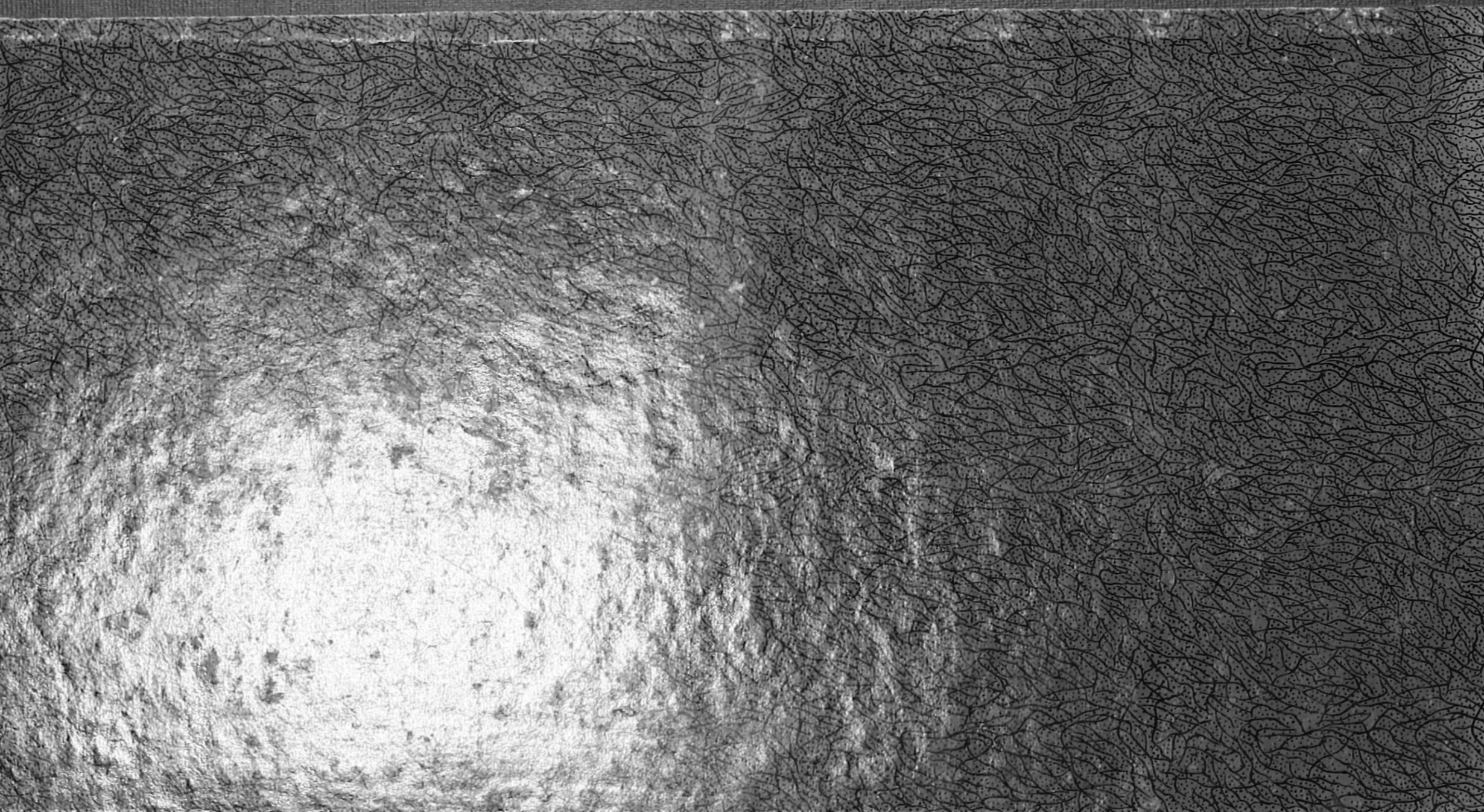
N.C.L. RESEARCH PROGRAMME. 1960-61.



NATIONAL CHEMICAL LABORATORY

RESEARCH PROGRAMME AND PILOT PLANT PROGRAMME FOR 1960-61 .

Contents

INORGANIC CHEMISTRY

(1) CHEMISTRY OF RARE METALS

(a) Studies in the Isