: (i) to determine the requirements for rapid growth of different plant cells and for the growth of single cells (ii) to study the growth of monocotyledonous cells and other plant tissues which have hitherto been investigated only to a limited extent and (iii) to study the metabolism of normal and tumour cells of plants. These studies on different plant tissues are of potential agricultural importance.

The requirements for the continuous growth *in vitro* of cells from different plants such as opium, cholai, maize etc., were determined earlier. Twenty-five plant cell cultures are being maintained at present of which some were isolated and established for the first time in this laboratory.

Maize, wheat, rice and jowar cultures, which are viable *in vitro* and which are obtained for the first time in this laboratory, were studied with regard to their nutritional requirements, response to plant hormones and variation in enzyme content during differentiation. Auxin was the only hormone which was required in the medium for the growth of the cereals, kinetin, gibberellic acid and zeatin had no significant effect on the growth of these tissues though kinetin and gibberellic acid altered the relative amounts of callus tissue and roots in the case of maize and wheat. With rice root, formation was very slow in solid media or in shake flasks. The effect of several nitrogen and carbohydrate sources and different compositions of basal salts media, and the effect of varying the concentrations of auxins, sucrose and nitrogen source on the growth of these cultures were determined. The variation in twelve enzymes in maize and wheat during the transition from callus to roots was determined but no significant differences in the contents of any of the glycolytic or citric acid cycle enzymes were observed.

Most plant tissues showed very poor or no growth with very small inocula and very few clones were obtained from single cells. Considerable progress was made in obtaining high cloning efficiency of apple tissue. Further work on obtaining growth of single cells is being continued.

Preliminary work on setting up inoculum and media rooms and obtaining chemicals and equipment for work on animal cells and viruses was completed.

### 13. ENZYMES

The study of enzymes is closely related to the study of intermediary metabolism. The work on enzymes is directed towards the isolation of enzymes and the study of regulatory mechanisms which control metabolism.

#### 13.1 *Hexokinase* : (B-12.1/63)

Animal tissue hexokinases have not hitherto been obtained in pure form. A new method was developed for the solubilization of hexokinase from brain and heart. The enzyme from brain was obtained in a state of high purity and its properties and kinetics were studied. The insoluble hexokinase of heart was solubilized and obtained in highly purified form. Its  $K_m$  for ATP was 0.6mM at 1.7 mM  $Mg^{++}$ . The  $K_i$  for G-6-P was 0.19 mM at 10 mM Pi and 0.23mM at 25 mM phosphate. It showed a broad pH optimum between 4.5 to 8. The effect of several carbohydrates on the activity of the enzyme and their  $K_m$  values were determined and inhibition by ADP, fructose 1-6-diP, 5-thio D-glucose, PCMB and iodoacetate were studied.

### 13·2 *Phytase* : (B-12-2/64)

A bacterial enzyme which specifically hydrolyses inositol hexaphosphate was discovered. It is the only known phytase which specifically hydrolyses inositol phosphate.

The enzyme from *Bacillus subtilis* was obtained in highly purified form and was shown to be ultracentrifugally homogeneous. Its sedimentation constant was 3·5 S. It showed high stability to proteolytic enzymes, to alkali and to acid. SH compounds, PCMB, DFP and 6M urea had no effect on the enzyme. Further characterization of this unusual enzyme is in progress.

### 13·3 *DPNase* : (B-12-4/65)

The isonicotinic acid hydrazide-insensitive DPNases have been purified from animal tissues but the isonicotinic acid hydrazide sensitive enzyme from ox brain has not hitherto been obtained in purified form.

The enzyme from ox brain was obtained in soluble form by the action of lipase and obtained in highly purified form. The method of purification was considerably simplified and the yield of the purified enzyme was increased. Several compounds including glycolytic intermediates, members of the citric acid cycle and compounds involved in DPN synthesis etc. were tested for their effect but none of the compounds tested had any effect on the activity of the enzyme or its inhibition by nicotinamide. Studies on the detailed kinetics and properties of the purified enzyme are in progress.

### 13·4 *Acylphosphatase* : (B-12-7/68)

Acylphosphatases of animal tissues have been studied in detail but their occurrence in plants has not been investigated. Since acylphosphates do not occur in plant or animal tissues (except 1-3-dip glyceric acid and carbamyl phosphate), the function of this enzyme and its role in the regulation of metabolism are obscure.

The acylphosphatase of chulai beans was purified about 800-fold. The properties of the enzyme obtained from different batches of seeds were found to be variable and some preparations of the enzyme showed activation by  $Mg^{++}$  but this activity was unstable. The effect of hormones and other compounds on the activity of the enzyme and its physiological role are being investigated.

During the course of work on acylphosphatase a highly active trypsin inhibitor was isolated in good yield. A crystalline preparation was obtained initially which had low specific activity but chromatography on DEAE cellulose increased the specific activity by about 10-fold.

### 13·5 *Enzymes and the metabolism of organic acids* : (B-12·8/68)

13·5·1 Bacterial degradation of (—) citramalate: Three enzymes which activate succinate to succinyl CoA, transfer CoA from succinate to citramalate and cleave citramalyl CoA to pyruvate and acetyl CoA respectively were shown to be present in cell free extracts of a *Pseudomonas* strain grown on citraconate or (—) citramalate. (+) Citramalate was not metabolized *in vitro*. The citramalyl CoA cleavage enzyme was purified about 15-fold.

13·5·2 *D-Malate dehydrogenase*: This enzyme was purified about 50-fold from a *pseudomonas* strain grown on maleate. It was separated from L-malic dehydrogenase and L-malic enzyme and was shown to have an absolute requirement for  $K^+$  and  $Mg^{++}$ . It is specific for DPN and catalyzes the oxidative cleavage of D-malate to pyruvate and  $CO_2$ .

### 13·6 *Metabolism of nitrate by A. fischeri* : (B-12·6/65)

Microorganisms play an important role in maintaining the nitrogen cycle. The activity of denitrifying bacteria accounts for substantial losses of nitrate-containing fertilizers. Biochemical studies undertaken with isolated enzymes will help understand the basic mechanisms involved in the denitrifications.

Fifteen milligrams of ultracentrifugally homogeneous nitrite reductase was prepared with a view to determine its heme and iron content. The sedimentation pattern showed no indication of heterogeneity, and no dependence of sedimentation rate on concentration was observed. The molecular weight of the enzyme protein was determined by the Archibald method and from the diffusion and sedimentation coefficients. Heme and iron content of the protein were also determined.

The heme of nitrite reductase was not split by acid acetone but was split by treatment with silver sulfate. The positions of the peaks of its cyanide and pyridine derivatives and the general shape of the spectra were similar to those of hematohematin and its hemochromes prepared from horse heart cytochrome c. The spectra of pyridine and cyanide hemochromogens formed directly from the protein were similar to those from muscle cytochrome c. It is concluded that the prosthetic group of nitrite reductase is heme c.

Disc electrophoresis has revealed the presence of a fast moving band on treatment of the enzyme with protein denaturants.

### 13·7 *Citrate-oxaloacetatylase (Citrase)* : (B-12·5/65)

Studies on the structure and function of the enzyme were continued. The enzyme was shown to contain only sulphhydryl groups and no disulphide

linkages. The sulphhydryl groups are not involved at the active site but are required for maintaining the quaternary structure of the enzyme molecule. The effect of C-14 labelled oxaloacetate and of structurally related compounds on the irreversible inhibition of the enzyme was studied. The inhibition phenomenon was found to be stereoselective and to be due probably to a conformational change in the enzyme molecule.

The enzyme isolated from different microbial sources is found to be distinct immunologically.

### 13·8 *Screening of microorganisms for the preparation of industrially important enzymes* : (AB-67/69)

Amylase, protease, cellulase etc., are well known and industrially useful enzymes finding applications in many industries including the pharmaceutical industry. Most of these enzymes are imported as such or in the form of pharmaceutical compositions. The object of this long range project is to screen and isolate microorganisms for the preparation of some of the industrially important enzymes.

#### *Cellulase*

Isolation and screening of cellulose-decomposing fungi from various sources were carried out using different carbon sources such as filter paper, absorbent cotton, cellulose powder and bagasse. Of the 25 fungal isolates so far obtained, only 3 gave appreciable cellulolytic activities when tested by reducing sugar and viscosity methods. The strains isolated belong either to *Chaetomium*, *Trichoderma* or *Aspergillus* species. Studies to establish optimum conditions for obtaining maximum formation of cellulose are being carried out. Work will also be carried out to study the mode of action of cellulases and the number of components involved in cellulose breakdown.

#### *Amylase*

The group of black *Aspergilli* is well known to produce under suitable conditions extracellular amylases and proteases. Hence all the available strains of *Aspergillus niger* (130 cultures) in NCIM were taken up for a preliminary screening. These have been initially grown under one set of well defined conditions using indigenously available materials as carbon and nitrogen sources. After growth under submerged conditions for a fixed time interval, the fermented broths were tested for protease and amylase activities using standard methods of assay; in the case of amylase, two methods viz., dextrinization time and colour with alkaline dinitrosalicylic acid were employed for ascertaining extracellular activity. Among the cultures tested so far, about six to eight strains showed reasonable amylase activity and another ten cultures were also

worthy of further investigation. The rest showed such weak activity that they need not be taken up for further work. The former set of six to eight strains of *Aspergilli* will be taken up for intensive study in order to increase the amylase activity excreted into the medium .

Besides, a screening of the *Aspergillus oryzae* cultures available in the NCIM has also been started since some of them may be potential amylase producers.

### 13.9 DFP-Susceptible enzymes and antidotes : (AB-65/68)

Modern insecticides like parathion and malathion are phosphoric acid esters which *per se* are not toxic, but become so after metabolism in the liver and conversion to more poisonous analogues. Diisopropyl phosphorofluoridate (DFP) is the most well-known prototype of organo-phosphorous esters, which are also potential chemical warfare agents. The lethal effect of organophosphates is attributable to the inhibition of acetylcholinesterase, the vital enzyme concerned in nerve activity. However, a less studied field in the toxicology of organo-phosphates is the whole spectrum of sub-lethal conditions in which the enzyme levels are kept in a chronic state of partial inhibition. This is likely to happen in those who are engaged in the manufacture, distribution and use of organo-phosphorus insecticides.

The aim of this project is to study such pathological conditions at the biochemical level. When DFP is injected to mice at the LD<sub>50</sub> dose, the K/Na ratio of liver tissue was found to increase. The differences were statistically significant. Since, *in vitro*, these changes may be indirect, due to DFP action elsewhere in the intact animal, the experiments were conducted *in vitro* with liver slices, both in Krebs-Ringer solution and 0.25 M sucrose medium buffered with 0.01 M Tris at pH 7.4. In both cases there was a significant rise in the K/Na ratio of the tissue. The results indicate an action of DFP at the membrane level. Studies are being continued to determine the effect of drugs such as atropine and ouabain on the DFP-stimulated shift in the K/Na ratio as also to correlate these results with possible changes in the (K<sup>+</sup>—Na<sup>+</sup>)—dependent ATPase and esterase contents of the liver tissue.

## 14. PHYSICO CHEMICAL STUDIES IN POLYMERS

### 14.1 Stereospecific polymerization : (B-13.1/60)

Styrene-acrylonitrile copolymers containing 20-35% acrylonitrile have excellent physical properties for different applications. It has been reported that if copolymerization is carried out with Ziegler catalysts, unbreakable glass-like product is obtained.

Copolymerization of styrene-acrylonitrile with  $\text{VOCl}_3\text{-AlEt}_3$  catalyst system at  $40^\circ$  was carried out in n-hexane. It was found that irrespective of the monomer feed ratio, the copolymer contained the two monomers in equimolar proportion. Such alternating copolymers have improved properties over the conventional copolymers. To understand the mechanism of reaction and to obtain higher yields, the use of other Ziegler catalyst systems such as chromium acetylacetonate- $\text{AlEt}_3$  is being studied.

Studies in the polymerization of acrylonitrile-methyl methacrylate with  $\text{VCl}_4\text{-AlEt}_3$  catalyst system in presence of acetonitrile, were extended so as to confirm the mechanism of polymerization. Acrylonitrile was polymerized with increasing concentration of inhibitor hydroquinone when rate of polymerization and molecular weight decreased, thereby exhibiting a free radical mechanism. The formation of free radicals was further confirmed by the increase in rate of polymerization and molecular weight with increasing concentration of acetonitrile suggesting that  $\text{CH}_2\text{CN}$  type free radical initiate the reaction.

In the case of methyl methacrylate, it was observed that both free radical and coordinate anionic mechanism operate simultaneously. The polymer had heteroblock structure in absence of hydroquinone and on addition of hydroquinone, it changed to a stereoblock structure.

Earlier studies in the use of chromium acetylacetonate- $\text{AlEt}_3$  in the polymerization of styrene, were extended to the polymerization of methyl methacrylate and acrylonitrile at  $40^\circ$  in benzene. It was found that in the polymerization of methyl methacrylate at a high ratio (12) of  $\text{Al/Cr}$  there is a linear dependence of rate of polymerization on the catalyst and monomer concentrations, which suggests a coordinate anionic mechanism. However, at such high ratio of  $\text{Al/Cr}$ , anionic mechanism may also be possible for the polymerization of polar monomers, and to examine this, further experiments to study the polymerization in presence of electron donors are in progress.

#### 14.2 Oxetane polymers : (AB-69/69)

These polymers prepared by the polymerization of 3,3,- bis (chloromethyl) oxetane possess excellent corrosion resistance properties and are being used for coating reaction kettles etc., where protection under demanding temperature and abrasive conditions is required. Such type of polymers are at present being used in foreign countries.

Pentaerythritol, the starting material for preparing this monomer is already being prepared in small quantities in the country. With the growing chemical industry in the country, there exists a large potential demand for these polymers.

3, 3-bis (chloromethyl) oxetane was obtained by the cyclization of the corresponding tetracetate of pentaerythritol. After conducting several laboratory experiments, conditions for the preparation of trichloromonoacetate from pentaerythritol were standardized. A few preliminary experiments were also carried out for preparing the intermediate by a one step process where pentaerythritol, acetic acid and HCL gas were reacted together. One step process should be more economical and in the course of a few more experiments, it should be possible to increase the yield. No work on polymerization of monomer undertaken.

It was, however, decided to discontinue further work on this project till some party shows interest in the product.

#### 14.3 *Degradation of long chain molecules by ultrasonics* : (B-13.3/63)

The ultrasonic degradation of macromolecules has some resemblance to mechanical degradation i.e., mastication and the process is investigated on different industrially useful polymers such as sol rubber, butyl rubber, Neoprene AD, etc.

The study of ultrasonic degradation of Neoprene AD in toluene has shown that the behaviour of this rubber is rather different from that of butyl rubber, a structurally irregular polymer. Unlike butyl rubber, the degradation rates, as obtained by the two methods, solution viscosity and free radical estimation as a function of DPPH consumed, were more or less the same. The rate of degradation for Neoprene AD (trans rubber) was found 1.6 times more than that of natural rubber (cis rubber) which may be explained as the trans rubber is not so elastic as cis rubber and accordingly may break more easily if the distorting force is high enough. Further work on molecular characteristics and other aspects of this polymer is being carried out.

#### 14.4 *Polymer characterization* : (B-14.14/69)

A Monograph: *Thermodynamic properties of monomers and polymers* was compiled and contributed to the *Encyclopedia of Polymer Science and Technology*, Wiley, New York. The programme of calculating thermodynamic properties from molecular-structural and spectroscopic data is progressing. The first paper on such properties of 10 monomeric compounds is being published in the Journal of Polymer Science, A2 (Polymer physics).

### 15. CHEMICAL ENGINEERING STUDIES

#### 15.1 *Fluidization* : (B-14.5/68)

Arrangements for some modifications which were considered necessary in the 18" dia. fluidization column were made. With the help of the instrumen-



tation group, a simple pressure transducer was developed, tested and found to be a suitable accessory to record pressure drop across the 12 pairs of points in the bed. These are now being fabricated. Tapered inserts for the fluidization column are also being fabricated. Three separate ring balance manometers are being constructed for determining the air flow rate, pressure drop across the distributor, and pressure drop across the whole bed. An arrangement was developed for continuous multiple point indication of the quality of fluidization. It may be noted that although this scheme was mentioned in previous reports, concentrated attention could be given only during the last few months.

#### 15·2 *Mass transfer* : (B-14·6/68)

Investigations on flooding and hold up in a pulsed liquid extraction column were completed. After examining over a 100 models statistically, a final model was selected which represented all the data satisfactorily for flooding in a pulsed packed column. Similarly, a model was also developed for holdup in a pulsed packed column. Prior to the development of these models, equations were developed for predicting flooding and holdup in a conventional packed column. These equations formed the basis for the mathematical models developed for the pulsed column.

#### 15·3 *Direct liquid-liquid heat transfer* : (B-14·2/66)

Studies on the rotary disc contractor (R. D. C.) were completed. Several combinations of internal geometry were used. About 250 runs were carried out. The results of the packed bed column obtained previously and those of the R. D. C. are both being interpreted and development of suitable correlations is being tried.

#### 15·4 *Diffusion in solid catalysts* : (B-14·7/68)

A complete unit has been set up for measuring the rates of diffusion in solid pellets. This employs a cell in which the pellet is formed *in situ* and a detector based on the gas chromatography. It is proposed to use this unit for measuring the effective diffusion coefficients for the catalysts developed in this Laboratory and also for basic studies in fluid-solid reactions.

#### 15·5 *Reaction models and reactor design* : (B-14·8/68)

A reactor has been developed which uses a pulsed fluid bed for obtaining differential rate data at integral conversion levels. An arrangement was also made to examine whether the reactor is operating under fully mixed conditions. Both these were found to operate satisfactorily. Accumulation of experimental data has commenced.

Using the data on the hydrogenation of glucose in a stirred autoclave, the complete kinetic behaviour of this liquid-gas-solid system was explained on the basis of a single model. There is a definite transition from diffusion to chemical control around 100°. In the region of kinetic control, Lagmuir-Hinshelwood models are being developed for the reaction.

A theoretical study was made of the comparative merits of fixed-bed, fluid-bed and moving bed reactors for a rapidly fouling catalyst. In this study several fouling models were assumed, and criteria were developed for each of these models for a quantitative analysis of the performances of the different reactor types.

A reactor was fabricated for determining the kinetics of a reaction under adiabatic conditions. The chief advantage of such a reactor was that product analysis is not involved and the complete kinetics could be studied exclusively from the temperature profile in the reactor.

The reaction between ethylene and hydrogen chloride in a medium of nitrobenzene was studied. The solubility of the two gases in nitrobenzene was determined as a first step in analysing this reaction. An experimental assembly has also been set up for determining the diffusivity of the gases in nitrobenzene. This unit is now being standardized by determining the diffusivity of known systems.

A reactor was set up for determining the kinetics of a reaction by impregnating the catalyst on the tube walls. The analysis of data from such a reactor involves the development of the diffusion-kinetic equation for the system. Although such reactors are not generally used, they have several advantages over conventional reactors, particularly for systems known to be first-order.

The isomerisation of 2-butene to isobutylene on several catalysts was studied. A total of 50 catalysts were prepared and these were evaluated by the statistical method of group screening. The most effective group was identified and the catalysts comprising this group are now being more closely examined. Of the catalysts used, fluorinated alumina was found to be the best, while all other catalysts including molecular sieves were found to be less satisfactory. Using the best catalyst obtained, a complete analysis is being made of the complex reaction network involved.

#### 15·6 *Distillation* : (B·14·11/68)

The correlation of available data on activity coefficients was completed and a method was developed which enabled the predication of activity coefficients. This work was presented at a symposium on distillation in England.

□ □ □

## APPENDICES

### A-I SERVICE PROJECTS

#### 1. PHYSICO-ANALYTICAL WORK

Analytical and quality control work was conducted for the various projects in progress in the laboratory. Over 200 samples have been analysed during this period.

#### 2. MICROANALYSIS

Microanalysis of organic compounds for various elements (C, H, N, halogen, sulphur), functional groups (methoxyl, acyl, C-methyl, N-methyl, active hydrogen), molecular weight etc., was carried out.

1837 analyses for elements and 24 for functional groups were carried out.

#### 3. SPECTROCHEMICAL WORK

Analytical and structure elucidation work was carried out by various physico-organic techniques such as UV, IR, NMR, visible spectra and mass spectra.

Number of samples studied :

NMR	..	2404
UV	..	583
UV visible	..	182
IR	..	3596
Mass spectra	..	930

#### 4. VPC/GLC ANALYSIS

6,910 samples were analysed.

#### 5. ULTRACENTRIFUGAL ANALYSIS

140 samples were analysed.

#### 6. INSTRUMENTATION

Servicing and maintenance of the following special equipments were carried out :

NMR Spectrometer; IR, UV and visible spectrophotometers; X-ray machine; electrical furnaces; gas chromatographs, colorimeters, servo-recorders, etc.

Running and maintenance of the liquid air plant and supply of liquid air to various groups in the NCL was attended to.

Total number of jobs completed : 266

7. WORKSHOP

Special equipment fabricated:

<i>Item</i>	<i>No.</i>
(i) Electron Diffraction Camera	3
(ii) Vacuum mixer	1
(iii) Dry ice preparing apparatus	2
(iv) M. S. coiled condenser	1
(v) M. S. reactor	1
(vi) Shaft furnace for reduction roast of a mixture of ilmenite ore	1

Total number of jobs completed : 2979

8. GLASS BLOWING

Jobs completed : 4401

Standard ground glass joints fabricated : 5035

9. TECHNICAL SERVICES

I. *Research Analysis*

1. Following proposals were submitted to the Process Release Committee for approval :

(i) Economics of the following NCL processes and terms and conditions of their release :

Catechol; Polyurethane coatings; Gaskets from coir pith; Chloro-methanes; Trioctyl phosphate; Triphenyl phosphate; Disperse dyes; Simazine and Atrazine; Dissolving pulp; Monochloroacetic acid; Theophylline, Aminophylline and Caffeine; Terpeneol.

(ii) Limited exclusivity for certain processes of non-exclusive nature.

2. Sponsord project on Titanium dioxide was programmed by the application of PERT (Programme Evaluation Review Technique). A master net-

work and squared net-work were drawn indicating the activities of NCL and the sponsor.

3. Economic evaluation of production programme of NCL's Fine Chemicals Project (FCP) and costing of the 23 fine chemicals produced in FCP, Bostik type adhesives and silicone tetramer was done.

4. Cost benefit analysis of NCL Glass Blowing Section was done.

5. A study to measure the input/output of various projects in the Laboratory is in progress.

## *II. Commercial intelligence*

1. Techno-economic data on glyceryl guaiacolate, dimethyl formamide were collected.

2. Market Survey was conducted on ultrasonic interferometer, vitamin D<sub>3</sub>, quinine to quinidine, lactic acid, glyoxal 40% and acetals.

## *III. Industrial liaison*

(A) 1. Information on the performance of the NCL processes released to various parties and their experience on the know-how supplied was collected.

2. 1955 technical enquiries from different individuals, industries, Government organizations and starred questions from the Parliament were attended to.

3. Comments were offered on 88 applications for industrial licence for the manufacture of various chemicals with foreign collaboration.

4. Agreement terms for the release of 15 processes based on NCL know-how were processed.

5. Over 3,000 visitors from educational institutions, industries and the public were shown round the Laboratory during the year.

6. Plans to reorganize the Museum were drawn keeping in view its shifting to the new premises.

### *(B) Liaison with CSIR*

A close liaison was maintained with the CSIR. Every month reports relating to NCL's activities and achievements are sent for

inclusion in the Monthly Report to the Union Cabinet, CSIR News and Research Utilization Data.

Reports were sent for publication in the following :

- (i) CSIR Annual Report 1969
- (ii) CSIR Handbook 1970
- (iii) Report on Science & Technology 1969

In addition, detailed reports were sent to the CSIR relating to :

- (i) Achievements of NCL during the last 20 years
- (ii) Public Accounts Committee
- (iii) Sarkar Committee
- (iv) NCL work relating to various Union Ministries.

#### *IV. Publications*

Following publications were brought out :

- 1. Annual Report 1968-69
- 2. Summary of Achievements 1968-69

#### *Internal reports*

- 1. Half-yearly report — April - September 1969
- 2. Research programme — 1970-71

#### *V. Publicity*

As part of the publicity programme —

(A) Following papers were published :

- (i) National Chemical Laboratory  
Ranganath J. Shirur  
*Industrial Bulletin*, pp-6-8, Vol. X, No. 6, June, 1969.
- (ii) New strides in chemical research and development at NCL  
Ranganath J. Shirur  
*Indian Chemical Journal*, pp 14-22, Vol. IV, No. 2, August, 1969.

(B) Following feature and news story were published in the dailies :

- (i) NCL's contribution to plastic industry  
N. D. Ghatge; *The Economic Times*, May 19, 1969.
- (ii) 'Acetanilide', *The Indian Express*, March 2, 1970.

## VI. *Research Management Studies*

The Division has undertaken active study on various aspects of research management. Some of the major aspects are : Resource allocation for R. & D, Improving the process efficiency, Patent system, Transfer of technology, Team work etc.

A paper on 'Periodic reports of research organizations,' (A.M. Lele and Ranganath J. Shirur) was presented at the 'Seminar on Information', organized by DESIDOC, New Delhi, December 5, 1960.

### *Publication :*

Creativity in scientific institution — the problem of job incentives  
J. V. Rajan and S. K. Subramanian  
*Indian Journal of Public Administration*, pp. 410-421, Vol. 15, No. 3, 1969.

## VII. *General services*

The Division continued to render statistical, photographic, museological and draftsman services to other divisions of the Laboratory.

## A-II SERVICES RENDERED TO OUTSIDE PARTIES

### 1. *Supply of cultures*

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

### 2. *Analytical services*

During the period a large number of analyses were done for universities, research institutes, Govt. departments, private parties, etc.

The following analyses were carried out : Microanalysis, NMR, IR, UV, VPC, GLC, mass spectral analysis, surface area measurements, etc.

### 3. *Technical aid involving ad-hoc experimental, instrumental, engineering and glass-blowing services :*

<i>Nature of work</i>	<i>Name of the party</i>
1. Repairs of Perkin- Elmer (model 137) UV-visible spectrophotometer.	Institute of Armament Technology, Girinagar, Poona-25.

2. Following glass-blowing work was carried out :

- |   |  |
|---|--|
| (a) Fabrication of separating funnels capacity 500 ml. (6 Nos.) 20 Lit. round bottom flasks (4 Nos.). | M/s Coated Fabrics (Pvt.) Ltd., Poona-16.                        |
| (b) Repairs of mercury seal, water condensers, stirrers, etc.   | —do—   |
| (c) Fabrication of 20 Lit. round bottom flask and providing B- 50 M joint.                            | Small Industries Research Institute, Poona-5.                    |
| (d) Repairs of condensers (2 Nos.) and connecting parts of distillation plant.                        | Central Water and Power Research Station, Khadakwasla, Poona-24. |
| (e) Repairs of soxhlet extractors (3 Nos.).   | M/s. Sandvic Asia Ltd., Poona-12.                                |
| (f) Repairs of fractionating column.  | Medicinal Plants Schemes, Cinchona Dept., Ootacamund.            |
| 3. CdS photoconductive cells prepared as per specifications and supplied. (20 Nos.)                   | Bhabha Atomic Research Centre, Trombay, Bombay.                  |
| 4. Analysis of seat ring (carbon).  | M/s. Kirloskar Pnuematic Ltd., Hadapsar, Poona.                  |
| 5. Detection of mercury in hydrosulphite.   | M/s. K. J. Chemicals Ltd., Thana.                                |
| 6. Decolorisation and analysis of jaggery for inorganic constituents.                                 | M/s. Jaknur Jaggery P. Ltd., Jaknur (Dist. Bijapur).             |
| 7. Detection of sodium in human nails.  | B. J. Medical College, Poona-1.                                  |
| 8. Analysis of pulp and acetate samples.  | M/s. Mysore Acetate & Chemicals Co. Ltd., Bangalore-25.          |



- |  |   |
|--|---|
| 9. Crystallographic analysis of Lime sludge.                     | M/s. West Coast Paper Mills, Ltd.,<br>Bangurnagar, Dandeli. |
| 10. DTA and TGA analysis of Kaolin samples.                      | M/s. Cable Corporation of India Ltd., Bombay-1.             |
| 11. Analysis of Lloyd's wool.                                    | M/s. Punj Sons Pvt., Ltd.,<br>Calcutta-16.                  |
| 12. Grinding and sieving of burnt rice husk.                     | M/s. Sahyadri Dyestuffs & Chemicals P. Ltd., Poona-9.       |
| 13. Facilities for carrying out certain experiments.             | M/s. Kirloskar Oil Engines Ltd.,<br>Poona-3.                |
| 14. Remedies for detection of fraudulently altered bank cheques. | M/s. Bank of India, Poona-2.                                |
| 15. Facilities for carrying out some laboratory experiments.     | M/s. Soil-Tronics, Poona-4.                                 |
- Total receipts from the above analytical and other services : Rs. 27,330.00

### A-III DEMONSTRATIONS

Following NCL processes were demonstrated :

<i>Process</i>	<i>Party</i>
1. CdS photoconductive cells	M/s. Gera Engineering Co., Poona-1.
2. Dioctyl phthalate	M/s. Alta Laboratories, Khopoli.
3. Hard ferrites	M/s. Semiconductors Ltd., Poona-14.
4. <i>p</i> -Menthane hydroperoxide	M/s. Camphor and Allied Products Ltd., Bareilly.
5. Di- <i>o</i> -tolybiguanide	M/s. Industrial Perfumes Ltd., Bombay-1.

#### A-IV PROCESSES LEASED OUT DURING 1969-70

<i>Process</i>	<i>Party</i>	<i>Remarks</i>
1. Aniline	M/s. Engineers India Ltd., New Delhi.	Exclusive for engineering the project.
2. Chlorination of methane	M/s. Standard Mills Ltd., Bombay.	Released for further pilot plant work at the works.
3. Di- <i>o</i> -tolylbiguanide	M/s. Industrial Perfumes Ltd., Bombay.	Exclusive.
4. <i>p</i> -Menthane hydroperoxide	M/s. Camphor & Allied Products, Bareilly (U.P.).	Exclusive.
5. Potentiometric strip chart recorder	M/s Associated Instruments Manufacturers (India) P. Ltd., New Delhi.	Non-exclusive.

#### A-V SPONSORED PROJECTS COMPLETED DURING 1969-70

<i>Project</i>	<i>Sponsor</i>
1. Isolation of borneol	M/s. Camphor and Allied Products Ltd., Bareilly.
2. Chlorobenzenes (Predominantly MCB)	M/s. Hindustan Organic Chemicals Ltd., Rasayani.
3. Electron Diffraction Camera	(i) M/s. Fertilizer Corpn., of India Ltd., Sindri. (ii) Defence Science Laboratory, New Delhi. (iii) Gauhati University, Jalukbari (Assam).
4. 8-Hydroxyquinoline	M/s. Alta Laboratories Ltd., Khopoli.
5. Megimide	M/s. Indian Schering Ltd., Bombay-1.
6. Pentachlorophenol and other chlorophenolic compounds	M/s. Bombay Chemicals Pvt. Ltd., Bombay-1.

7. $\beta$ -Phenethyl alcohol	M/s. Sunanda Aromatics Industries, Mysore-1.
8. Pulping of Kerala hardwoods	Preinvestment Survey of Forest & Resources, Govt. of India, New Delhi.
9. Liquid stabilizer for PVC	M/s. Swastik Rubber Products Ltd. Poona-3.
10. Screening of NCL compounds for pharmacological activity	M/s. Bristol Laboratories, Syracuse, New York (USA).
11. Sulphacetamide and its sodium salt	M/s. Indian Schering Ltd., Bombay-1.

#### A-VI RECEIPTS THROUGH SPONSORED PROJECTS

	(Rs. in lakhs)
(i) Total receipts from above completed sponsored projects (Nos.11).	2.06
(ii) Total receipts from other sponsored projects during the year 1969-70 which are being continued in 1970-71.	5.11
(iii) Total receipts from sponsored projects during 1969-70.	<u>7.17</u>
(iv) Receipts on account of overhead charges credited to CSIR for undertaking above sponsored research projects.	2.86

#### A-VII TRAINING

More than 40 scientists from different universities, industries and research institutes were trained in one of the following :

Electron diffraction techniques; microanalytical techniques; IR, UV and NMR spectroscopy techniques etc.

Two scholars from National Talent Scheme (Govt. of India) received training in instrumental analysis and some synthetic techniques in organic chemistry during the summer vacation (1969).

## A-VIII SEMINARS AND LECTURES

Weekly seminars are held at NCL in which members of the scientific staff take an active interest. In addition to the talks by several NCL scientists, the following lectures were delivered by outside eminent scientists at NCL.

1. Dr. Gordon Freeman, University of Alberta, Canada.
  - (i) Conductance of organic liquids under irradiation
  - (ii) Solvated electrons in hydraulic liquids in radiolysis.
2. Prof. Roy L. Whistler, Purdue University, Lafayette, U.S.A. New starch derivatives of practical value.
3. Dr. John J. Wright, Research School of Chemistry, The Australian National University, Canberra, Australia. The brevianamides : a new class of fungal alkaloids.
4. Dr. Edwin E. Hays, Riker Laboratories, Northridge, California, U.S.A. The science of new drug development
5. Dr. L. Noda, Dartmouth College, New Hampshire, USA. Studies on myokinase
6. Prof. P. T. Narasimhan, Tata Institute of Fundamental Research, Bombay.
  - (i) Hybridization of atomic orbitals.
  - (ii) Structure and spectra of thiophenium compounds.
7. Dr. H. F. Barber, May and Baker Ltd., Essex, U. K. Some contributions of drug research to organic chemistry.
8. Prof. Ing. J. Tomko, Dept. of Alkaloids, Chemical Institute of Slovak Academy of Sciences, Bratislava, Czechoslovakia The steroid alkaloids of berabrum group.
9. Prof. E. Pungor, Univ. of Chemical Industries, Veszprem, Hungary. Ion selective electrodes, their theory and application.

10. Prof. L. Hough, Dept. of Chemistry Queen Elizabeth College, London, U. K. (i) Steric and electronic factors in the nucleophilic substitution of carbohydrates. (ii) Sucro chemistry. The chemical reactivity of sucrose and possible commercial applications.
11. Prof. J. Topping, Brunel University, U. K. Development of technical universities and their links with industries.
12. Dr. E. J. Hart, Argonne National Laboratory, Argonne, Chicago, USA Recent developments in radiation chemistry.
13. Prof. H. A. Staab, Organisch-Chemisches Institut der Universität, Heidelberg, W. Germany (i) New synthetic methods using heterocyclic amides (Azolides). (ii)  $^{13}\text{C}$  Nuclear magnetic resonance as a tool of investigating organic reaction.
14. Prof. F. Marta, Univ. of Szeged, Hungary. The interpretation of limiting rate and of induction period in oxidation benzaldehyde catalysed by cobaltous acetate.
15. Dr. S. P. Acharya, Fairdeal Corporation (P) Ltd., Bombay. Hydroboration of terpenes.

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

<i>Name of the scientist</i>	<i>Subject</i>	<i>Place</i>
1. Dr. M. N. S. Murthy	Crystallography. (10 lectures) Symmetry.	Wadia College, Poona-1. Fergusson College, Poona-4.
2. Dr. A. P. B. Sinha	Solid state devices. (5 lectures)	Winter School, I. I. T., Kanpur

- |    |                              |   |  |
|----|------------------------------|---|--|
| 3. | Dr. A. Goswami               | Thin film physics.<br><br>Defects in crystals.  | Physical Research<br>Laboratory, Ahmeda-<br>bad.<br><br>Fergusson College,<br>Poona-4. |
| 4. | Dr. Sukh Dev                 | Structure of lac resin.   | Convention of<br>Chemists, I. I. T.,<br>Kanpur.  |
| 5. | Dr. C. R. Narayanan          | Physical methods in<br>organic chemistry.<br>(3 lectures)   | Yeshwant Mahavi-<br>vidyalaya, Nanded.   |
| 6. | Dr. K. G. Das                | Organic mass spectro-<br>metry.<br><br>(i) Fragmentation of<br>germacranolides<br>and furanoger-<br>macranolides under<br>electron impact.<br><br>(ii) Structure of inter-<br>mediate ions. | I. I. T., Madras.<br><br>Indian Institute of<br>Science, Bangalore.                    |
| 7. | Mr. V. S. Krishnama-<br>char | Microbiology.<br>(6 lectures)   | Ahmednagar Science<br>College, Ahmednagar.   |

#### A-IX STAFF NEWS

##### 1. *Foreign deputations/training, etc.*

- (i) Dr. J. Gupta visited USA under the India-U.S.A. Scientific Exchange Programme 1969. He visited selected research and development laboratories of universities as well as of the chemical industry in U.S.A. (Sept./October 1969).
- (ii) Dr. Sukh Dev visited University of Georgia, Chemistry Department, Athens, Ga. U. S. A. as a visiting professor to give a series of lectures on recent advances in terpenoids (March/May 1969).
- (iii) Dr. B. D. Tilak toured USA under the auspices of the India-USA Scientific Exchange Programme 1969 visiting university and industries' research laboratories, where he delivered a series of 17

lectures. He also attended the American Chemical Society Organic Chemistry Symposium at Salt Lake city. He visited industrial research and university laboratories in Switzerland, W. Germany, Belgium, U. K., Japan and Singapore to give lectures (May-June 1969).

## 2. Participation of NCL scientists in seminars

<i>Symposia, Seminar, etc., and place</i>	<i>Name of the scientist</i>
1. Winter School in Solid State Chemistry; I. I. T., Kanpur.	Dr. A. P. B. Sinha, Dr. V. J. Rao, Dr. M. N. S. Murthy, Miss N. R. Pawaskar
2. Materials Science Symposium, NAL, Bangalore.	Dr. A. P. B. Sinha
3. National Conference on Electronics; T.I.F.R., Bombay.	Dr. C. A. Menezes, Mr. S. D. Bakare
4. Chemistry Symposium, Punjab University, Chandigarh.	Dr. A. P. B. Sinha, Dr. M. N. S. Murthy, Dr. D. N. Sen
5. Nuclear Physics & Solid State Symposium, University of Roorkee, Roorkee.	Mr. S. S. Shah
6. Indo-Soviet symposium on "The chemistry of natural products including pharmacology", National Institute of Sciences of India, New Delhi.	Dr. Sukh Dev, Dr. U. R. Nayak, Dr. A. S. Gupta, Mr. A. P. Joshi, Mrs. V. Gogte, Mr. A. S. Narula, Dr. C. R. Narayanan, Dr. K. K. Chakravarti, Dr. A. S. Rao, Dr. K. Venkataraman, Dr. A. V. Rama Rao
7. Convention of Chemists, I.I.T., Kharagpur	Dr. Sukh Dev, Dr. C. R. Narayanan, Dr. V. Jagannathan, Mr. V. K. Pawar
8. Mass Spectrometry Workshop Seminar, I.I.T., Kanpur.	Dr. K. G. Das, Mr. M. S. B. Nayar

9. U.S. Science Foundation and UGC-Workshop on chemical Education; Bangalore. Dr. B. D. Tilak
10. Convention of Society of Biological Chemists (India), Hindustan Lever House, Bombay. Dr. V. Jagannathan  
Mr. H. I. Rahatekar,  
Miss F.S. Maskati
11. Symposium on Applied Thermodynamics; University Dept. of Chemical Technology Bombay. Dr. L. K. Doraiswamy
12. Lecture series on 'Recent Advances in Dyes and Pigments', Indian Chemical Manufacturers' Association, Bombay. Dr. B. D. Tilak
13. Indian National Science Academy & National Academy of Sciences (USA) — Seminar on 'Management of Industrial Research', Baroda. Dr. B. D. Tilak  
Dr. R. B. Mitra  
Mr. A. M. Lele

### 3. Awards and Honours

1. Dr. A. Goswami was awarded D.Sc., degree by London University, U. K. for his work on 'Structures and physics of thin films.'
2. Dr. B. D. Tilak was awarded the K. Venkataraman Lectureship of the Bombay University. He delivered two lectures in this connection at Bombay University Dept. of Chemical Technology, Bombay.

### 4. Post-graduate degrees received by NCL staff and research fellows

Name	Degree	University
1. Abhyankar, S. M.	Ph.D. (Tech.)	Nagpur
2. Arjungi (Mrs.), Kusum	Ph.D.	Bombay
3. Badrinarayanan, S.	Ph.D.	Madras
4. Bozidar, Antic	Ph.D.	Poona
5. El-Namaky Hassan M. El-Shazly	Ph.D.	Poona



<i>Name</i>	<i>Degree</i>	<i>University</i>
6. Gadgil, D. D.	Ph.D.	Poona
7. Gadgil, L. H.	Ph.D.	Poona
8. Hungund, B. L.	Ph.D.	Poona
9. Kalyanaraman, S.	Ph. D. (Tech.)	Bombay
10. Ladwa, P. H.	Ph.D.	Poona
11. Mahadik, S. P.	Ph.D.	Poona
12. Nikam P. S.	Ph.D.	Poona
13. Pathak, Gopal	Ph.D.	Poona
14. Pradhan, S. D.	M.Sc.	Bombay
15. Quasim, Chelat	Ph.D.	Poona
16. Rangarao, S.	M.Sc. (Tech.)	Bombay
17. Salema (Mrs.)Mowalia Abd-EL Rahman Mohamed	Ph.D.	Poona
18. Sansare, S. D.	M.Sc.	Bombay
19. Shah, J. N.	Ph.D. (Tech.)	Bombay
20. Shaikh, I. N.	Ph.D.	Poona
21. Tirodkar, S. V.	Ph.D.	Shivaji
22. Vaidya, A. S.	Ph.D.	Poona
23. Yadav, S. D.	Ph.D.	Poona

Of the above, 14 are NCL staff, 7 are research fellows and 2 are guest workers.

*5. NCL scientists recognized by different universities as research guides*

Dr. Ayyangar, N. R.	Poona
Dr. Bose, J. L.	Bombay, Poona, Nagpur, Shivaji
Dr. Chakravarti, K. K.	Poona, Shivaji, Karnatak
Dr. Dadape, V. V.	Bombay, Poona, Karnatak, Vikram, Banaras
Dr. Damodaran, V.	Shri Venkateswara University, Tirupathi
Dr. Das, K. G.	Poona, Bombay, Kerala
Dr. Doraiswamy, L. K.	Poona, Bombay, Nagpur, Calcutta, Jadavpur.
Dr. Ghatge, B. B.	Poona.
Dr. Ghatge, N. D.	Poona, Bombay, Shivaji,
Dr. Gogate, V. N.	Shivaji
Dr. Goswami, A.	Calcutta, Poona, Punjab
Dr. Gupta, J.	Bombay, Poona, Punjab, Madras
Dr. Ingle, T. R.	Poona

Dr. Jagannathan, V.	Bombay, Poona
Dr. Joshi, R. M.	Poona
Dr. Kapur, S. L.	Bombay, Poona, Punjab
Dr. Katti, S. S.	Bombay
Dr. Kelkar, G. R.	Poona
Dr. Kulkarni (Miss) S. B.	Poona
Dr. Kulkarni, S. N.	Poona, Bombay, Karnatak, Shivaji
Dr. Mathur, H. B.	Poona, Agra, Madras, Bombay, I I T., Bombay.
Dr. Nair, P. M.	Poona, Shivaji, Andhra.
Dr. Narayanan, C. R.	Poona, Bombay, A. B. Uni., Zaria, Nigeria.
Dr. Pai, M. U.	Bombay,
Dr. Pant, L. M.	Poona.
Dr. Ramchandran, B. V.	Shivaji, Poona
Dr. Rangachari, P. N.	Poona, Shivaji.
Dr. Rao, A. S.	Poona, Shivaji.
Dr. Roychowdhury, P.	Marathwada
Dr. Sadana, J. C.	Poona, Aligarh.
Dr. Sen, D. N.	Poona, Bombay.
Dr. Sinha, A. P. B.	Poona, Banaras, Vikram, Karnatak, Andhra.
Dr. Sivaraman, C.	Poona.
Dr. Subbaraman, P. R.	Poona, Bombay, Gujarat, Kerala.
Dr. Sukh Dev	Poona, Bombay, Agra, Punjab, I. I. Science, Bangalore.
Dr. Tilak, B.D.	Bombay, Poona.
Dr. Venkataraman, K.	Poona, Bombay, Madras, Banaras,

## 6. Consultancy

### *NCL Scientists working as institutional consultants*

1. Dr. Chakravarti, K. K. M/s. Sunanda Aromatic Industries,  
Mysore.
2. Dr. Ghatge, N. D. M/s. Swastik Rubber Products Ltd.,  
Poona.
3. Dr. Mitra, R. B. M/s. Alta Laboratories, P. Ltd.,  
Khopoli.
4. Dr. Sukh Dev M/s. Camphor & Allied Products  
Ltd., Bareilly.

5. Dr. Tilak, B. D.

M/s. Aniline Dyestuffs & Pharmaceuticals (P) Ltd., Bombay.

M/s. Indian Dyestuff Industries, Ltd., Kalyan.

M/s. Hindustan Organic Chemicals Ltd., Rasayani, have appointed NCL as their technical consultants on a lump sum annual remuneration of Rs. 50,000/- initially for one year.

CSIR share on account of technical consultancy.

Rs. 80,000/-

## A-X PUBLICATIONS

### *Research Papers*

1. Neurgaonkar, R. R. and Sinha, A. P. B.  
Structural & Optical Properties of (Ca, Cd) halophosphates.  
*Indian J. Pure Appl. Phys.* **7**, 402 (1969).
2. Neurgaonkar, R. R.  
Structural and optical properties of (Ca, Mg) & (Ca, Be)  
fluorophosphates.  
*Indian J. Pure Appl. Phys.*, **7**, 480 (1969).
3. Murthy, M. N. S., Sinha, A. P. B. and Sinha, K. P.  
Optical absorption spectra of  $d^2$  and  $d^3$  ions in trigonal  
bipyramidal complexes.  
*Indian J. Pure Appl. Phys.*, **7**, 665 (1969).
4. Murthy, M. N. S.  
Recovery and reactivation of nickel from spent catalyst of the oil  
hydrogenation industry.  
*Indian J. Tech.*, **7**, 224 (1969).
5. Rao, V. J., Rao, D. R.. and Sinha, A. P. B. Optical properties of some  
Europium chelates.  
*Indian J. of Chem.*, **8**, 270 (1970).
6. Srivastava, S. K. and Sinha, A. P. B.  
Space charge limited current through chromium cadmium sulphide—  
chromium diodes.  
*Solid State Electronics*, **13**, 57 (1970).
7. Sinha, A. P. B.  
Studies on some magnetically ordered solids.  
Proceedings of the Chemistry Symposium, Chandigarh, 104 (1969).
8. Pethe, L. D. and Mathur, H. B.  
Transition from internal to external oxidation in dilute tin-silver alloys.  
*Indian J. Chem.*, **7**, 340 (1969).

9. Jagannathan, R., Thacker (Miss), R. and Mathur, H. B.  
Mossbauer spectroscopic study of the effects of electron capture in  $K^{57}\text{CoF}_3$ .  
*Indian J. Chem.*, **7**, 353 (1969).
10. Rao, U. B. and Mathur, H. B.  
Thermochemical studies: The effect of substituents on the heat of ionization of  $\beta$ -diketones.  
*Indian J. Chem.*, **7**, 1234 (1969).
11. Raju, E. V. and Mathur, H. B.  
Thermochemical studies: The heats and entropies of reactions of transition metal ions with histidine.  
*J. Inorg. & Nucl. Chem.*, **31**, 425 (1969).
12. Jagannathan, R. and Mathur, H. B.  
Mossbauer spectroscopic determination of the valency states of iron formed in the decay of  $^{57}\text{Co}^{24}$  in  $\text{Co}[\text{Cr}_2]_{10}$  and  $\text{CoCr}_2\text{S}_4$ .  
*J. Inorg. Nucl. Chem.*, **31**, 3363 (1969).
13. Srinivasan, T. K. K., Balakrishnan, I. and Reddy, M. P.  
On the nature of the products of gamma radiolysis of aerated aqueous solutions of benzene.  
*J. Phys. Chem.*, **73**, 2071 (1969).
14. Balakrishnan, I. and Reddy, M. P.  
Mechanism of the reaction of the hydroxyl radical with benzene in the radiolysis of the aerated aqueous benzene system.  
*J. Phys. Chem.*, **74**, 850 (1970).
15. Mathur, H. B. and Gupta, M. P.  
A Mossbauer spectroscopic study of the nature of the bonding in bis-dithioacetylacetonate tetrachloroferrate (II),  
*Chemical Physics Letters*, **3**, 191 (1969).
16. Yagnik, C. M. and Mathur, H. B.  
Electric field gradients in normal spinels.  
*Molecular Physics*, **16**, 625 (1969).
17. Goswami, A.  
Growth of cadmium and zinc electro-deposits of copper on single crystals.  
*Indian J. Pure Appl. Phys.*, **7**, 232 (1969).

18. Goswami, A. and Jog, R.H.  
Electron diffraction studies of SnTe films.  
*Indian J. Pure Appl. Phys.*, **7**, 273 (1969).
19. Dhere, N. G. and Goswami, A.  
Growth of vapour phase deposits of CdSe and CdTe on single crystal substrates. .  
*Indian J. Pure Appl. Phys.*, **7**, 398 (1969).
20. Deokar, V. D. and Goswami, A.  
Hall effect of vacuum deposited mercury selenide films.  
*Indian J. Pure Appl. Phys.*, **8**, 93 (1970).
21. Dhere N. G. and Goswami, A.  
Electron diffraction studies of zinc telluride and zinc selenide films.  
*Thin Solid Films*, **3**, 439 (1969).
22. Dhere, N. G. and Goswami, A.  
Growth of vapour phase deposits of Ag<sub>2</sub>Se and Ag<sub>2</sub>Te on single crystals.  
*Thin Solid Films*, **5**, 137 (1970).
23. Gadgil, L. H. and Goswami, A.  
Epitaxial growth of Sb<sub>2</sub>Te<sub>3</sub> films.  
*J. Vac. Sci. Tech.*, **6**, 591 (1969).
24. Pawaskar (Miss), N. R., and Menzes, C. A.  
Electrical conductivity and photoconductivity relaxation in chemically deposited cadmium sulphide layers.  
*Japanese J. Appl. Phys.*, **9**, 212 (1970).
25. Roy Chowdhury, P.  
Ultrasonic velocity and adiabatic compressibility of poly (methacrylamide) in aqueous solution at 25°.  
*Indian J. Chem.*, **7**, 692 (1969).
26. Roy Chowdhury, P.  
Adiabatic compressibility of polyelectrolytes in aqueous solution, II poly (4-vinyl N-n butyl pyridinium bromide).  
*J. Polymer Sci. A-2*, **7**, 1451 (1969).
27. Sen, D. N., Umopathy, P. and Hundekar, A. M.  
Spectrophotometric determination of Fe<sup>3+</sup> with salicylic acid-formaldehyde polymer.  
*Indian J. Appl. Chem.*, **31**, 185 (1968).

28. Sen, D. N., and Kantak, U. N.  
Cyclopentadienyltitanium (IV) chelates.  
*J. Indian Chem. Soc.*, **46**, 358 (1969).
29. Parulekar, A. D. and Subbaraman, P. R.  
Polarographic behaviour of manganese in sulphosalicylic acid.  
*Indian J. Chem.*, **8**, 266 (1970).
30. Narayanan, A. and Subbaraman, P. R.  
Molybdenum catalysed hydrogen waves of the dropping mercury electrode.  
Proceedings of Symposium on Electrode Processes, Jodhpur, 17, (1969).
31. Narula, A. S. and Sukh Dev  
Oxidation of ozonides with chromic acid.  
*Tetrahedron letters*, 1783, (1969).
32. Bisarya, S. C., Nayak, U. R. and Sukh Dev  
Further Rearrangement of Isolongifolene.  
*Tetrahedron Letters*, 2323, (1969).
33. Wadia, M. S., Khuřana, R. G., Mhaskar, V. V. and Sukh Dev  
Chemistry of lac resin—I; Lac acids (Part I): butolic, jalaric and laksholic acids.  
*Tetrahedron*, **25**, 3841 (1969).
34. Singh, A. N., Upadhye, A. B., Wadia, M. S., Mhaskar, V. V. and Sukh Dev  
Chemistry of lac resin—II; Lac acids (Part 2): laccijalaric acid.  
*Tetrahedron*, **25**, 3855 (1969).
35. Ranganathan, R., Nayak, U. R., Santhanakrishnan, T. S. and Sukh Dev  
Studies in sesquiterpenes—XL isolongifolene (Part 1): structure.  
*Tetrahedron*, **26**, 621( 1970).
36. Prahlad, J. R. and Sukh Dev  
Studies in sesquiterpenes—XLI isolongifolene (Part 2) : dehydrogenation.  
*Tetrahedron*, **26**, 631 (1970).
37. Santhanakrishnan, T. S., Nayak, U. R. and Sukh Dev  
Studies in sesquiterpenes—XLII  
Isolongifolene (Part 3): systematic degradation.  
*Tetrahedron*, **26**, 641 (1970).

38. Sobti, R. R. and Sukh Dev  
Studies in sesquiterpenes—XLIII.  
isolongifolene (Part 4) : synthesis.  
*Tetrahedron*, **26**, 649 (1970).
39. Santhanakrishnan, T. S., Sobti, R. R., Nayak, U. R. and Sukh Dev  
Studies in sesquiterpenes—XLIV isolongifolene (Part 5):  
Rearrangement of isolongifolene epoxide.  
*Tetrahedron*, **26**, 657, (1970)
40. Prahlad, J. R., Nayak, U. R., and Sukh Dev  
Studies in sesquiterpenes —XLV structure of an alcohol from  
hydration of longifolene.  
*Tetrahedron*, **26**, 663 (1970).
41. Narayanan, C. R., Sarma, N. R., Srinivasan, T. K. K. and Wadia, M. S.  
Conformation of the esters of steroid hydroxyl groups by infrared  
spectroscopy.  
*Canadian J. Chem.*, **47**, 1061 (1969).
42. Narayanan, C. R. and Parkar (Miss), M. S.  
A novel reaction of nitric acid with steroids.  
Abstracts of the II Indo-Soviet symposium on the chemistry of  
natural products, Part II, 82 (1970).
43. Ingle, T. R. and Bose, J. L.  
Nature of hemicelluloses of bamboo (*D. strictus*).  
*Indian J. Chem.*, **7**, 783, (1969).
44. Landge, A. B., Ingle, T. R. and Bose, J. L.  
Stannic chloride catalysed synthesis of aryl thioglycoside.  
*Indian J. Chem.*, **7**, 1200 (1969).
45. Negi, J. S., Ingle, T. R. and Bose, J. L.  
Structure of main hemicellulose of bamboo (*D. strictus*).  
*Indian J. Chem.*, **8**, 44 (1970).
46. Bose (Miss), B., Janaki (Miss), N., Ingle, T. R. and Bose, J. L.  
Action of hydrochloric acid on CNS-pulp.  
*Indian J. Tech.*, **7**, 256 (1969).
47. Ingle, T. R. and Bose, J. L.  
Stannic chloride catalysed synthesis of glycosides.  
*Carbohydrate Res.*, **12**, 459 (1970).



48. Das, K. G. and Nayar, M.S.B.  
Fragmentation of anilides.  
*Indian J. Chem.*, 7, 650 (1969).
49. Bannore, S. N., Bose, J. L., Das, K. G. and Gogte, V. N.  
Mass Spectral identification of arylcinnolines and arylquinoxolines.  
*Indian J. Chem.*, 7, 654 (1969).
50. Sengupta, P., Dey, A. K., Mukherjee, J., Ghosh, S. and Das, K. G.  
Terpenoids of the bark of *Rhododendron falconeri* Sm.  
*J. Indian Chem. Soc.*, 46, 775 (1969).
51. Sathe (Miss), R. N., Kulkarni, G. H., Kelkar, G. R. and Das, K. G.  
Fragmentation of costunolide and its derivatives under electron impact.  
*J. Org. Mass Spectrom.*, 2, 935 (1969).
52. Singh, B. B., Ranganathan, S. and Das, K. G.  
A new approach to octaphenyl fulvalene.  
Convention of Chemists, I.I.T., Kharagpur,  
Abstract 65 (1969).
53. Tilak, B. D. and Panse, G. T.  
Synthesis of sulphur heterocyclics: Part V — Cyanine dyes derived  
from thianaphthalenium and thiaphenanthrenium salts.  
*Indian J. Chem.*, 7, 311 (1969).
54. Tilak, B. D. and Panse, G. T.  
Synthesis of sulphur heterocyclics : Part VI —  
Synthesis of thiacomarins and 5, 6-benzo-4-phenyl-thiacoumarin.  
*Indian J. Chem.*, 7, 315 (1969).
55. Subbaswami, K. N. and Tilak, B. D.  
Synthesis of nitrogen heterocyclics : Part IV —  
Synthesis of dihydroquinolines.  
*Indian J. Chem.*, 7, 419 (1969).
56. Subbaswami, K. N., Gogte, V. N., Das, K. G. and Tilak, B. D.  
Synthesis of nitrogen heterocyclics : Part V —  
Reactions of dihydroquinolines.  
*Indian J. Chem.*, 7, 422 (1969).
57. Jindal, S. L. and Tilak, B. D.  
Synthesis of sulphur heterocyclics : Part VII —  
Cationoid sulphur containing analogues of carcinogenic hydrocarbons.  
*Indian J. Chem.*, 7, 637 (1969).

58. Tilak, B. D. and Jindal, S. L.  
Synthesis of sulphur heterocyclics : Part VIII —  
Cyclodehydration of 2-arylmercaptomethylene — I — cycloalkanones  
involving a novel rearrangement.  
*Indian J. Chem.*, 7, 737 (1969).
59. Tilak, B. D., Gogte, V. N. and Jindal, S. L.  
Synthesis of sulphur heterocyclics : Part IX —  
NMR spectra of polycyclic sulphur compounds.  
*Indian J. Chem.*, 7, 741 (1969).
60. Jindal, S. L. and Tilak, B. D.  
Synthesis of sulphur heterocyclics : Part X —  
Mono- and di-cationoid heterocyclic systems  
containing 1 and 2 — S-atoms.  
*Indian J. Chem.*, 7, 948 (1969).
61. Arjungi, K. N., Gogte, V. N. and Tilak, B. D.  
Synthesis of nitrogen heterocyclics : Part VII —  
Synthesis of 2, 3-dihydrofuro (3,2-C) quinolines,  
*Indian J. Chem.*, 7, 952 (1969).
62. Tilak, B. D. and Malte, A. M.  
Synthesis of sulphur heterocyclics : Part XI —  
Synthesis of 3-(6'-methoxy-2'-thianaphthenyl) —  
2-methylpentanoic acid.  
*Indian J. Chem.*, 7, 1173 (1969).
63. Tilak, B. D. and Malte, A. M.  
Synthesis of *dl*-mycolipenic acid and other branched chain fatty acids.  
*Indian J. Chem.*, 7, 1175 (1969).
64. Tilak, B. D.  
Studies in inter-relation of structure of some dyes and their dyeing  
properties for synthetic fibres.  
*Textile Dyers & Printers* (India), 3, 34 (1969).
65. Tilak, B. D., Berde (Miss), H. V., Gogte, V. N. and  
Ravindranathan, T.  
Synthesis of tetrahydroacridine and phenanthridines by novel re-  
arrangement.  
*Indian J. Chem.*, 8, 1 (1970).

66. Gogte, V. N., El-Namaky, H. M., Salama, M. A. and Tilak, B. D. Synthesis of tetrahydroquinolines involving rearrangement of N-arylazetidines as likely intermediates. *Tetrahedron Letters*, 3319 (1969).
67. Gogte, V. N., Salama, M.A. and Tilak, B. D. Synthesis of nitrogen heterocyclics, Part VI - Stereochemistry of hydride transfer in acid-catalysed disproportionation of 3, 4-disubstituted-1, 2- dihydroquinolines. *Tetrahedron*, 27, 178 (1970).
68. Ayyengar, N. R., Rama Rao, A. V. and Venkataraman, K. Applications of NMR and mass spectroscopy to some problems concerning synthetic dyes, Part IV. NMR spectra and structures of reduction products of purpurin and its 2-methyl ether. *Indian J. Chem.*, 7, 533 (1969).
69. Pandhare, E. D., Rama Rao, A. V. and Shaikh, I. N. Lac pigments, Part III. Isolation of laccaic acids A & B and the constitution of laccaic acid A. *Indian J. Chem.*, 7, 977 (1969).
70. Bhide, N. S., Pandhare, E. D., Rama Rao, A. V., Shaikh, I. N. and Srinivasan, R. Lac pigments, Part IV. The constitution of laccaic Acid B. *Indian J. Chem.*, 7, 987 (1969).
71. Bhide, N. S. and Rama Rao, A. V. Lac pigments, Part V. The constitution and synthesis of erythrolaccin. *Indian J. Chem.*, 7, 996 (1969).
72. Bhagwanth, M.R.R., Rama Rao, A. V. and Venkataraman, K. Applications of NMR and mass spectroscopy to some problems concerning synthetic dyes, Part V. Kolbe-Schmitt reaction products from 2-hydroxycarbazole and the structure of naphthol AS-LB. *Indian J. Chem.*, 7, 1065 (1969).
73. Patil, V. B., Rama Rao, A. V. and Venkataraman, K. Cassiamin A, B & C. Three 2, 2-bianthraquinonyls in *Cassia siamea*. *Indian J. Chem.*, 8, 109 (1970).

74. Bhagwanth, M. R. R., Manjrekar, T. G., Rama Rao, A. V. and Venkataraman, K.  
Application of NMR and mass spectroscopy to some problems concerning synthetic dyes.  
Proceedings of the fourth symposium, Centre of advanced study in applied chemistry, Deptt. of Chemical Technology, University of Bombay, Feb., 1969.
75. Patil, L. J., Dixit, S. M., and Rao, A. S.  
Synthesis of (+) 4-dimethyl dihydroeudesmol and some transformations of (—)  $\alpha$ -santonin.  
*Indian J. Chem.*, **1**, 224 (1969).
76. Vaidya, A. S. and Rao, A. S.  
Synthesis of *Saussurea lactone*.  
*Indian J. Chem.*, **7**, 307 (1969).
77. Unde (Miss), N. R., Kulkarni, G. H. and Kelkar, G. R.  
Action of formic acid on dehydrocostus lactone.  
*Indian J. Chem.*, **7**, 832 (1969).
78. Narayanswamy, M., Sathe, V. M. and Rao, A. S.  
Some stereospecific addition reactions of carvone and *cis*-carveol  
*Chem. & Ind.*, 921 (1969).
79. Devgan, O. N., Bokadia, M. M., Bose, A. K., Trivedi, G. K. and Chakravarti, K. K.  
The structure and stereochemistry of lyratol, a new C<sub>10</sub> alcohol from *Cyathocline lyrata*.  
*Tetrahedron*, **25**, 3217 (1969).
80. Umrani, D. C., Gore, K. G. and Chakravarti, K. K.  
Khusimol, a new sesquiterpene primary alcohol from vetiver oil.  
*P. & E. O. R.*, **60**, 307 (1969).
81. Umarani, D. C., Gore, K. G. and Chakravarti, K. K.  
Iso-khusimol, a sesquiterpene primary alcohol from vetiver oil.  
*P. & E. O. R.*, **60**, 314 (1969).
82. Gupta, G. L., Nigam, S. S., Sastry, S. D. and Chakravarti, K. K.  
Investigation on the essential oil from *Cajanus cajan*.  
*P. & E. O. R.*, **60**, 329 (1969).
83. Gundiah, S. and Kawai, T.  
Studies on polyvinyl acetate solutions by means of radiation induced crosslinking.  
*Indian J. Chem.*, **7**, 1125 (1969).

84. Kapur, S. L., Sehra, J. C. and Subramanian, Y. V.  
Polystyrene foam.  
*Petroleum and Hydrocarbons*, **4**, 29 (1969).
85. Dixit, S. S., Deshpande, A. B., Anand, L. C. and Kapur, S. L.  
Stereoblock polymerization of methyl methacrylate with  $\text{VOCl}_3$ -  
 $\text{Al}(\text{C}_2\text{H}_5)_3$  catalyst system.  
*J. Polymer Sci. A-1*, **7**, 1973 (1969).
86. Ranga Rao, S., and Kapur, S. L.  
Grafting of acrylonitrile onto cellulose initiated by ceric ion.  
*J. Appl. Poly. Sci.*, **3**, 2649 (1969).
87. Gundiah, S., Mohite, R. B. and Kapur, S. L.  
The unperturbed dimensions of polymethyl methacrylate.  
*Makromol. Chem.*, **123**, 151 (1969).
88. Hungund, B. L., Bhattacharyya, P. K. and Rangachari, P. N.  
Methylisopropyl ketone from a terpene fermentation by the soil  
*Pseudomonad*, PL-Strain.  
*Indian J. Biochem.*, **7**, 80 (1970).
89. Phadtare, P. G. and Doraiswamy, L. K.  
Koble-Schmitt carbonation of 2-naphthol :  
(1) Confirmation of the mass transfer model, and  
(2) Process optimization.  
*Ind. Eng. Chem./Process Development & Design*, **8**, 165 (1969).
90. Goyal, P. and Doraiswamy, L. K.  
Controlling mechanisms in the vapour-phase ethylation of aniline.  
*Ind. Eng. Chem./Process Development & Design*, **9**, 26 (1970).
91. Sastri, S. R. S., Ramana Rao, M. V., Reddy, K. A. and Doraiswamy,  
L. K.  
A generalised method for estimating the latent  
heat of vaporization of organic compounds.  
*Brit. Chem. Eng.*, **14**, 959 (1969).
92. Reddy, K. A. and Doraiswamy, L. K.  
Controlling mechanisms in the selective oxidation of toluene to  
benzaldehyde.  
*Chem. Eng. Sci.*, **24**, 1415 (1969).
93. Babu Rao, K. and Doraiswamy, L. K.  
Combined reactors.  
*A. I. Ch. E. Journal*, **16**, 273 (1970).

94. Parande, M. G.  
Studies on the solubility of anhydrous hydrogen chloride gas in benzene, toluene and xylene.  
*Chemical Age of India*, 4, 18 (1969).
95. Bhat, N. A.  
Chlorination of ethylene thermodynamic consideration.  
*Chemical Age of India*, 20, 839 (1969).
96. Parande, M. G. and Deshpande, A. B.  
PVC and its application in chemical plants.  
*Indian Chemical Manufacturers' Journal*, 7, 21 (1969).
97. Parande, M. G. and Balakrishnan, S.  
Continuous fractional crystallisation.  
*Chemical Processing and Engineering*, 4, 26 (1970).

*Papers sentread at symposium, seminar, etc.*

1. Sinha, A. P. B. and Murthy, M. N. S.  
Absorption spectra of trigonal bipyramidal vanadous complexes.  
Department of Atomic Energy Chemistry Symposium on  
'Structural inorganic chemistry' Chandigarh, September, 1969.
2. Sen, D. N. and Kantak, U. N.  
*Bis* ( $\pi$ -cyclopentadienyl) titanium (IV) complexes with sulphur containing ligands.  
Department of Atomic Energy-Chemistry symposium on  
'Structural inorganic chemistry', Chandigarh, September, 1969.
3. Sukh Dev and Nayak, U. R.  
Absolute configuration of (+) -bakuchiol.  
Indo-Soviet symposium on 'The chemistry of natural products including pharmacology', New Delhi, February, 1970.
4. Sukh Dev and Gupta, A. S.  
Terpenoids of *Dipterocarpus pilosus*.  
Indo-Soviet symposium on 'The chemistry of natural products including pharmacology', New Delhi, February, 1970.
5. Sukh Dev and Joshi, A. D.  
Photochemistry of longibornyl iodide.  
Indo-Soviet symposium on 'The chemistry of natural products including pharmacology', New Delhi, February, 1970.

6. Sukh Dev and Gogte (Mrs.), V. V.  
Hydroximalabaricol — a new member of malaboricane class of triterpenoids.  
Indo-Soviet symposium on 'The chemistry of natural products including pharmacology', New Delhi, February, 1970.
7. Sukh Dev and Narula, A. S.  
A novel systematic degradation of side chain of cycloartenol.  
Indo-Soviet symposium on 'The chemistry of natural products including pharmacology', New Delhi, February, 1970.
8. Narayanan, C. R.  
A novel reaction of nitric acid with steroids.  
Indo-Soviet symposium on 'The chemistry of natural products including pharmacology', New Delhi, February, 1970.
9. Rao, J. M., Patil, S. D. and Nair, P. M.  
Structure and mutarotation of ascorbic acid osazones.  
Symposium on 'Magnetic resonance', Kharagpur, January, 1970.
10. Jagannathan, V. and Powar, V. K.  
*Bacillus subtilis* phytase.  
Convention of chemists, Kharagpur, December, 1969.
11. Rahatekar, R. I., Jagannathan V. and Rao, M.R.R.  
Purification and properties of D (+) -malic enzyme.  
Convention of society of biological chemists (India), Bombay, December, 1969.
12. Jagannathan, V., Rao, M.R.R. and Maskati (Miss), F. S.  
Bacterial degradation of laevorotatory citramalate.  
Convention of society of biological chemists (India), Bombay, December, 1969.
13. Doraiswamy, L. K.  
Estimating heats of fusion.  
Symposium on 'Applied thermodynamics', Bombay, March, 1970.
14. Balakrishanan, S., Krishnan, V. and Eduljee, H. E.  
Prediction of activity coefficients from the properties of pure components.  
International symposium on 'Distillation', Brighton, England, September, 1969.

*Other publications*

1. Subbaraman, P. R., Cordes, S. M. and Henry Freiser.  
Effect of auxiliary complexing agents on the rate of extraction of zinc and nickel with diphenyl thiocarbazone.  
*Anal. Chem.*, **41**, 1878 (1969).
2. Rangachari, P. N.  
The total synthesis of an enzyme.  
*J. Sci. Industr. Res.*, **28**, 279 (1969).



## A-XI PATENTS IN FORCE

### *Patents sealed*

1. **54867\***  
A process for the manufacture of nicotine sulphate from tobacco and tobacco wastes.  
Bijawat, H. C., Razdan, R. and Potnis, G. V.
2. **56726\***  
Preparation of water dispersible DDT as an oil bound paste.  
Rao, K. V. N., Bhide, S. P., Kulkarni (Miss), S. B. and Biswas, A. B.
3. **57888\***  
Improvements in or relating to the production of hydroxy, alkoxy or aryloxy substituted aryl alkyl ketones.  
Bose, J. L. and Shah, R. C.
4. **58868**  
A process for the preparation of azelaic acid semiester suitable for making civetone dicarboxylic acid.  
Nayak, U. G., Chakravarti, K. K. and Bhattacharyya, S. C.
5. **59419**  
A process for the preparation of tridecane 1 : 13 dicarboxylic acid or its ester, suitable for the preparation of exaltone (cyclopentadecanone).  
Ghatge, B. B., Nayak, U. G., Chakravarti, K. K. and Bhattacharyya, S. C.
6. **59497\***  
Production of porous polymer suitable for preparing cation exchange-resins.  
Govindan, K. P., Pandya, R. N. and Krishnaswamy, N.
7. **59606\***  
Preparation of cation exchange resin from porous cashewnut shell liquid polymer.  
Krishnaswamy, N., Pandya, R. N. and Govindan, K. P.
8. **59608\***  
Rigid filters.  
Kapur, S. L. and Pandya, R. N.

9. **60555\***  
Production of liquid rubber.  
Uma Shankar.
10. **60826**  
Improvements in or relating to the production of hydroxy, alkoxy or aryloxy substituted deoxybenzoins and particularly of deoxyanisoin.  
Bose, J. L. and Shah, R. C.
11. **62890\***  
A new process for the production of 4-hydroxycoumarin and its derivatives.  
Shah, V. R., Bose, J. L. and Shah, R. C.
12. **63083\***  
A new method for the preparation of 4-hydroxycoumarins.  
Shah, V. R., Bose, J. L. and Shah, R. C.
13. **64958\***  
Improvements in or relating to polishing compositions.  
Shah, S. M., Hinge, V. K., Mhaskar, V. V. and Shah, R. C.
14. **65440\***  
A process for the extraction of wax from sisal waste.  
Shah, S. M., Hinge, V. K., Mhaskar, V. V. and Shah, R. C.
15. **65543**  
A process for the preparation of  $\omega$ -dicarboxylic acids and  $\omega$ -hydroxy acids suitable for conversion to macrocyclic compounds.  
Mathur, H. H. and Bhattacharyya, S. C.
16. **65778**  
Improvements in or relating to the production of transdiethylstilbestrol dimethyl ether and allied stilbenes.  
Joshi, C. G., Bose, J. L. and Shah, R. C.
17. **65976\***  
Improvements in or relating to suspension polymerization of vinyl monomers.  
Joshi, R. M. and Kapur, S. L.
18. **65977\***  
Rubber-base adhesive.  
Uma Shankar.

19. **66096\***  
A process for the production of bacterial diastase by submerged culture.  
Babbar, I. J., Bekhi, R. M. and Srinivasan, M. C.
20. **66194\***  
Improvements in or relating to can sealing composition.  
Raghunath, D. and Kapur, S. L.
21. **66803**  
Improvements in or relating to the manufacture of pressure sensitive adhesive tapes.  
Kapur, S. L. and Rao, B. R. K.
22. **66966\***  
An improved process for the manufacture of porous rigid filters.  
Kapur, S. L. and Pandya, R. N.
23. **67490**  
Improvements in or relating to the preparation of adhesive tape.  
Kapur, S. L. and Rao, B. R. K.
24. **70670**  
Improvements in or relating to the controlling of water evaporation for conserving water in lakes and reservoirs.  
Kulkarni (Miss), S. B., Gharpurey, M. K., Deo, A. V., Sanjana, N. R., Abraham, K.O. and Subba Rao, B.C.
25. **71063**  
Production of bacterial protease by submerged culture.  
Babbar, I.J., Powar, V. K. and Jagannathan, V.
26. **71190\***  
Preparation of anion exchange resins.  
Krishnaswamy, N., Govindan, K. P., and Dasare, B. D.
27. **72425**  
A direct process for preparing the chlorides of barium and strontium from their sulphate minerals.  
Iqbal, S. H., Lobo, J. and Gupta, J.
28. **73702**  
A process for the preparation of cyclopentadecanolide (exaltolide).  
Dhekne, V. V., Ghatge, B. B. and Bhattacharyya, S. C.

29. **74356\***  
Preparation of insoluble reaction products of polystyrene for use as cation exchange materials.  
Govindan, K. P. and Krishnaswamy, N.
30. **74451\***  
Preparation of covering materials from anacardic materials.  
Raghunath, D., Suryanarayana, N. P. and Krishnaswamy, N.
31. **77080**  
A process for the preparation of ambrettolide.  
Sabnis, S. D., Mathur, H. H. and Bhattacharyya, S. C.
32. **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.
33. **77225\***  
A process for the preparation of  $\beta$ -ionone from pseudoionone.  
Joshi, B. N., Chakravarti, K. K., Shah, R. C. and Bhattacharyya, S. C.
34. **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.
35. **83364**  
Manufacture of hexachloroethane.  
Bhat, N. A., Goswami, M. and Pai, M. U.
36. **83716\***  
Manufacture of nicotine sulphate from tobacco wastes.  
Potnis, G. V., Goswami M., Ramachandran, V. and Pai, M. U.
37. **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.
38. **86991\***  
Preparation of polyurethane printing rollers.  
Ghatge, N. D. and Kapur, S. L.

39. **90574**  
A process for the preparation of *dl*-muscone.  
Nair, M. S. R., Mathur, H. H. and Bhattacharyya, S. C.
40. **91412**  
Manufacture of 2, 3-hydroxynaphthoic acid from 2-naphthol.  
Phadtare, P. G., Srinivasan, K. R., Baliga, B. A., Kotasthane, M. G.  
and Doraiswamy, L. K.
41. **92977**  
Improvements in or relating to the manufacture of hexachloroethane.  
Mukherjee, S. P., Gowsami, M., Soundararajan, S., Sadasivan, N.,  
Sen, R. K. and Doraiswamy, L. K.
42. **96801**  
A new method for the improvements in the figure of merit of  
thermoelectric material.  
Narasimhan, K. S. V. L. and Sinha, K. P.
43. **98155\***  
An improved method for the hydration of sulphonated mixtures of  
vinyl copolymer beads containing 90-95% sulphuric acid and the sul-  
phonated vinyl copolymer beads.  
Kapur, S. L. and Ramakrishnan, K.
44. **98156\***  
Preparation of solvent modified copolymers of vinyl monomers  
in bead form.  
Kapur, S. L. and Ramakrishnan, K.
45. **106804**  
Improvements related to the manufacture of carboxy methyl cellulose.  
Bendale, D. S., Mahajan, M. B. and Khadilkar, H. P.
46. **106808**  
Improvements in or relating to the preparation of mixed oxide ceramic  
compositions.  
Murthy, M. N. S. and Sinha, A. P. B.
47. **108413**  
A process for treatment of costus roots (*Saussurea lappa* Clarke) for  
isolation of inulin.  
Kulkarni, G. H., Kelkar, G. R., Bose, J. L. and Bhattacharyya, S. C.

48. **108414**  
A process for treatment of costus roots for inulin isolated therefrom for the production of fructose.  
Kulkarni, G. H., Kelkar, G. R., Bose, J. L. and Bhattacharyya, S. C.
49. **111311**  
A process for the recovery of anhydrous pyridine bases from their aqueous solution.  
Buzruk, N. K., Paul, R. N., Potnis, G. V. and Pai, M. U.
50. **112575**  
Thermal treatment of oil with catalyst.  
Sukh Dev and Nayak, U. R.
- \* These patents (25) are being commercially exploited.

*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 oil and isolation of a coumarin constituent therefrom.  
Unni, I. R., Maheshwari, M. L., Paknikar, S. K. and Bhattacharyya, S. C.
4. **109489**  
Production of aryl-glycosides.  
Ingle, T. R. and Bose, J. L.
5. **113406**  
A new thermistor composition exhibiting high positive temperature coefficient of resistivity (posistor).  
Brahmecha, B. G. and Sinha, K. P.

6. **113703**  
Improvements in or relating to magnesium zinc ferrites.  
Krishna Rao, V. V., Kanade (Miss), S. B. and Sinha, A. P. B.
7. **113825**  
11 ORT nickel zinc ferrites.  
Krishna Rao, V. V., Kanade (Miss), S. B. and Sinha, A. P. B.
8. **116453**  
Disperse dyes for polyesters with good affinity and sublimation fastness.  
Raman, S. K. and Tilak, B. D.
9. **116675**  
A process for the preparation of polyamide polymers using diisocyanates.  
Ghatge, N. D., Patil, S. B. and Patil, V. S.
10. **117210**  
A direct process for the manufacture of dibutyltin di-iodide and of the oxide therefrom.  
Gupta, J., Gopinathan, C., Gopinathan (Mrs.), S. and Awasarkar, P. A.
11. **117403**  
Preparation of water thinnable emulsion paints from linseed oil.  
Kapur, S. L. and Bakshi, S. H.
12. **118476**  
Methods of preparing isocyanates, mono and polyureas from pentadecenyl phenol (anacardol) and their uses.  
Ghatge, N. D. and Yadav, S. D.
13. **121538**  
A process for the shellac-polyester urethane compositions for use in textile water proofing adhesives, paints and varnishes and in electrical insulating varnishes.  
Ghatge, N. D. and Srinivasan, S. R.
14. **125138**  
Method of preparing polyisocyanates and polyureas from pentadecenyl phenol (anacardol).  
Ghatge, N. D. and Mahajan, S. S.

A-XII TABLE I : PRODUCTS MANUFACTURED ON THE BASIS OF NCL KNOW-HOW

T=Metric tonnes

S. No.	Name of the process & Indian Patent Number	Field of utilization	Name of the manufacturer (year of commencement of production)	Production		Remarks
				1969-70 Qty./Value in Rs. lakhs	upto March 1969 Qty./Value in Rs. lakhs	
1	2	3	4	5	6	7
1.	Acetanilide	Intermediate	M/s Hindustan Organic Chemicals Ltd., Rasayani, through project engineers M/s R. L. Dalal & Co., Bombay-18 (1969).	247 T 15.54	—	Exclusive.
2.	Acriflavine (sponsored)	Pharmaceuticals	M/s Western India Fine Chemicals, Bombay-80 (1968).	847 kg. 3.67	—	Exclusive.
3.	Adhesives for decorative laminates (sponsored)	Laminates	M/s Swastik Rubber Products Ltd., Poona-3 (1969).	12 T 1.02	—	Exclusive.
4.	Anion exchange resin from melamine (71190)	Deminerlization of liquids	M/s Tulsi Industries, Hadapsar Industrial Estate, Poona-13 (1963).	—	565 cft. 1.75	Non-exclusive.
5.	Antipriming compositions (77081)	Antipriming in locomotives	Research, Design and Standard Organisation, M & C Wing, Chittaranjan (1964).	14 T 0.42	64.70 T 1.57	Non-exclusive.



1	2	3	4	5	6	7
6.	Bacterial diastase (66096)	Textile desizing	M/s Chemaux Pvt. Ltd., Mahim, Bombay-16 (1967).	125.50 T 3.76	85 kg. 0.86	Exclusive. Capacity 450 TPA.
7.	Benzoic acid (sponsored)	Pharmaceuticals	M/s Aniline Dyestuffs & Pharmaceuticals (P) Ltd., 28, Apollo Street, Bombay-1 (1969).	—	— 0.17	Exclusive. Trial production started.
8.	Berberine hydrochloride (sponsored)	Pharmaceuticals	M/s G. Vallabhadas & Co., 180/82, Samuel Street, Bombay-9 (1965).	—	300 kg. 0.36	Exclusive. According to the party, production being done based on foreign know-how
9.	Calcium hypophosphite (sponsored)	Pharmaceuticals	M/s Hypophosphite & Co., 79-F, Princess street, Bombay-2 (1967).	9 T 4.50	3.25 T 1.80	Exclusive.
10.	Calcium silicate (sponsored)	Low density insulators	M/s Newkem Products Corp., Harganga Mahal, Bombay-14 (1968).	210 T 7.50	112 T 3.90	Exclusive.
11.	Can sealing composition (66194)	Metal can industry	M/s Arya Chemical Works, 114/2A, Dharmatolla Street, Calcutta-13 (1962).	21 T 1.13	112 T 5.85	Exclusive.
12.	Cation exchange resin from CNSL (59497, 59606)	Demineralization of liquids	M/s Tulsi Industries, Poona-13 (1960).	—	7,310 cft. 6.14	Exclusive.

1.	2	3	4	5	6	7
13.	Chloral hydrate (sponsored)	Industrial chemicals	M/s Hindustan Insecticides Ltd., Industrial Area, New Delhi-15 (1963).	4.56 T 0.47	14.90 T 1.42	Exclusive.
14.	Coating for oil filter papers	Oil filtration	M/s White Cloud Paper Mills, 412, Gultekdi Road, Poona-9 (1968).	—	—	Non-exclusive. Technical aid. Trial production started.
15.	DDT-water dispersible (56726)	Insecticides	(i) M/s South India Research Institute (P) Ltd., Sirinagar Industrial Estate, Vijayawada-7 (1963).	—	19.84 T 1.35	Non-exclusive.
			(ii) M/s Lotus Industries, 62/20, Luz Church Road, Madras-4 (1967).	425 kg. 0.04	470 kg. 0.04	Non-exclusive.
16.	Dihydroisojasmone	Perfumery	M/s S. H. Kelkar & Co. (P) Ltd., Bombay-Agra Road, Bombay-80 (1965).	115 kg. 0.40	340 kg. 1.14	Non-exclusive. Technical aid.
17.	Ethylene oxide condensates (sponsored)	Surface active agent	M/s Hico Products (P) Ltd., Mogal Lane, Bombay-16 (1965).	421 T 41.74	689 T 77.33	Exclusive.
18.	Ferrites (hard)	Electronics	M/s Semiconductors Ltd., Nagar Road, Poona-14 (1968).	—	—	Non-exclusive. Experimental pilot plant production started.

1	2	3	4	5	6	7
19.	Hexylresorcinol (57888)	Pharmaceuticals	M/s Unichem Laboratories Ltd., Jogeshwari, Bombay-60 (1964).	—	13 kg. 0·08	Exclusive.
20.	4-Hydroxycoumarin (62890, 63083)	-do-	-do- (1964).	135 kg. 0·29	130 kg. 0·52	Exclusive.
21.	Heat sealable coating composition	Adhesives	M/s Chandmal Fakirchand Jain, Khajuri Bazar, Indore-2 (1963).	—	—	Non-exclusive. Technical aid. Reported to be in production.
22.	$\beta$ -Ionone (77225)	Perfumery, Pharmaceuticals	(i) M/s S. H. Kelkar & Co. (P) Ltd., Bombay (1964).	—	—	Non-exclusive. The firm is producing the item. Accordingly to them, they are not producing it by NCL process.
			(ii) M/s Industrial Perfumes Ltd., Army & Navy Bldg., M. G. Road, Fort, Bombay-1 (1968).	5·01 T 3·46	7·63 T 4·75	Non-exclusive. The firm has processed 5 T of $\beta$ -ionone for M/s. Roche Products Ltd., Bombay.
23.	Liquid rubber (60555)	Adhesives, Rubber rollers	M/s K. N. Chari & Co., 144, Nyncappa Naick, Street, Madras-3 (1962).	—	2·2 T	Non-exclusive. Production for captive use.

1	2	3	4	5	6	7
24.	Perfumery products based on Longifolene (Capinone) (sponsored)	Perfumery	M/s Camphor & Allied Products, Bareilly (1968).	9.1 T 7.12	4.6 T 3.28	} Exclusive. Two perfumery products are manufactured.
25.	Perfumery products based on $\Delta^3$ -Carene (Meracene) (sponsored)	-do-	-do- (1968).	18 kg. 0.06		
26.	<i>l</i> -Menthol from dementholized peppermint oil (sponsored)	Fine chemicals, Drugs	M/s Bhavana Chemicals Ltd., Laxmi Insurance Bldg., Bombay-1 (1968).	—	—	Exclusive. Experimental adoption of work in progress.
27.	Nicotine sulphate (54867, 83716)	Insecticides	M/s Urvkunj Tobacco By-products Ltd., Dharmaj (Dist. Kaira) (1963).	5.51 T 1.10	20.30 T 3.02	Exclusive on territorial basis.
28.	Peach aldehyde	Perfumery	M/s S. H. Kelkar & Co., (P) Ltd., Bombay (1965).	830 kg. 1.66	1.65 T 2.70	Non-exclusive. Technical aid.
29.	$\beta$ -Phenethyl alcohol (sponsored)	Perfumery	M/s Sunanda Aromatics Industries, D-14, Sayaji Rao Road, Mysore-1 (1969).	—	—	Exclusive. Trial production. will commence in July 1970. Estimated production 150 TPA valued at Rs. 45 lakhs.
30.	Phthalates-dioctyl and dibutyl	Plasticizers	M/s Alta Laboratories Ltd., Giri Vihar, Khopoli (Dist. Kolaba) (1969).	197 T (DOP) 14.62	990 kg. (DOP) 0.07	Non-exclusive.

1	2	3	4	5	6	7
31.	Polyurethane printing rollers (86991)	Printing	(i) M/s Saraswathy Printing Press, 32, Acharya P. C. Road, Calcutta-9 (1965).	—	—	Non-exclusive. According to the firm they are not producing by NCL process.
			(ii) M/s Dabholkar Bros. East Street, Poona-1 (1965).	—	—	Non-exclusive. Trial production
32.	Rigid filters (59608,66966)	Tube wells	M/s Ashim Filters, 196, Defence Colony, New Delhi (1965).	1,865 mtrs. 3·77	2,840 mtrs. 5·20	Exclusive.
33.	Rubber base contact adhesive (65977)	Adhesives	M/s K. N. Chari & Co., Madras-3 (1962).	600 Ltrs 0·04	16,325 Ltrs. 1·33	Non-exclusive.
34.	Rubber blowing agent	Rubber chemicals	M/s Swastik Rubber Products Ltd., Poona-3 (1968).	40 T 5·03	19 T 1·89	Non-exclusive. Technical aid.
35.	Rubberized cork sheet	Gaskets	M/s Bharat Casements (P) Ltd, P. B. No 89, Baroda (1966).	9,91,000 Nos. 2·31	6,49,600 Nos. 3·55	Non-exclusive.
36.	Rubber reclaiming agent	Rubber chemicals	M/s Swastik Rubber Products Ltd, Poona-3 (1968).	7·26 T 0·94	1·67 T 0·15	Non-exclusive.
37.	Silica gel (sponsored)	Humidity control	M/s Minco Products, 301/27 T. H. Road, Madras-21 (1963).	9 T 0·77	53 T 3·21	Exclusive.

1	2	3	4	5	6	7
38.	Sisal wax (64958, 65440)	Cosmetics, Polishes	M/s Aphali Pharmaceuticals Ltd., Station Road, Ahmednagar (1966).	1·10 0·11	14·20 T 1·53	Non-exclusive.
39.	Sorbide nitrate (sponsored)	Pharmaceuticals	M/s Indian Schering Ltd., Mercantile Chambers, Ballard Estate, Bombay-1 (1969).	40 kg. 1·20	90 kg. 2·70	Exclusive.
40.	Styrene DVB-base cation exchange resin (65976, 98155, 98156)	Deminerlization of liquids	M/s Industrial & Agricul- tural Engg. Co. (P) Ltd., 43, Forbes Street, Bombay-1 (1967).	—	—	Non-exclusive. Trial production started.
41.	Thermistors	Electronic devices	(i) M/s Semiconductors Ltd., Poona-14 (1963).  (ii) M/s Tempo Semiconductors Vile Parle (E), Bombay-57 (1963).	4,22,700 Nos. 3·16  8,000 Nos. 0·32	21,40,000 Nos. 19·58  17,000 Nos. 0·69	Non-exclusive. Technical aid.  -do-
42.	Titanium tetrachloride (sponsored)	Industrial chemicals	M/s Travancore Titanium Products Ltd., Trivandrum-7 (1967).	370 kg. 0·03	450 kg. 0·03	Exclusive. Trial production started.
43.	Vapour phase chromatograph	Instruments	M/s Associate Instruments Manufacturers (India) P. Ltd., 26-27, Asaf Ali Road, New Delhi (1969).	12 units 2·40	5 units 0·95	Exclusive. Value is excluding the cost of imported components.
44.	Warfarin	Rodenticides	M/s Unichem Laboratories, Bombay (1968).	—	67 kg. 0·40	Exclusive.

*Table—I Continued.*

*Production and value of some major chemicals produced in Fine  
Chemicals Project :*

	<i>Qty.</i>	<i>Value in Rs.</i>
45. Alumina	160 kgs.	13,850/—
46. Azo-bis-iso-butyronitrile	102 kgs.	25,000/—
47. Costus root oil	12 kgs.	12,000/—
48. Di- <i>o</i> -tolylbiguanide	340 kgs.	9,250/—
49. 8-Hydroxyquinoline	470 gms.	5,640/—
50. Ethyl-iso-thiocyanate	6 kgs.	9,000/—
51. Methyl-iso-thiocyanate	5 kgs.	7,500/—
52. Phenyl-iso-thiocyanate	10 kgs.	5,000/—
53. Silica gel	1043 kgs.	76,840/—

## SUMMARY OF TABLE I

(Rs. in lakhs)

Year	No. of items manufactured (inclusive of major FCP items)	Value of production	*Foreign exchange saving	**Cumulative value of production
1967—68	29	29·98	15·00	92·71
1968—69	47	69·55	24·50	162·26
1969—70	53	130·22	48·80	292·48

\* Majority of the the items under production have led to import substitution. For such items 40% of the indigenous production is assumed as foreign exchange saving.

In addition, perfumery products based on Longifolene and Carene have been exported worth Rs. 7·18 lakhs during 1969-70.

\*\* Until the last Annual Report, cumulative value of production was given for 31st March of the previous year. From this Annual Report these values refer to 31st March of the year of the publication of the Report.



**A-XIII TABLE II (A) : PROCESSES RELEASED AND AWAITING PRODUCTION**

S. No.	Name of the process and Indian Patent No.	Field of utilization	Name of the party & year of release	Nature of licence/present state of development
1	2	3	4	5
1.	Aniline	Industrial chemicals	M/s Hindustan Organic Chemicals Ltd., Rasayani, through project engineers M/s Engineers India Ltd., New Delhi (1969).	Exclusive agreement for engineering the project. Proposal for turn-key plant submitted to HOC.
2.	BD-catalyst (sponsored)	Catalyst for synthetic rubber	M/s Synthetic and Chemicals Ltd., 284, Frere Road, Bombay-1 (1969).	Exclusive. Test trials in progress.
3.	Cadmium sulphide photo-conductive cells	Electronics	M/s Gera Engineering Co. 'Pritam Villa', 8/352, Boat Club Road, Poona-1 (1969).	Non-exclusive.
4.	Cation exchange resin polystyrene base (74356)	Demineralization of liquids	M/s Tulsi Industries, Poona-3 (1965).	Non-exclusive.
5.	Chlorinated copper phthalocyanine (sponsored)	Dye intermediate	M/s Sudarshan Chemical Industries (P.) Ltd., 162, Wellesley Road, Poona-1(1968).	Exclusive. Pilot plant trials in progress.
6.	Chlorination of methane	Industrial chemicals	M/s Standard Mills Co. Ltd., P. B. No. 1038, Bombay-20 (1969).	Agreement in progress. Released for further pilot plant work.

1	2	3	4	5
7.	Chlorobenzenes (sponsored)	Industrial chemicals	M/s Hindustan Organic Chemicals Ltd., Rasayani (1969).	Exclusive.
8.	Covering materials from CNSL (74451)	Chemical resistant flooring	M/s Natson Manufacturing Co., Bhadra, Ahmedabad (1964).	Exclusive.
9.	Diethyl- <i>m</i> -aminophenol (sponsored)	Dye intermediate	M/s Sahyadri Dyestuffs and Chemicals (P.) Ltd., 117, Vithalwadi Road, Poona-9 (1966).	Exclusive. Demonstration again given recently to the new proprietors.
10.	Di- <i>o</i> -tolylbiguanide	Soap industry	M/s Industrial Perfumes Ltd., Bombay-1 (1969).	Exclusive. Licence for 5 TPA awaited.
11.	Dithranol	Pharmaceuticals	M/s Indian Dyestuffs Industries Ltd., Kalyan (1968).	Exclusive.
12.	8-Hydroxyquinoline (sponsored)	Pharmaceuticals	M/s Alta Laboratories Ltd., Khopoli (1970).	Exclusive.
13.	Isolation of borneol (sponsored)	Perfumery	M/s Camphor & Allied Products, Bareilly (1969).	Exclusive.
14.	Liquid stabilizer for PVC (sponsored)	Plastics	M/s Swastik Rubber Products Ltd., Poona-3 (1969).	Exclusive.

1	2	3	4	5
15.	Megimide (sponsored)	Pharmaceuticals	M/s Indian Schering Ltd., Bombay-1 (1969).	Exclusive.
16.	Nonylphenol (sponsored)	Surface active agents	M/s Surfactants (P.) Ltd., 28, Apollo Street, Bombay-1 (1969).	Exclusive.
17.	Opium alkaloids	Pharmaceuticals	Govt. of India, Ministry of Finance, New Delhi (1966).	Exclusive. Capacity planned 4.7 TPA value Rs. 1 crore. Detailed designs of civil, mechanical and electrical works completed by the NIDC, New Delhi.
18.	<i>p</i> -Menthane hydroperoxide	Synthetic rubber	M/s Camphor & Allied Products Ltd., Bareilly (1969).	Exclusive. Capacity 40 TPA.
19.	Pentachlorophenol and other chlorophenolic compounds (sponsored)	Fungicide	M/s Bombay Chemicals Pvt. Ltd., Bombay-1 (1969).	Exclusive.
20.	Perfumery grade geraniol, citronellal and citronellol (sponsored)	Perfumery	M/s Radhakishan Rajaram & Co., 143, Princess Street, Bombay-2 (1968).	Exclusive.
21.	Phthalates—diethyl and dimethyl	Plasticizers	M/s Mysore Acetate & Chemi- cals Co. Ltd., Sri J. W. Road, Bangalore-2 (1968).	Non-exclusive. Trial runs in progress at the works.

1	2	3	4	5
22.	Potentiometric strip chart recorder	Recording instruments	M/s Associated Instruments Manufacturers (India) Pvt. Ltd., New Delhi (1970).	Non-exclusive.
23.	Radio opaque dyes (sponsored)	Pharmaceuticals	M/s Unichem Laboratories, Bombay (1968).	Exclusive.
24.	Sulphacetamide and its sodium salt (sponsored)	Pharmaceuticals	M/s Indian Schering Ltd., Bombay-1 (1969).	Exclusive.
25.	Terpene G (sponsored)	Perfumery, Insecticides	M/s Camphor & Allied Products, Bareilly (1968).	Exclusive.
26.	Vat Golden Yellow G. K. (sponsored)	Dye intermediate	M/s Amar Dye-Chem Ltd., P. B. 6471, Bombay-16 (1967).	Exclusive.
27.	Vitamin C	Pharmaceuticals	Hindustan Antibiotics Ltd., Pimpri, Poona (1966).	Exclusive. Capacity 125 TPA. Civil construction of the plant building in progress.

TABLE II (B)

*Following processes which have been mentioned in Table I are also licensed on non exclusive basis to the following additional firms and are awaiting production :*

<i>Name of the process</i>	<i>Name of the party &amp; year of release</i>
1. DDT-water dispersible	M/s Pesticides Ltd., 28 Sayani Road, Bombay-28 (1967).
2. Liquid rubber	M/s Swastik Rubber Products Ltd., Poona-3 (1962).
3. Nicotine sulphate	(i) M/s. M. M. Patel, P. O. Sunav, Gujarat (1966). (ii) M/s. National Tobacco Bye-Products, Dal Mandi, Saharanpur (1963). (iii) M/s. Tobacco By-Products P. Ltd. Fort Narsarawpet, Guntur (1963).
4. Phthalates—dioctyl and dibutyl	M/s India Carbon Ltd., Shiv Sagar Estate, Dr. A. B. Road., Bombay-18, through M/s R. L. Dalal & Co., Bombay (1969). (Plant for 3,500 T/y is under construction).
5. Polyurethane printing rollers	M/s United Ink & Varnish Co., P. B. 6862, Vile Parle, Bombay-57 (1965).
6. Sisal wax	(i) M/s Shalimar Rope Works, 14, Netaji Subhash Road, Calcutta (1966). (ii) M/s Deccan Paint & Chemical Industries, 298, G. T. Toad, North Calcutta (1968). (iii) M/s Industrial & Engg. Corp. 84-B, Bondel Road, Calcutta (1966).
7. Styrene DVB-cation exchange resin	(i) M/s Tulsi Industries, Poona-13 (1963). (ii) M/s Bird & Co. Ltd., Chartered Bank Bldg. Calcutta-1 (1963).
8. Thermistors	M/s Adept Laboratories, Poona-4 (1963).

TABLE II (C) : SPONSORED PROCESSES RELEASED WHEREIN COMMERCIAL PRODUCTION HAS NOT BEEN ESTABLISHED SO FAR

S. No.	Name of the process	Name of the party & year of release	Remarks
1	2	3	4
1.	Alizarin	M/s Aniline Dyestuffs & Pharmaceuticals Ltd., Bombay-1 (1968).	Market not attractive.
2.	Aminotriazole	M/s Agromore Co. Ltd., Bangalore-26 (1967).	Party not interested unless amino- guanidine bicarbonate becomes available indigenously.
3.	Anthraquinonoid dyes	M/s Indian Dyestuffs Industries Ltd., Kalyan (1968).	This was only an analytical problem; no production was envisaged.
4.	Chemicals from castor oil	Indian Central Oilseeds Committee, Hyderabad (1962).	No production is envisaged.
5.	Corrosion studies	Department of Atomic Energy, GOI., Bombay (1965).	No production is envisaged.
6.	Constituents of costus root oil	Director of Industries, Govt. of Punjab, Chandigarh (1967).	Process uneconomic due to uncer- tainty of market.
7.	Trial cultivation of essential oil bearing plants and extraction therefrom	M/s Tata Oil Mills, Bombay (1964).	No production is envisaged now.
8.	Fermentation problem	M/s Aphali Pharmaceutical Ltd., Ahmednagar (1966).	Project successfully concluded; no production is envisaged.

1	2	3	4
9.	Indian silk—physico chemical studies	Central Silk Board, Bombay (1962).	Basic research; no production is envisaged.
10.	Isolation of emetine from ipecac roots	M/s Aphali Pharmaceuticals Ltd., Ahmednagar (1968).	Party apparently not interested in production of this item.
11.	Industrial chemicals from diketene	M/s Aniline Dyestuffs and Pharmaceuticals Ltd., Bombay-1 (1968).	Party is not interested now, as a plant has been established by another firm.
12.	Lac dye	Indian Lac Cess Committee, Ranchi (1968).	Basic research; production not envisaged.
13.	Studies in pine oil	M/s Prabhat General Agency, Bombay-2 (1966).	No production is envisaged.
14.	Pulping of Kerala hard-woods	Preinvestment Survey of Forest & Resources, New Delhi (1969).	Applied basic research study of natural resources.
15.	Rayon grade pulp	M/s Century Rayons, Kalyan (1967).	No follow-up action by party or NCL.
16.	Reactive dyes	M/s Amar Dye-Chem Ltd., Bombay (1964).	Work concluded; party already producing dyes based on its know-how.
17.	Rubber research	Indian Rubber Manufacturers Research Association, Bombay (1964).	No production is envisaged.

1	2	3	4
18.	Screening of NCL compounds for their pharmacological activity	Bristol Laboratoreis, Syracuse, New York (1970).	No production is envisaged; basic exploratory work.
19.	Sodium cyclamate	M/s Aphali Pharmaceuticals Ltd., Ahmednagar (1968).	As this item is now considered dangerous for human consumption; production is not envisaged.
20.	Steroids from sugarcane wax	M/s Brihan Maharashtra Sugar Syndicate Ltd., Poona-2 (1968).	—
21.	Tetrabromoindigo	M/s Shree Dyes and Chemicals, Poona-4 (1961).	Party is not interested due to changed market conditions.
22.	Thiodiglycol	M/s Hico Products Pvt. Ltd., Bombay (1961).	Party apparently not interested in producing the item so far.
23.	Tung oil	U. S. Department of Agriculture, Washinton, D. C. (1968), (Under PL-480 scheme).	Basic research; production not envisaged.
24.	Vanillin and ethyl vanillin	M/s Aniline Dyestuffs and Pharmaceuticals Ltd., Bombay-1 (1968).	Process not economic; production not envisaged.
25.	Water evaporation control	U. S. Department of Agriculture, Washington, D. C. (1968), (Under PL-480 scheme).	Basic research; production not envisaged.

For a variety of reasons and in some cases due to the nature and objectives of the above sponsored projects, no production has been established so far and it does appear that no production is also likely to materialize in the foreseeable future. Reference to these projects will be omitted in future Annual Reports, unless progress is made.



A-XIV TABLE III : PROCESSES APPROVED FOR RELEASE TO INDUSTRY BY 'PROCESS RELEASE COMMITTEE, NCL'

(T = Metric tonnes)

S.No.	Name of the process and Indian Patent No.	Field of utilization	Scale of development	Production envisaged per year	Ex-factory selling price Rs. /unit	Expected annual turnover Rs. in lakhs	Remarks
1	2	3	4	5	6	7	8
1.	Aniline	Industrial chemicals	5 kg./hr.	600 T	3,090	18.54	Exclusive to the project engineer; being released.
2.	Atrazine	Herbicides	Lab. scale	50 T	35,000	1.75	—
3.	Butyl titanate	Varnishes & enamels	10 kg./batch	20 T	22,000	4.40	Being released.
4.	Cadmium sulphide photo-conductive cells	Electronic instruments	Lab. scale	30,000 cells	25	7.50	Released; awaiting production.
5.	Catechol	Pharmaceuticals	Lab. scale	100 T	12,700	12.70	—
6.	Chlorobenzenes						
	(i) MCB	Industrial chemicals	0.25 T/day	3,500 T	2,900	101.50	
	(ii) ODCB			300 T	3,320	9.96	—
	iii) PDCB			600 T	4.330	25.98	

1	2	3	4	5	6	7	8
7.	Chloromethanes						
(i)	Carbon tetrachloride	Industrial	2.5 kg./hr.	228 T	3,400	7.75	Released for further pilot plant work at the works.
(ii)	Perchloroethylene	solvents		30 T	3,500	1.05	
(iii)	Chloroform			15 T	5,000	0.75	
8.	Coating for oil filter paper	Paper	6 kg./batch	1,700 kg.	16	0.27	Released; under trial production.
9.	Costus root oil	Perfumery	1 kg./batch	300 kg.	600	1.80	Giving credit for fructose.
10.	Dimethylaniline	Industrial chemicals	9 kg./batch	600 T	6,150	36.90	Negotiations in progress.
11.	Disperse dyes (116453)	Dyes	Lab. scale	—	—	—	Negotiations in progress.
12.	Ferric oxide (calcined & precipitated)	Catalyst	Lab. scale	1,100 kg.	40	0.44	—
13.	Double boiled linseed oil substitute	Foundry	100 kg./batch	1,200 T	1,750	21.00	—
14.	Fructose and chamazulene (108413, 108414)	Pharmaceuticals, Cosmetics	Lab. scale	3 T	60,000	1.80	—

1	2	3	4	5	6	7	8
15.	Gaskets from coir pith	Gaskets	18" × 18" × 1/8"	5·32 T	21,250	1·13	—
16.	<i>p</i> -Menthane hydroperoxide	Synthetic rubber	5 kg./batch	70 T	13,000	9·10	Exclusive. Released; awaiting production.
17.	Monochloroacetic acid	Weedicides, CMC	40 kg./batch	600 T	4,650	27·90	Negotiations in progress.
18.	<i>p</i> -Nitrophenol	Insecticides	40 kg./batch	300 T	11,000	33·00	Negotiations in progress.
19.	Optical whitening agent for synthetic fibres	Textiles	Lab. scale	5 T	1,08,000	5·40	—
20.	Phenacetin	Pharmaceuticals	50 kg./batch	100 T	35,000	35·00	Negotiations in progress.
21.	Phenoxyacetic acid	Pharmaceuticals	50 kg./batch	20 T	18,500	3·70	Negotiations in progress.
22.	Phthalates (i) Dioctyl (ii) Dibutyl	Plasticizers	60 kg./batch 25 „	1,500 T 3,500 T	8,000 8,000	120·00 280·00	In production. —
23.	Polyurethane coatings	Protective coatings	Lab. scale	10,000 ltrs.	15	1·50	Negotiations in progress.

1	2	3	4	5	6	7	8
24.	Potassium ethyl/amyl xanthate	Mineral beneficiation	Lab. scale	300 T	7,600	22·80	—
25.	Potentiometric strip chart recorder	Recording instruments	—	1,200 units	7,500	90·00	Released; awaiting production.
26.	Recovery of pyridine bases (113111)	Dyestuffs	Lab. scale	—	—	—	—
27.	Rubber blowing agent	Rubber chemicals	10 kg./batch	30 T	13,500	4·05	In production.
28.	Rubber reclaiming agent	„	20 kg./batch	100 T	8,400	8·40	In production.
29.	Rubberised cork sheets	Gaskets	6"×9"×1/8"	5·32 T	21,500	1·14	In production.
30.	Hot and cold sachets	Domestic heating & cooling	20×10 cm.	1,00,000 sachets	1·37	1·37	—
31.	Sealants for canvas bags	Adhesives	5 kg./batch	500 kg.	40	0·20	—
32.	Simazine	Herbicides	Lab. scale	50 T	35,000	17·50	Negotiations in progress.
33.	Di- <i>o</i> -tolybiguanide	Soap	50 kg./batch	5 T	25,000	1·25	Exclusive. Released; awaiting production.

1	2	2	3	4	5	6	7	8
34.	Staple pins adhesive		Stationery	10 kg./batch	5 T	20,000	1·00	—
35.	eTrpineol		Perfumery	Lab. scale	100 T	13,300	13·30	—
36.	<i>p</i> -tert-Butyl catechol		Synthetic rubber	10 kg./batch	25 T	32,200	8·05	Negotiations in progress.
37.	(i) Theophylline & Aminophylline (ii) Caffeine		Pharmaceuticals	Lab. scale	30 T	85,000	25·40	—
					30 T	55,000	16·50	—
38.	Triethyl phosphate		Catalysts	Lab. scale	60 T	16,500	9·90	Standardization is still in progress.
39.	tris-Nonylphenyl phosphite		Synthetic rubber	150 kg./ batch	90 T	8,850	7·97	—

**A-XV TABLE IV : KNOW-HOW AVAILABLE**

(A) Know-how is available on various products/processes which are listed under (A-XIV, page 133); These 39 processes are authorized for release by the Process Release Committee.

(B) In addition, know-how is also available on the following products/processes.

S. No.	Product/Process	Field of utilization	Scale of development	Remarks
1	2	3	4	5
1.	Anion exchange resin from melamine	Demineralization of liquids	Lab. scale	Non-exclusive; already in production.
2.	Antipriming composition	Antipriming in locomotives	Lab. scale	Non-exclusive; already in production.
3.	BON acid	Dyestuff	3 kg./batch	Indigenous capacity established.
4.	Civetone & dihydrocivetone	Perfumery	Lab. scale	Restricted market.
5.	DDT-water dispersible	Insecticides	15 kg./batch.	Non-exclusive; licensed to 3 parties; one in production.
6.	Dihydroambrettolide & Isoambrettolide	Perfumery	Lab. scale	Restricted market.
7.	Dihydroisojasmone	Perfumery	Lab. scale	Non-exclusive; one firm already in production.
8.	Dinitromonomethylaniline	Explosives	50 kg./batch	Restricted market.

1	2	3	4	5
9.	<i>d</i> -Menthol from citronellal	Perfumery	Lab. scale	Restricted market.
10.	Ethylene dichloride	Industrial solvent	3 kg./hour	Indigenous capacity established.
11.	Exaltolide	Perfumery	Lab. scale	Restricted market.
12.	Exaltone	Perfumery	Lab. scale	Restricted market.
13.	Ferrites (Hard)	Electronics	Lab. scale	Non-exclusive; licensed to one firm; trial production started.
14.	Ferrites (Soft)	Electronics	Lab. scale	Negotiations in progress.
15.	Heat sealable coating composition	Adhesives	Lab. scale	Non-exclusive; licensed to one firm; believed to be in production.
16.	Hexachloroethane	Smoke screens, Foundry industry	10 kg./hour	Negotiations in progress.
17.	$\beta$ -Ionone	Perfumery	5 kg./batch	Non-exclusive; licensed to two firms; one in production.
18.	Liquid rubber	Adhesives, Rubber rollers	Lab. scale	Non-exclusive; licensed to two firms; one in production.
19.	Modified sugarcane wax	Polishing compositions	5 kg./batch	Restricted market
20.	Monoethylaniline	Explosives	10 kg./hour	Negotiations in progress.

1	2	3	4	5
21.	Neolavandulol	Perfumery	Lab. scale	Restricted market.
22.	Nicotine sulphate	Insecticides	25 kg. of raw material/batch	Non-exclusive on territorial basis; one firm in production.
23.	Peach aldehyde	Perfumery	Lab. scale	Non-exclusive; licensed to one firm; in production.
24.	Phthalates ( i ) diethyl ( ii ) dimethyl	Plasticizers	22 kg./batch 30 kg./batch	Non-exclusive; licensed to one firm.
25.	Polyurethane printing rollers	Printing	Lab. scale	Non-exclusive; licensed to three firms; one in experimental production.
26.	Rubber base contact adhesive	Adhesives	Lab. scale	Non-exclusive; licensed to 7 firms; one in production.
27.	Sisal wax	Cosmetics, Polishes	3 kg./batch	Non-exclusive; licensed to 5 parties; one in production.
28.	Sorbitol	Pharmaceuticals	10 kg./batch	Negotiations in progress.
29.	Styrene DVB anion exchange resin	Demineralization of liquids	15 kg./batch	Indigenous capacity established.
30.	Thermistors	Electronic devices	100 pieces/batch	Non-exclusive; released to three parties as technical aid; two in production.



A-XVI STAFF STRENGTH (1969-70)

1. Scientific	..	273
2. Technical	..	207
3. Non-technical	..	100
4. Class IV technical	..	142
5. Class IV non-technical	..	68
	<b>Total ..</b>	<u>790</u>
6. Research workers		
(a) JRF and SRF	..	36
(b) CSIR Pool Officers	..	8
(c) Guest workers	..	20
	<b>Total ..</b>	<u>64</u>
7. Scientific staff working on sponsored projects		
(a) NCL staff	..	16
(b) Staff from sponsors	..	28
	<b>Total ..</b>	<u>44</u>

## A-XVII COST-BENEFIT ANALYSIS FOR 1969-70

### ACTUAL EXPENDITURE

	Rs. in lakhs
1. <i>Recurring expenditure</i>	
(a) Pay and allowances	.. 37·362
(b) Chemicals and apparatus	.. 10·523
(c) Maintenance and contingencies	.. 7·096
Total	.. <u>54·981</u>
2. <i>Capital expenditure</i>	
(a) Building	.. 0·136
(b) Equipment and apparatus	.. 2·385
(c) Services and miscellaneous	.. 1·636
Total	.. <u>4·157</u>
3. <i>Pilot plant expenditure</i>	
(a) Pay and allowances	.. 2·044
(b) Raw materials and miscellaneous	.. 1·269
(c) Equipment, buildings and services	.. 1·949
Total	.. <u>5·262</u>
4. Expenditure on account of sponsored projects	.. 7·170

### BENEFITS

#### *Direct benefits*

1. Premia and royalties*	.. 1·81
2. Overheads receipts on account of sponsored projects	.. 2·86
3. Analytical & testing charges, technical aid etc.	.. 0·27

4. Institutional consultancy (CSIR share)	..	0·80
5. FCP (Fine Chemicals Project) sales	..	2·71
6. Miscellaneous receipts	..	1·96
	Total ..	<u>10·41</u>

\*Fig. for royalty relates to calendar year 1969 only.

*Indirect benefits*

1. Total no. of processes in production		53
2. Total value of production (Rs. in lakhs)		130·22
3. Estimated saving in foreign exchange on account of above production (Rs. in lakhs)		48·80
4. Total cumulative value of production upto March 1970 (Rs. in lakhs)		292·48
5. Research papers published during 1969-70		97
6. Doctorate and Masters degrees awarded during 1969-70		23
7. Patents		
(a) New patents filed		2
(b) Total patents in force		50
(c) Patents which are being commercially exploited		25
8. Total no. of technical enquiries on which information was supplied		1955

A-XVIII N. C. L. EXECUTIVE COUNCIL MEMBERS

1. Shri Arvind N. Mafatlal (Chairman)  
Mafatlal House, Bombay-1.
2. Prof. B. K. Bachhawat, Prof. of Biochemistry,  
Christian Medical College, Vellore.
3. Dr. H. E. Eduljee, Director,  
National Peroxide Ltd., Bombay-1.
4. Dr. T. R. Govindachari, Director,  
CIBA Research Centre, Bombay-63.
5. Shri. L. Kumar, Adviser (Petrochemicals),  
Ministry of Petroleum & Chemicals, New Delhi.
6. Prof. R. P. Mitra, Head of the Chemistry Dept.,  
Delhi University, Delhi.
7. Dr. W. D. Patwardhan, Director,  
ERDL, Poona-21.
8. Dr. C. N. Rao, Prof. of Chemistry,  
I. I. T., Kanpur.
9. Shri. Darbari S. Seth, Director,  
Tata Chemicals, Ltd., Bombay-1.
10. Shri. D. M. Trivedi, Project Manager,  
Synthetics & Chemicals Ltd., Bombay-20.
11. Director General, Scientific and Industrial Research
12. Financial Adviser, CSIR
13. Director, NCL

## A-XIX OUR ADVISORS

### (A) PROCESS RELEASE COMMITTEE MEMBERS

1. Shri. Arvind N. Mafatlal,  
Mafatlal House, Bombay-1.
2. Dr. W. D. Patwardhan,  
ERDL, Poona-21.
3. Shri. V. G. Rajadhyaksha, Chairman,  
Hindustan Lever Ltd., Bombay-1.
4. Shri. L. Kumar,  
Ministry of Petroleum & Chemicals, New Delhi.
5. Shri. G. B. Newalkar, Chairman,  
Maharashtra Small Scale Industries  
Development Corporation Ltd., Bombay-1.
6. Shri. Darbari S. Seth,  
Tata Chemicals Ltd., Bombay-1.
7. Shri. D. M. Trivedi,  
Synthetics & Chemicals Ltd., Bombay-20.
8. Dr. H. E. Eduljee,  
National Peroxide Ltd., Bombay-1.

### (B) ADVISORY PANEL MEMBERS

- I. *Solid State Chemistry Group*
1. Dr. B. H. Wadia, Managing Director,  
M/s. Semiconductors Ltd., Poona-14.
2. Dr. B. R. Marathe, Scientist,  
Central Electronics Engineering Res. Institute, Pilani.
3. Shri. K. R. Savoor, Manager,  
Bharat Electronics Ltd., Bangalore-13.
4. Dr. A. R. Verma, Director,  
National Physical Laboratory, New Delhi-12.
5. Dr. S. C. Jain, Director,  
Solid State Physics Laboratory, Delhi-7.

## II. *Inorganic Chemistry Group*

1. Shri. J. D. Adhia, Managing Director,  
Hindustan Zinc Ltd., Udaipur.
2. Prof. V. A. Altekar, Director,  
National Metallurgical Laboratory, Jamshedpur-7.
3. Shri. K. S. Hinge, Chief Executive Officer (Development),  
Tata Chemicals Ltd., Bombay-1.
4. Shri. B. K. Kamat,  
Dharmsi Morarji Chem. Co., Ltd.,  
Ambarnath, (Thana).
5. Shri. V. N. Kasturirangan, Chief Project Officer,  
Min. of Petroleum & Chemicals, New Delhi.
6. Shri. P. K. Seshan, Industrial Adviser (Chem.),  
DGTD, New Delhi.
7. Shri. Sarat K. Vakil, Vice President (Works),  
Saurashtra Chemicals, Porbandar.

## III. *Organic Synthesis and Natural Products Group*

1. Dr. L. K. Behl, General Manager,  
Indian Drugs & Pharmaceuticals Ltd., Hyderabad-37.
2. Dr. T. R. Govindachari,  
CIBA Research Centre, Bombay-63.
3. Prof. A. B. Kulkarni, Dept. of Chem.,  
Bombay University, Bombay-19.
4. Prof. T. R. Seshadri, FRS,  
University of Delhi, Delhi-7.
5. Prof. S. V. Sunthakar, Prof. of Dye Tech.,  
Bombay Univeristy, Bombay-19.
6. Shri. S. V. Desai,  
Amar Dye-Chem Ltd., Bombay-16.
7. Shri. Joginder Singh, Industrial Adviser (Chem.),  
DGTD, New Delhi-1.

8. Dr. R. J. Rathi, Managing Director,  
Sudarshan Chemical Industries (P) Ltd., Poona-1.
9. Shri. B. V. Shah,  
Atul Products Ltd., Atul (Dist. Bulsar).

#### IV *Polymer Group*

1. Dr. S. P. Bhattacharyya, Development Officer (P. C.),  
DGTD, New Delhi.
2. Dr. S. K. Datta,  
NOCIL, Bombay-8.
3. Shri. V. V. Ketkar,  
Indian Plastics Ltd., Bombay-67.
4. Shri. L. Kumar,  
Min. of Petroleum & Chemicals, New Delhi.
5. Dr. K. Narayan Rao, Chemistry Dept.,  
Bhabha Atomic Research Centre, Bombay-85.
6. Shri. T. G. Punawani, Technical Director,  
Bright Brothers Pvt. Ltd., Bombay-78.
7. Shri. Raman M. Patel, General Manager,  
Bhor Industries Ltd., Bombay-1.
8. Shri. V. S. Vaidya, Jt. Managing Director,  
Swastik Rubber Products Ltd., Poona-3.
9. Shri D. M. Trivedi,  
Synthetics & Chemicals Ltd., Bombay-20.

#### V. *Chemical Engineering & Process Development Group*

1. Dr. C. J. Dadachanji, Administrative Managing Director,  
NOCIL, Bombay-18.
2. Dr. H.E. Eduljee,  
National Peroxide Ltd., Bombay-1.
3. Shri. S. J. Shah, Managing Director,  
Aniline Dyestuffs & Pharmaceuticals Pvt. Ltd., Bombay-1.
4. Dr. W. D. Patwardhan,  
ERDL, Poona-21.

5. Shri. V. K. Dixit, Chief Chem. Eng.,  
Indian Dyestuffs Industries Ltd., Kalyan.
6. Prof. N. R. Kamat, Head of the Dept. of Chem. Eng.,  
I. I. T., Bombay-76.
7. Dr. A. Sitaramaiah, Industrial Adviser (Chem.),  
DGTD, New Delhi-1.
8. Prof. M. M. Sharma, Prof. of Chem. Eng.,  
Bombay University, Bombay-19.

VI. *Polysaccharides Project Group*

1. Dr. D. C. Tapadar, Principal,  
Institute of Paper Technology, Saharanpur.
2. Dr. S. R. D. Guha, Officer-in-Charge,  
Forest Research Institute, Dehra Dun.
3. Shri. M. S. Parkhe, Managing Director,  
Paper & Pulp Conversions Ltd., Poona-5.
4. Shri. N. Narsimhan, Development Officer (Paper),  
DGTD, New Delhi.
5. Shri. V. G. Navelkar, Chief Chemist,  
Shri Ram Rayons, Kota.

DISTRIBUTION OF OUR ADVISORS

<i>Members from :</i>	<i>No.</i>
1. Industry	22
2. Govt. Departments including DGTD experts	8
3. Public sector	3
4. Sister laboratories	3
5. Research institutes, universities, etc.	9
6. Defence research laboratories, Bhabha Atomic Research Centre etc.,	2
Total	47



**NATIONAL CHEMICAL LABORATORY**  
**REVIEW OF PROGRESS**  
**1950-70**

## OBJECTIVES, RESEARCH MANAGEMENT POLICIES AND THEIR EXECUTION

In January 1970, the NCL completed its twenty years of service to the cause of development of science and technology. The objectives set out for the Laboratory at the time of its inauguration were "*the advancement of knowledge and the application of chemical science for the good of the people*". The Laboratory was also expected to play a significant role in the development of chemical industry, which in 1950 was still in its infancy.

Two decades are not really long enough in the life of a research institution to provide data for a judgement on its performance. Nevertheless, it may be useful to review the progress of the Laboratory during the last 20 years. Whereas the initial years were naturally spent in building up the organizational structure by collecting and training research/technological expertise and establishment of adequate research and developmental facilities, there has more recently been considerable intensification of efforts in research and development work related to the chemical industries which have been rapidly developing during the last 10-15 years.

While increasing attention is being devoted to applied research, the Laboratory has also maintained an excellent supporting scientific infra-structure, as it believes that for effecting real breakthroughs in technology a sound base of fundamental research and a high order of scientific capability and adaptability for innovation are necessary. Thus the NCL has earned a reputation of being one of the leading centres of research in chemistry in the country. It has also gained international recognition by its contributions to basic research.

A review of the work of NCL for 1950-60 was published earlier and this was then followed, after a gap of 5 years, by annual publication of progress reports since 1965-66. The present report is the fifth Annual Report which is being published.

Whereas for details of work done a reference will have to be made to the above reports, in the present twenty year review of the progress of the NCL an attempt has been made to measure the results of research in relation to investment. We are fully conscious that several large areas of scientific activity cannot be evaluated in quantitative terms and the problem of measuring scientific, techno-economic and social benefits of scientific research is still

an unsolved problem the world over. Nevertheless, for an institution whose objectives are not only research but also development of economy, it seems desirable to make an attempt to measure (however imperfectly) its direct and indirect benefits to society and discuss these in relation to scientific/ technological/administrative inputs and their cost. The present review is one such attempt, our first in this direction.

Considerable attention has been recently devoted to research management in order to ensure that the total efforts of the Laboratory and its administrative policies are integrated and directed towards reaching the objectives and goals of the Laboratory as economically and speedily as possible.

Some of the important steps which were taken as a part of the overall administrative and research strategy to coordinate the work of the NCL with the short term and long term needs of the industry are briefly enumerated below:

1. Six advisory panels were constituted in 1967-68 for different disciplines, areas of work and important projects. The panels assist the NCL in identification of problems of industrial importance and give advice on the formulation of research policies and programmes.

Presently there are 47 members on the panels drawn from the following : private industry and trade associations, 22; Govt. officials, and advisers including those from DGTD, 8; public sector undertakings, 3; Atomic Energy and Defence establishments, 2; sister CSIR laboratories, 3; and 9 from universities, I. I. T.'s and other similar institutions.

As a result of the industrial liaison through these Advisory Panels the research programmes of the laboratory have assumed greater relevance to the needs of the industry.

2. Institutional consultancy has been encouraged. These have helped NCL scientists to develop live contacts with industry and become increasingly appreciative of industry's needs and problems. Such contacts have also led to greater participation by industry in the NCL work through sponsored research projects.
3. The Division of Technical Services was activated and entrusted with the work of cost/benefit analysis of major research groups and projects, PERT-programming of urgent and more important research projects, preparation of techno-economic reports on completed projects in a form needed by entrepreneurs to enable them to take investment policy decisions for commercializing the projects.

4. Studies undertaken under item 3 led to reorientation of research groups and divisions, and remedial measures to correct imbalances in research inputs. As a result, the efforts of the laboratory assumed greater relevance to what was needed by industry.
5. Due recognition was given to contributions to and training in industrial research as well as plant experience during recruitments and promotions.
6. The setting up of time targets for all product/process development and research projects introduced a sense of urgency which speeded up research activity.
7. Chemical engineering design data are now collected for processes where large productions are envisaged. These data are based on operation of suitably sized pilot plants. If guarantees for performance are desired by entrepreneurs then the know-how for turn-key plants is offered through some firms of project engineers who offer such guarantees. This approach which was initiated in 1965-66, has removed the major hurdle in technology transfer to industry.
8. A Process Release Committee consisting of the country's top industrialists was constituted in 1967. This Committee screens each process as regards its techno-economic viability before authorising NCL to release it to industry. The Committee has also rendered invaluable advice to the NCL regarding establishment of norms for calculation of production costs, financial terms for release, terms for sponsorship of projects by industry, etc.
9. Apart from establishment of industrial liaison by taking the above steps, several other contacts were established with industry through:—
  - (a) encouragement of two way traffic of experts from NCL and industry for purposes of discussion;
  - (b) participation of industry in sponsored research at NCL and / or at factory sites. Government of India have also helped the NCL in establishing contacts with public sector undertakings by appointing the Director and a few senior NCL scientists on the Boards of Directors of public sector undertakings related to chemical industry.
10. Publication of Annual Reports since 1965-66 and their wide distribution to trade associations, sponsors of research, interested industries, Government authorities, research institutions and to R and D Group and Project Leaders in the NCL have proved useful in bridging

the information gap between the NCL and users of research. The reports revealed useful basic data which led to introspection regarding NCL's activities and policies. These reports thus provided the motivation for a possible change for the better.

There has been a continuous improvement in the performance of the NCL, and its efforts are being increasingly appreciated in recent years by industry. This is in no small measure due to modifications of research and administrative policies and strategies enumerated above.

#### PROGRESS OF NCL DURING 1950-70

A factual overall analysis of the progress of the Laboratory during 1950-70 is presented in the form of cost/benefit analysis. Only those direct and indirect benefits which could be quantified to some extent have been considered. As mentioned earlier, in presenting the analysis we are fully conscious that benefits accruing from research and development work in a laboratory such as the NCL, which does not form an integral part of an industry, are difficult to measure quantitatively. Nevertheless we do hope that this introspection would be interesting and useful to the scientists in the NCL and perhaps also to our wide circle of readers.

#### INPUTS

ACTUAL EXPENDITURE : ( TABLE No. 1, Fig. No. 1 )

##### (a) *Recurring expenditure*

Actual figures of recurring expenditure, which include pay and allowances, maintenance and contingencies, chemicals and apparatus, are presented from 1950 onwards when some staff from DSIR, Delhi Laboratories, were shifted to the incomplete building of the NCL.

The year-wise recurring expenditure curve shows a steadily increasing trend. The rate of increase is shown below.

Year	1950-51	1954-55	1959-60	1964-65	1969-70
Recurring expenditure (Rs. in lakhs)	10.35	20.10	25.08	37.30	54.98
Percentage increase over the previous figure		94	25	43	47

The reduction in recurring expenditure by Rs. five lakhs between 1968-69 and 1969-70 is partly attributed to diversion of some NCL staff on projects sponsored by industry.

(b) *Capital Expenditure*

Capital expenditure on the NCL actually started from 1946-47. The total capital expenditure upto 1949-50 was Rs. 30.76 lakhs. This is not shown in Fig. 1. In the initial years (1946-55) there was considerable expenditure on building and capital equipment. Cumulative capital expenditure in each of the four succeeding quintets starting from 1950-51 is shown below:

Quintet*	1950-55	1955-60	1960-65	1965-70
Rs. in lakhs	44.17	10.99	46.38	32.01

\* (Quintet 1950-55 means April 1950 to March 1955 period)

The sudden spurt in capital expenditure during 1960-65 was due to installation and equipping of the Laboratory and two pilot plant buildings which were established in connection with the new Organic Intermediates & Dyes Division, started during these years to cater to the needs of the growing organic chemical industries. Later, this Division and the Chemical Engineering Division were merged into the larger Division of Chemical Engineering and Process Development. This period also saw the start up of the new Radio Tracer Laboratory and installation of modern analytical tools such as the Mass & NMR spectrometers. The period following 1965 has been one of steady growth, consolidation and greater use of facilities created rather than large scale expansion.

(c) *Pilot Plant Expenditure*

The expenditure on pilot plants was not shown separately until 1955-56 although in the earlier period a few pilot plants were also operated. A majority of NCL pilot plants were versatile, general purpose plants which, with some modifications, have been/are being used for other projects. The installation and running of these pilot plants have not involved heavy capital or recurring expenditure. Thus the total expenditure on pilot plant projects (43) for the years 1956-70 has been only Rs. 62.52 lakhs. As against this the benefit due to sales, premia, royalties and present assets (buildings, and equipment taken

as 50% depreciated present value) amount to Rs. 35.11 lakhs, so that the net expenditure on pilot plant projects has been only Rs. 27.41 lakhs during 1956-70. The percentage of expenditure on pilot plants, when compared with the total expenditure, is as follows:

Period	1956-60	1960-65	1965-70
Percentage	11	9	9

Apart from direct benefits and assets referred to above, the indirect benefits accruing to the economy have been substantial as will be seen from the benefit analysis which follows later.

STAFF : (Table No. 2 Fig. No. 2)

Staff strength of NCL is shown in five categories and the position reviewed every five years. There is a progressive increase in each category except Class IV-non-technical staff. As regards the percentages of different categories to the total strength of the laboratory, the situation appears to be fairly established within the first quintet, after which the same pattern is maintained.

The percentage rise in number of staff in each category at the end of each quintet over the base year of the quintet is shown in the following table:

Year	Sc.	Tech.	Admin.	Class-IV tech.	Class-IV non-tech.	Total
1950—55	51	567	108	360	64	134
1955—60	31	54	50	48	20	41
1960—65	52	42	16	27	25	36
1965—70	26	18	14	9	—	17

It will be seen that during the first quintet alone, the supporting staff increased at a higher rate, whereas in all the succeeding years (1955-70), the growth rate of supporting staff has been steadily decreasing and in the case of Class IV-non-technical staff there was no addition at all in 1965-70. In the case of scientific, as against all other staff, the growth rate has been higher although in the quintet 1965-70 the growth rate has been considerably reduced.

It will be seen that there has been a healthy growth of the scientific and technological expertise whereas the growth of Administrative and Class IV—non-technical staff has been purposefully restricted. It is interesting to record that although recruitment of all categories of staff during the 1965-70 period was restricted due to financial stringency and constraints and self-discipline, the productivity of the Laboratory as regards industrial research rose rapidly during the same period as will be seen from the benefit analysis which will follow.

#### INPUTS ON ACCOUNT OF SPONSORED RESEARCH

During recent years the industry is sponsoring research at the NCL on an increasing scale. The inputs and corresponding benefits resulting on account of sponsored research are discussed separately later.

#### BENEFITS

As against the inputs mentioned above, various direct and indirect benefits that have accrued during the last twenty years are discussed below.

#### DIRECT BENEFITS

##### (a) *Premia and royalties received* : (Table No. 3; Figs. 3 and 3 A)

The first NCL process (Liquid rubber) was released in 1958—59 for a premium of Rs. 5,000/-. The number of processes for which premia and royalties have been received are shown year-wise in Table 3. First royalty payment of Rs. 362/- was received when the Cation Exchange Resin from CNSL was produced commercially in 1960-61. Total premia received upto 1970 amount to Rs. 5.15 lakhs of which Rs. 1.37 lakhs against 23 releases were realized upto 1965, whereas during the last five years Rs. 3.78 lakhs were realized against 20 releases. The total royalty figure upto 1968-69 was Rs. 87.34 thousand of which in the period 1958-65 Rs. 34.26 thousand were obtained and in the last four years (1965-69) Rs. 53.08 thousand were realized. Although the premia figures have shown good improvement during 1966-70 period the same is not true for royalties received. This is due to the fact that during the last five years, processes are being released mostly on lump sum premia basis instead of royalties which were hard to realise for a variety of reasons.

##### (b) *Other receipts* :

Receipts on account of FCP sales, fees for analysis and other assignments from outside parties, CSIR share of institutional consultancy agreements, overhead charges for sponsored schemes which were credited to CSIR



account and miscellaneous receipts are shown below for each quintet starting from 1950-55.

(Rs. in lakhs)

Period	FCP sales	Testing & Analytical fees	CSIR share of consultancy	Overhead charges for Sp. Projects	Misc. receipts	Total
1950-55	—	—	—	—	—	—
1955-60	0.09	0.03	—	—	0.78	0.90
1960-65	0.68	0.13	—	—	8.91	9.72
1965-70	9.33	0.66	1.09	3.95	10.16	25.19
<b>Total</b>	<b>10.10</b>	<b>0.82</b>	<b>1.09</b>	<b>3.95</b>	<b>19.85</b>	<b>35.81</b>

(Fine Chemicals Project was started only in 1964. Prior to the inception of FCP, figures denote sales of the pilot plant products. Miscellaneous receipts include sale of fruits, vegetables, unserviceable stores, rent of premises, private use of transport, interest on various advances, application fees etc.).

(c) *Total Receipts :*

Total receipt (premia, royalties and other receipts) as percentage of recurring expenditure is given below for each of the four quintets.

Quintet		1950-55	1955-60	1960-65	1965-70
1. Total receipts	} Rs. in lakhs	—	1.08	11.26	28.49
2. Total recurring expenditure		76.51	110.01	163.91	263.02
3. (1) as percentage of (2)		—	0.98	6.87	10.83

It will be seen from the above that the total receipts under various headings have been rising. In 1969-70 these amounted to Rs. 10.41 lakhs corresponding to 18.9 % of the recurring expenditure on NCL for the same period.

## INDIRECT BENEFITS

### (a) *NCL processes under commercial exploitation* : (Table Nos. 4 & 5, Fig. Nos. 4 & 5)

The first NCL process (Cation Exchange Resin from CNSL) was commercially exploited in 1960-61 and the value of production during that period was Rs. 0.92 thousand. In 1968-69 as many as 15 new processes went into production. One of the reasons for this sudden rise was the start-up of productions based on work which was sponsored during 1964-68 period.

The yearly value of production curve shows a rising trend particularly from 1966. This is due to the fact that processes which were exploited in the years following 1966 had a larger commercial impact.

The total number of processes which were under commercial exploitation during 1969-70 was 44 and the value of production amounted to nearly Rs. 129 lakhs. The value of commercial production based on NCL processes has increased rapidly from Rs. 29 lakhs in 1966-67 to Rs. 129 lakhs in 1969-70.

Nearly 40% of the value of all productions based on NCL know-how could be claimed as saving in foreign exchange. Figure 5 shows also cumulative savings in foreign exchange.

### (b) *Sponsored Research* : (Table Nos. 6 & 7, Fig. Nos. 6, 7 and 7A)

The first sponsored scheme was undertaken in 1957-58. In 1961-62, 4 schemes were sponsored by industry at the NCL and subsequently this number is steadily increasing. In 1967-68, 25 schemes were sponsored by industry. During the last two years there is a marginal reduction in the number of schemes sponsored by industry. Out of the 60 sponsored schemes completed upto 1969-70, 14 were completed upto 1965, whereas during the last five years as many as 46 sponsored schemes have been completed. The same trend is depicted in the receipts from sponsored schemes. Out of the total of Rs. 42.62 lakhs so far received, Rs. 6.17 lakhs were received upto 1965, whereas during the last five years the receipts went up to Rs. 36.45 lakhs.

Guidelines and rules for sponsored research laid down in 1968-69 provided for charging overhead fees to sponsors for use of common facilities and assistance from administrative, scientific and technical infra-structure. These receipts on account of such overhead charges as are credited to CSIR funds are shown in Table No. 7.

Table Nos. 6 & 7 give overall data on schemes sponsored by Govern-

ment agencies, PL-480 and industry, while participation of industry alone is depicted in the following table.

Year	61-62	62-63	63-64	64-65	65-66	66-67	67-68	68-69	69-70
(i) No. of schemes in progress	4	6	6	5	13	21	25	19	14
(ii) No. of schemes under commercial exploitation	—	1	2	2	4	4	5	9	9
(iii) Value of production (Rs. in lakhs)	—	0.37	0.68	0.83	3.00	20.72	23.50	47.80	60.89

The above data indicate that the industry is becoming increasingly appreciative of NCL's efforts and is now willing to commit more and more funds for development of know-how at the NCL. Such collaborative participation by industry has also led to substantial gains in economy. The cumulative value of production based on sponsored processes which were commercialized upto 1970 amounted to Rs. 157.80 lakhs. Figures for 1969-70 alone amounted to 9 processes of which the value of production was Rs. 60.89 lakhs.

(c) *Patents* : (Table No. 8, Fig. No. 8)

Patents are generally taken for new approaches to known facts, modifications and improvements in known methods or new processes and products etc., visualized by the scientific worker. 17 patents taken by the chemical section of DSIR, New Delhi, were inherited by NCL in 1950-51, and 10 more new patents were taken in that year. Upto the Year 1962-63, there have been substantial additions in the number of patents every year. However, this trend appears to have changed from 1963-64 and during the last 7 years only 21 new patents have been added. In addition, during the last few years patents which were not commercially significant and which were hitherto unexploited were allowed to lapse. Consequently the total number of patents in force have decreased whereas the number of patents licensed for commercial exploitation

have increased and the gap between unused patents and those that have been commercially exploited has narrowed down considerably. The value of productions based on commercially exploited patents for 1969-70 was Rs. 14.08 lakhs, and the cumulative value of such productions upto 1970 was Rs. 50.23 lakhs.

(d) *Publications* : (Table No. 9, Fig. No. 9)

The number of papers published in scientific journals reflects the inventive and innovative strength of the research institution. The number of publications per year shows an increasing trend upto the year 1965-66. Thus as many as 161 research papers were published during 1965-66. Later on, the number of publications have been decreasing. This decrease may be attributed to the substantial increase in the number of industrial research projects undertaken during the last five years. The number of publications in foreign journals also follow the same trend.

(e) *Research degrees* :

Whereas a substantial proportion of the scientific staff is engaged in industrial research, basic research is also encouraged and is conducted largely through CSIR Research Fellows (Junior and Senior), Guest Workers and CSIR Scientific Pool Officers. These research fellows, guest workers and NCL staff whose research work is also of fundamental interest, have been permitted to carry out research work for the Master's and Doctor's degrees in chemistry, chemical technology and chemical engineering. The number of M.Sc. and Ph.D. degrees awarded to such scientists for each of the four quintets is given below :

Quintet	1950-55	1955-60	1960-65	1965-70	Total
M.Sc.	1	8	11	12	32
Ph.D.	2	26	57	151	236
M.Sc.(Tech.)	—	—	4	6	10
Ph.D.(Tech.)	—	—	—	12	12
<b>Total</b>	<b>3</b>	<b>34</b>	<b>72</b>	<b>181</b>	<b>290</b>

During 1965-70, in all 37 NCL-staff scientists were recognized as Research Guides. Data for earlier periods is not available.

Young and bright chemists coming to NCL from all over India as Research Fellows & Guest Workers have provided the NCL staff a welcome opportunity of contact with the younger generation. These young men engaged in basic research projects (many of which have arisen out of the needs of the applied projects in progress) have furnished the necessary counterpoise to our applied research activity so that the total research effort in both basic and industrial research is well integrated and appropriately balanced. While the clamour for the Laboratory to do more and more industrial research is growing, it is necessary to emphasize the fact that the NCL is a *discipline-oriented* laboratory unlike several other CSIR laboratories which are either product or utility-oriented, or are created to cater the needs of particular regions or industries.

The NCL may justly be proud of its excellent record in turning out well qualified and trained research scientists, thereby strengthening the scientific and technological capability of the NCL and of the country as well. Several projects, which started initially as Ph.D. projects, eventually led to the development of industrially useful products/processes, of which the following few examples may be cited:—Thermistors, aniline by hydrogenation of nitrobenzene, acetanilide, new series of disperse dyes for synthetic fibres, hard ferrites etc. Thus basic research, undertaken as a part of thesis work, has also contributed in no small measure to the development of technology in many cases.

□   □   □

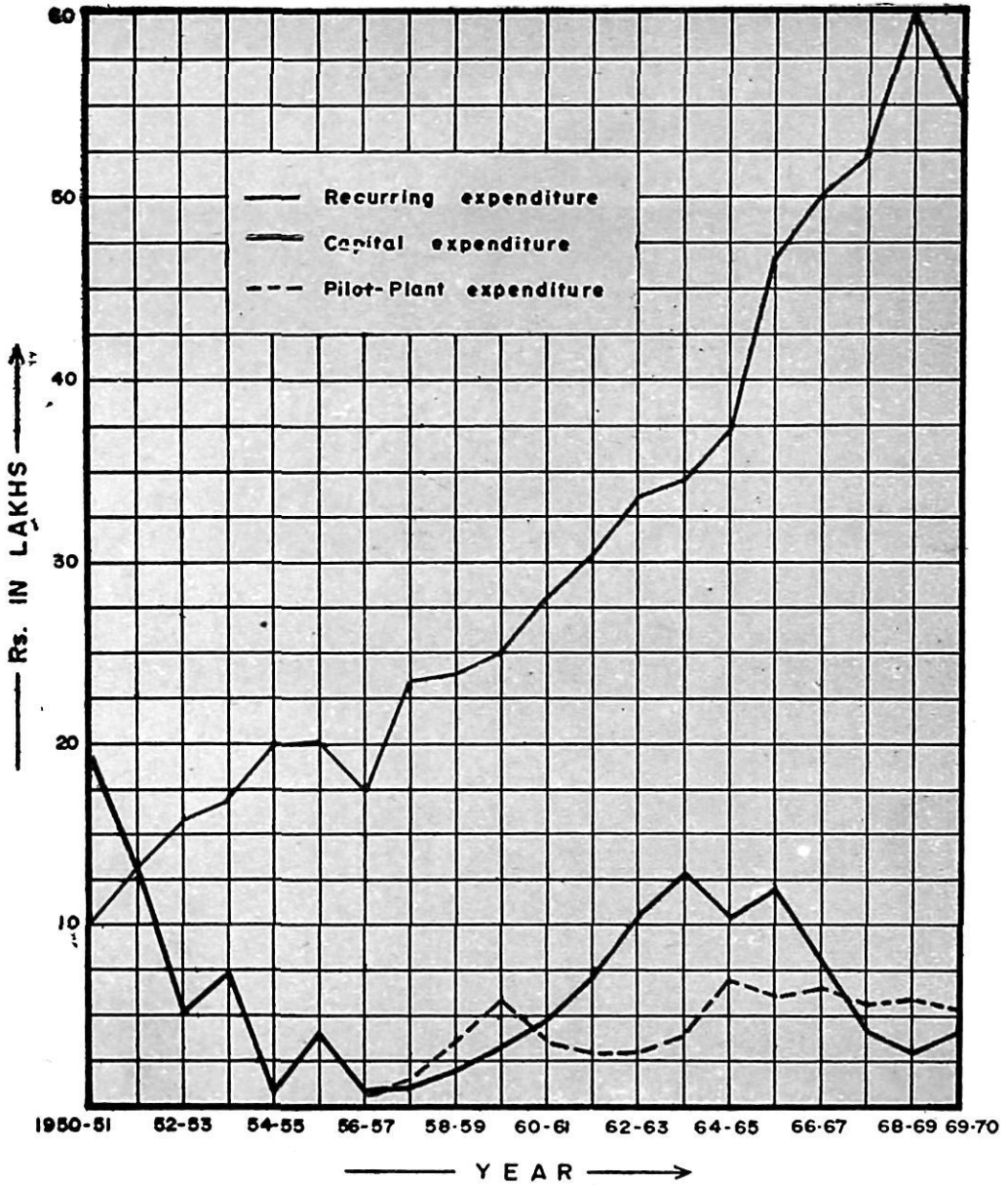
## **TABLES AND GRAPHS**

TABLE 1 : YEARWISE EXPENDITURE OF NCL

( Rs. in lakhs )

Year	Recurring Expenditure	Capital Expenditure	Pilot Plant Expenditure	Total
50—51	10·35	18·73	—	29·08
51—52	13·35	12·58	—	25·93
52—53	15·80	5·06	—	20·86
53—54	16·91	7·17	—	24·08
54—55	20·10	0·63	—	20·73
55—56	20·20	4·04	—	24·24
56—57	17·46	0·74	0·70	18·90
57—58	23·46	0·94	1·65	26·05
58—59	23·81	2·02	3·68	29·51
59—60	25·08	3·25	5·93	34·26
60—61	28·13	4·79	3·81	36·73
61—62	30·33	7·18	3·12	40·63
62—63	33·60	10·74	3·17	47·51
63—64	34·55	13·08	3·99	51·62
64—65	37·30	10·59	6·90	54·79
65—66	46·64	12·20	6·20	65·04
66—67	49·71	8·09	6·41	64·21
67—68	51·88	4·35	5·71	61·94
68—69	59·81	3·21	5·99	69·01
69—70	54·98	4·16	5·26	64·40
<b>Total :</b>	<b>613·45</b>	<b>133·55</b>	<b>62·52</b>	<b>809·52</b>

EXPENDITURE OF NCL Fig. 1





*TABLE 2 : STAFF IN POSITION — ACTUAL WORKING — AS ON 1st. APRIL OF EVERY YEAR*

Year	Scientific		Technical		Administrative		Class IV Technical		Class IV Non-Technical		Total No.
	No.	% of total	No.	% of total	No.	% of total	No.	% of total	No.	% of total	
1950	72	48 %	12	8 %	24	16 %	15	10 %	28	18 %	151
1955	109	31 %	80	23 %	50	14 %	69	20 %	46	12 %	354
1960	143	29 %	123	25 %	75	15 %	102	20 %	55	11 %	498
1965	217	32 %	175	26 %	87	13 %	130	19 %	69	10 %	678
1970	273	34 %	207	26 %	100	13 %	142	18 %	68	9 %	790

STAFF IN POSITION Fig. 2

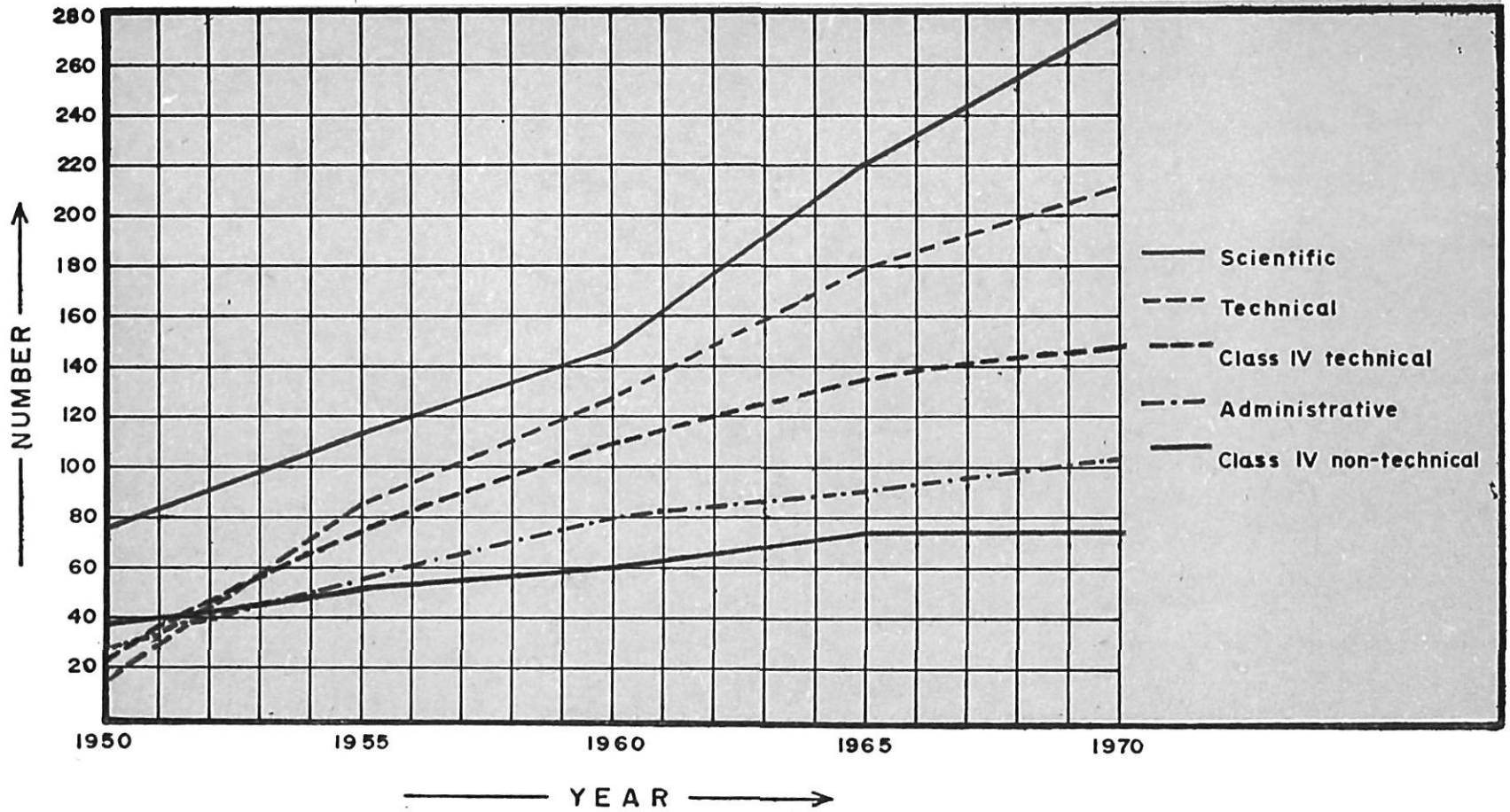


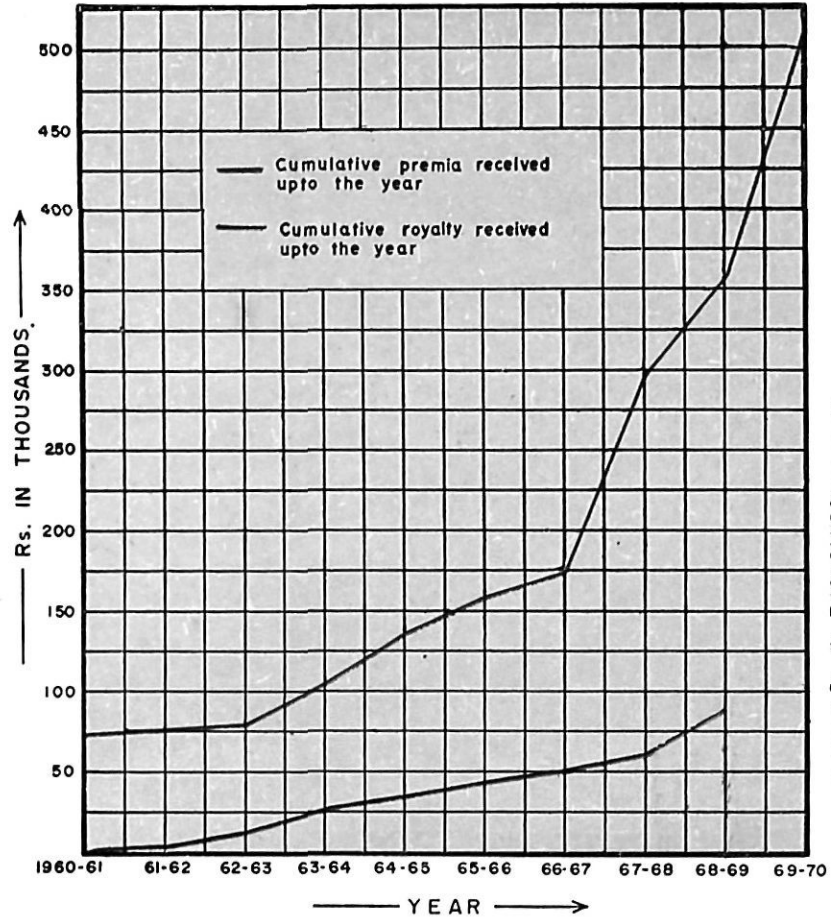
TABLE 3 : PREMIA & ROYALTY

(Rs. in thousands)

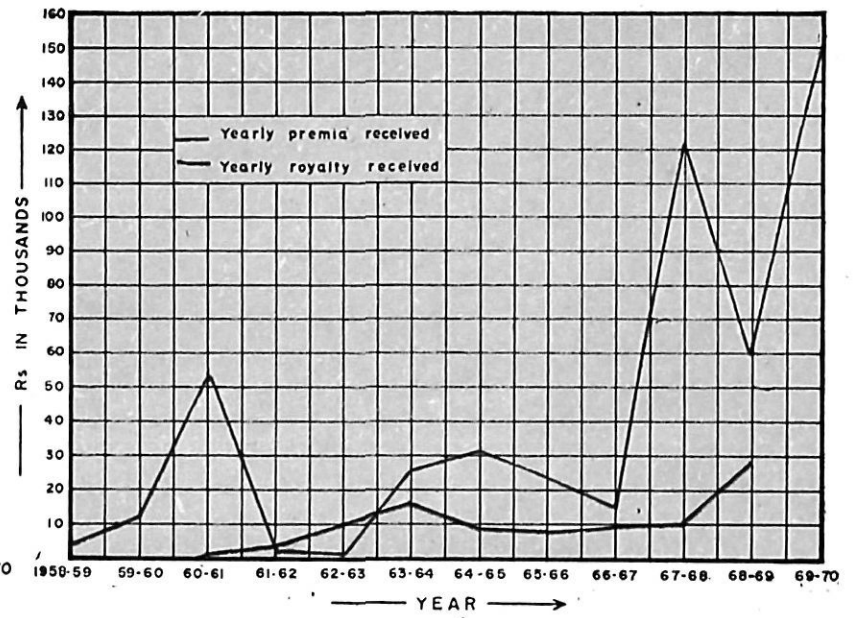
Year	58—59	59—60	60—61	61—62	62—63	63—64	64—65	65—66	66—67	67—68	68—69	69—70
1. Year-wise premia received.	5.00	13.00	55.50	3.50	2.00	26.50	32.00	24.00	15.00	123.00	60.00	155.00
2. No. of processes for which above premia is received.	1	2	7	1	1	7	4	5	1	3	5	6
3. Cumulative premia received upto the year.	5.00	18.00	73.50	77.00	79.00	105.50	137.00	161.00	176.50	299.00	359.50	514.50
4. Year-wise royalty received.	—	—	0.36	2.40	8.97	14.77	7.76	7.31	8.52	9.62	27.63	26.42*
5. No. of processes for which above royalty is received.	—	—	1	2	3	4	8	7	7	6	8	—
6. Cumulative royalty received upto the year.	—	—	0.36	2.76	11.73	26.50	34.26	41.57	50.09	59.71	87.34	—

\* Figure upto December 1969.

CUMULATIVE PREMIA & ROYALTY RECEIPTS Fig. 3A



PREMIA & ROYALTY RECEIPTS Fig. 3



*TABLE 4 : NCL PROCESSES UNDER COMMERCIAL EXPLOITATION*

Year	60—61	61—62	62—63	63—64	64—65	65—66	66—67	67—68	68—69	69—70
1. Year-wise new additions to number of processes in the production	1	1	3	6	4	1	7	3	15	3
2. Total number of processes in production	1	2	5	11	15	16	23	26	41	44

NCL PROCESSES UNDER COMMERCIAL EXPLOITATION Fig. 4

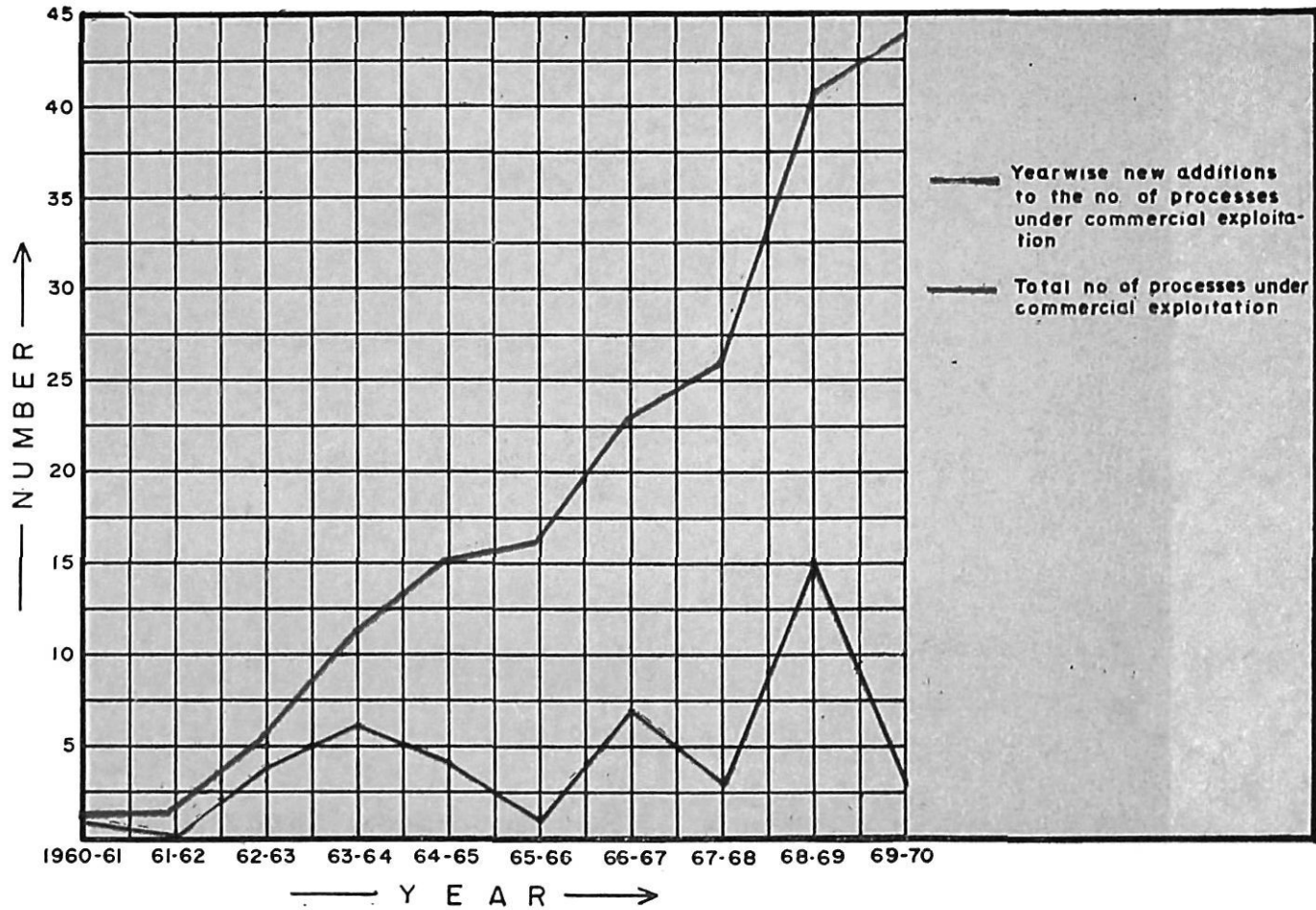


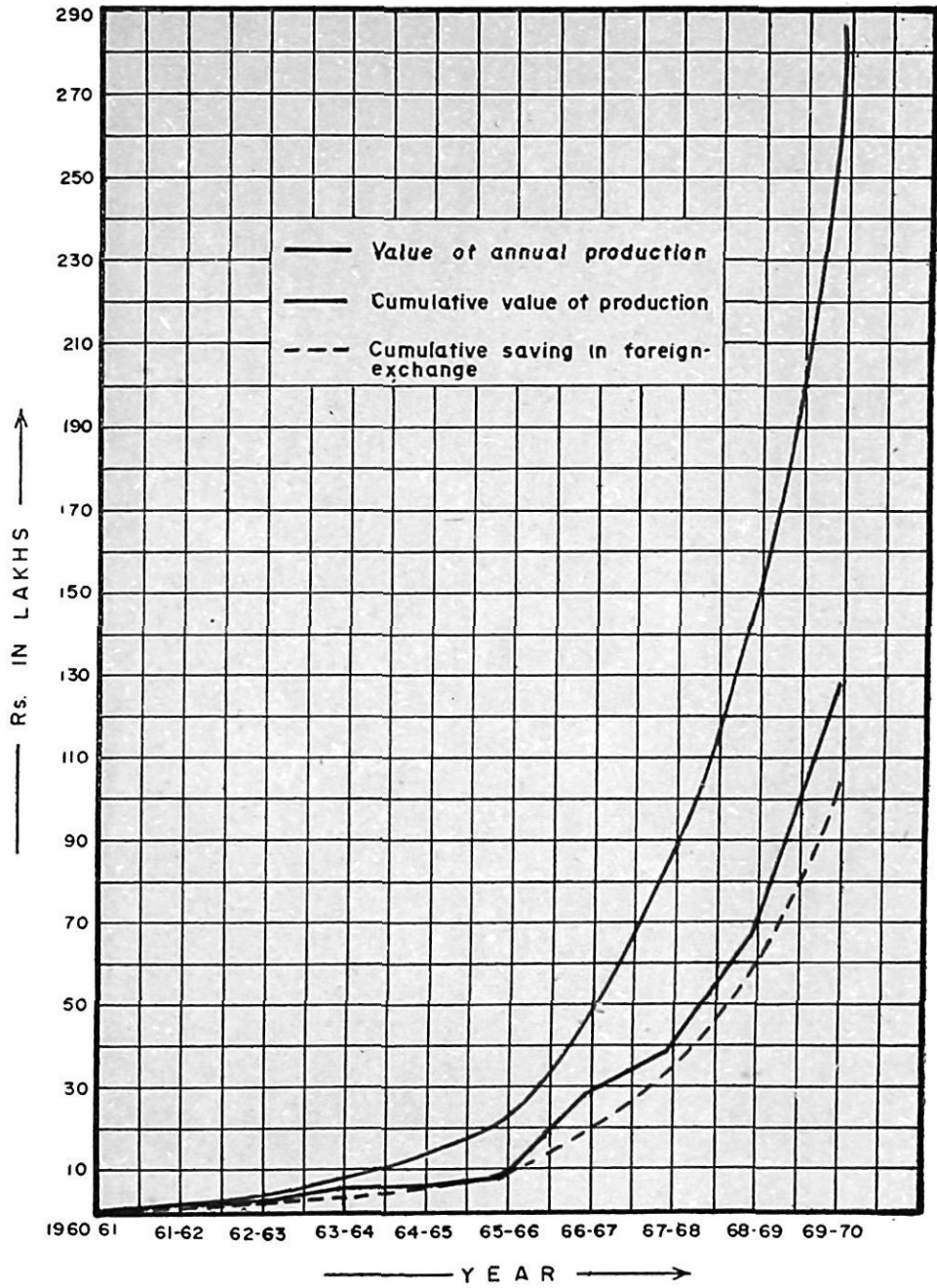
TABLE 5 : VALUE OF PRODUCTION BASED ON NCL KNOW-HOW

(Rs. in lakhs)					
Year	60—61	61—62	62—63	63—64	64—65
1. Value of yearly production.	0·92	0·92	2·25	5·22	5·51
2. Cumulative value of production.	0·92	1·84	4·09	9·31	14·82
3. Cumulative saving in foreign exchange.*	0·37	0·74	1·64	3·72	5·93
	65—66	66—67	67—68	68—69	69—70
1.	7·86	28·82	39·27	68·05	128·58
2.	22·68	51·50	90·77	158·82	287·40
3.	9·07	20·60	36·31	63·53	114·96

\* @ 40% of (2)

Values of major FCP productions mentioned in printed Annual Reports of 1966-70 are not included.

VALUE OF PRODUCTION BASED ON NCL KNOWHOW Fig. 5

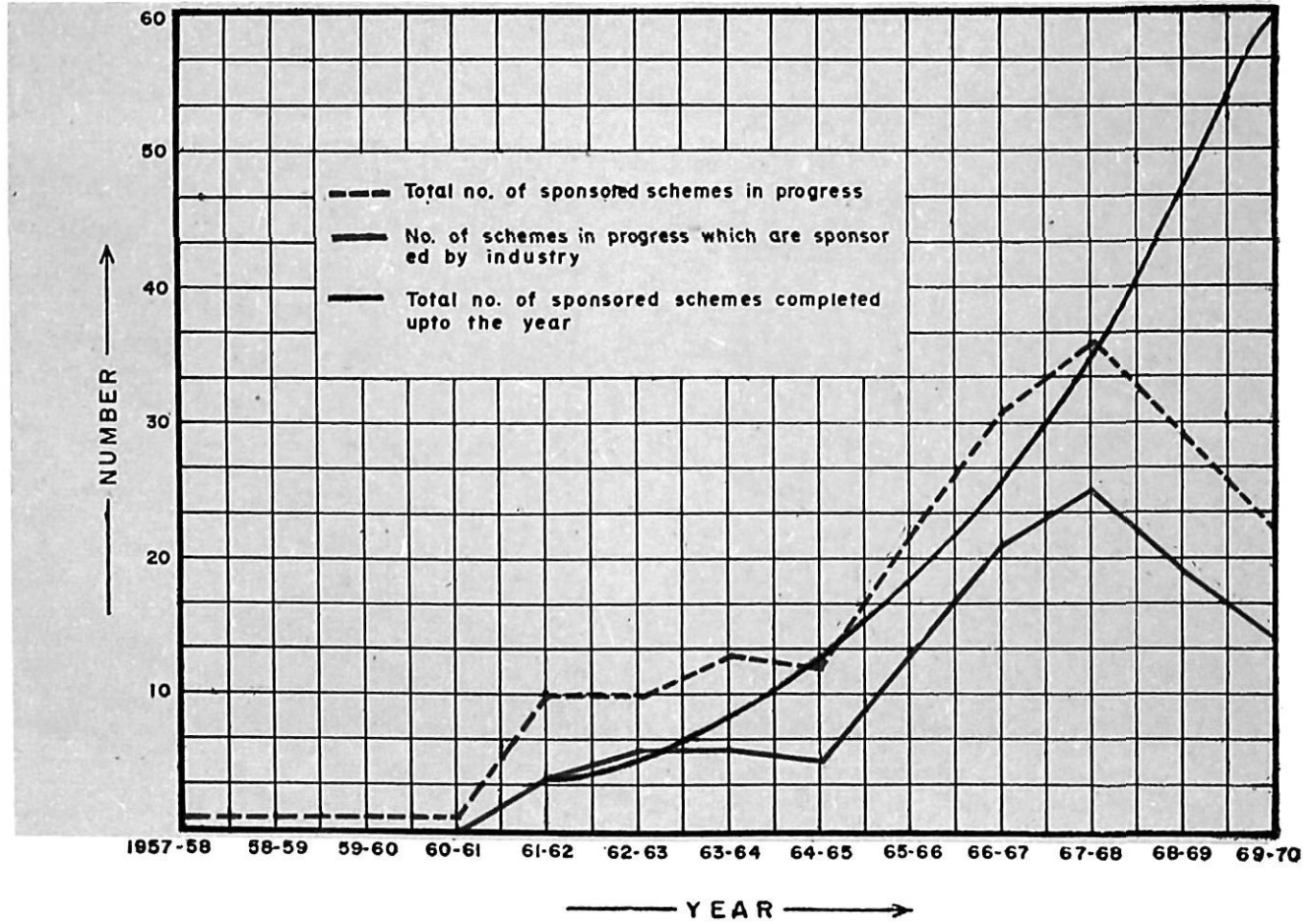




**TABLE 6 : NUMBER OF SPONSORED SCHEMES**

Year	57—58	58—59	59—60	60—61	61—62	62—63	63—64	64—65	65—66	66—67	67—68	68—69	69—70
1. No. of sponsored schemes in progress.	1	1	1	1	10	10	13	12	22	31	36	29	22
2. No. of schemes in progress sponsored by industry.	—	—	—	—	4	6	6	5	13	21	25	19	14
3. Total No. of sponsored schemes completed upto the year.	—	—	—	—	4	5	9	14	19	23	34	49	60

INFORMATION ABOUT THE SPONSORED SCHEMES Fig. 6

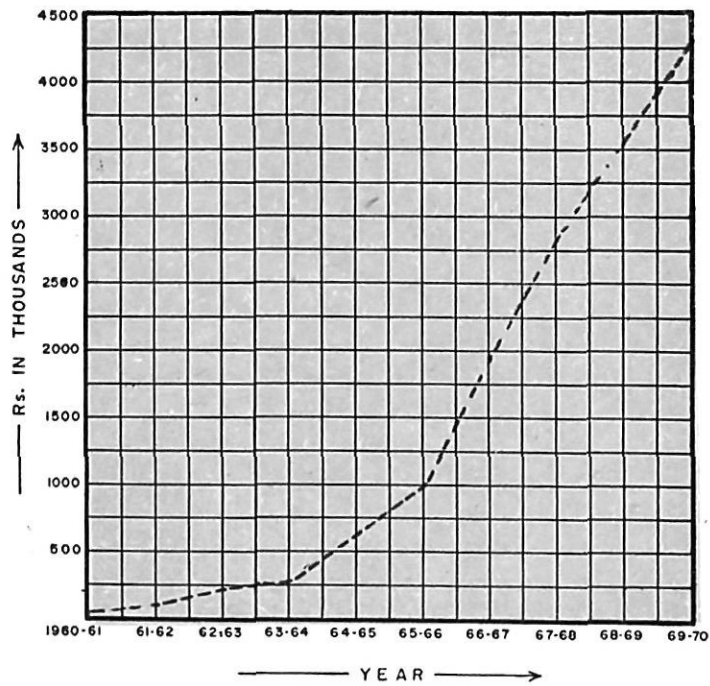


**TABLE 7 : RECEIPTS FROM SPONSORED SCHEMES**

( Rs. in thousands )

Year	57—58	58—59	59—60	60—61	61—62	62—63	63—64	64—65	65—66	66—67	67—68	68—69	69—70
1. Overhead charges credited to CSIR funds.	—	—	—	—	—	—	—	—	—	—	32·66	76·02	286·00
2. Yearly receipts from industry.	—	—	—	—	17·47	45·88	20·45	63·00	96·74	101·21	206·33	120·73	415·80
3. Total yearly receipts.	4·23	4·56	9·59	11·90	49·59	138·06	33·67	365·11	355·00	951·00	911·00	711·00	717·00
4. Cumulative receipts	4·23	8·79	18·38	30·28	79·87	217·93	251·60	616·71	971·71	1922·71	2833·71	3544·71	4261·71

CUMULATIVE RECEIPTS FROM SPONSORED SCHEMES  
Fig. 7A



RECEIPTS FROM SPONSORED SCHEMES Fig. 7

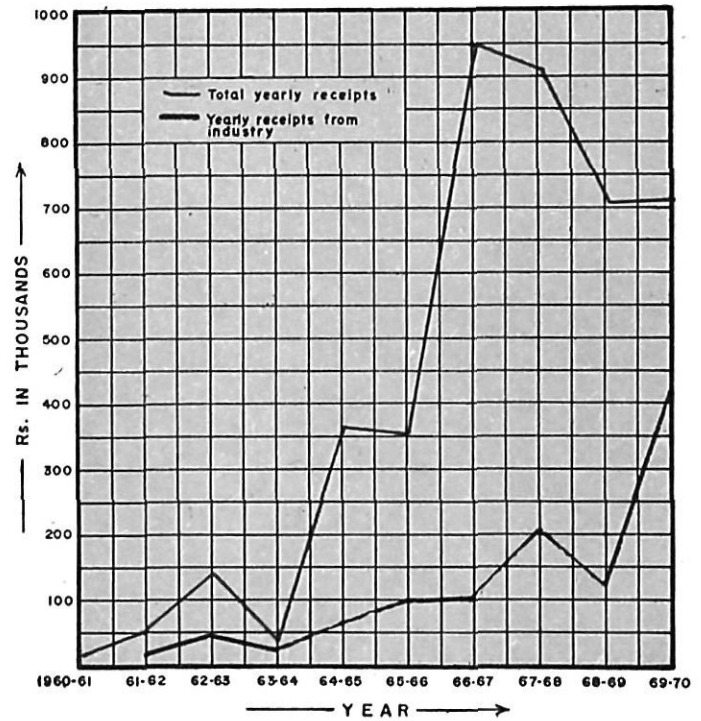
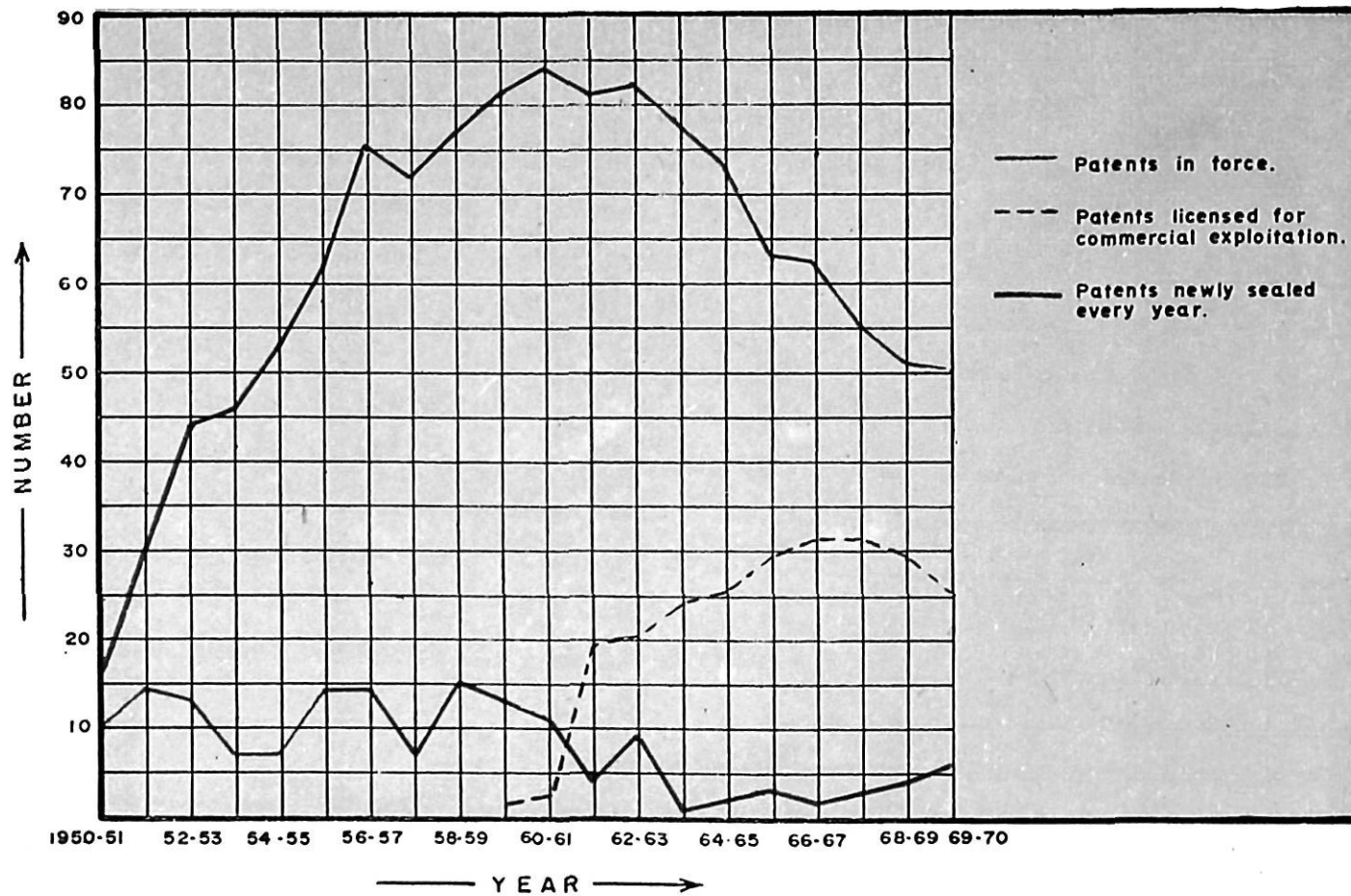


TABLE 8 : PATENTS

Year	50—51	51—52	52—53	53—54	54—55	55—56	56—57	57—58	58—59	59—60	60—61	61—62	
1. Patents in force.	17	31	44	46	53	62	75	72	77	81	81	81	
2. Patents newly sealed every year.	10	14	13	7	7	14	14	7	15	13	11	4	
3. Patents licensed for commercial exploitation.	—	—	—	—	—	—	—	—	—	1	2	19	
						62—63	63—64	64—65	65—66	66—67	67—68	68—69	69—70
1. ... ..						82	77	73	63	62	55	51	50
2. ... ..						9	1	2	3	2	3	4	6
3. ... ..						20	24	25	29	31	31	29	25

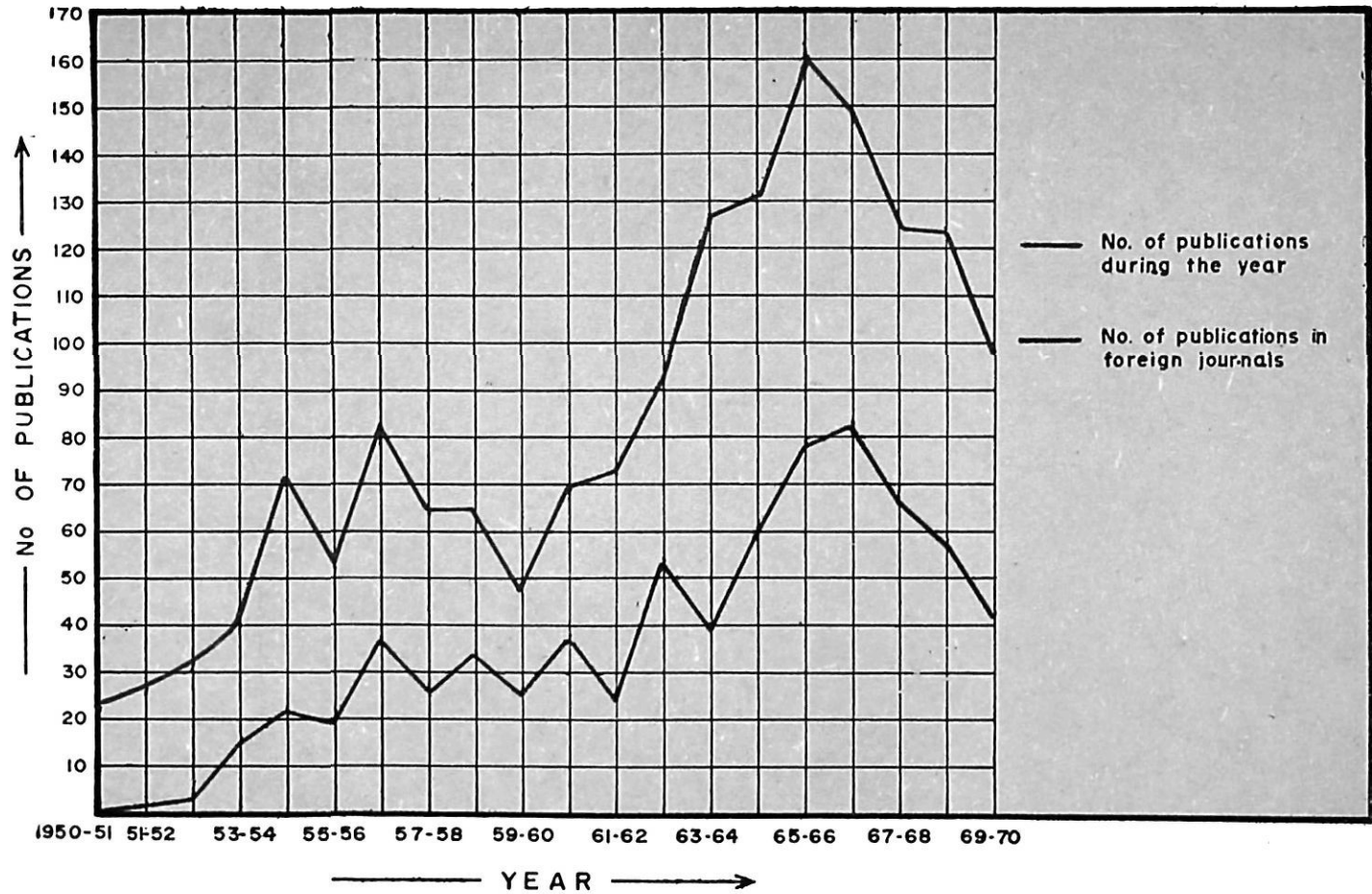
PATENTS Fig. 8



**TABLE 9 : PUBLICATIONS**

Year	50—51	51—52	52—53	53—54	54—55	55—56	56—57	57—58	58—59	59—60		
1. No. of publications during the year.	23	26	31	39	71	53	82	64	64	47		
2. No of publications in foreign journals.	1	2	3	15	22	19	37	26	34	26		
	60—61	61—62	62—63	63—64	64—65	65—66	66—67	67—68	68—69	69—70 <sup>a</sup>	Total	
1. ... ..	69	73	92	127	131	161	149	125	124	97	1648	
2. ... ..	38	24	54	39	61	79	83	67	58	42	730	

PUBLICATIONS Fig. 9





## CREDITS

<i>Editor</i>	Dr. Gupta, J.
<i>Compiled by</i>	Mr. Lele, A. M. Mr. Tambe, S. A. Mr. Shirur, R. J. Mr. Joshi, S. S. Mrs. Adke, S. S. Miss Khandekar, V. V.
<i>Photographs</i>	Mr. Oak, N. C.
<i>Illustrations and graphs</i>	Mr. Rawal, A. H. Mr. Deo, S. N.
<i>Cover design</i>	Mr. Patil, C. H.
<i>Published by</i>	Dr. Tilak, B. D. Director, NCL
<i>Printed by</i>	S. J. Patwardhan, Sangam Press Limited, Poona-4.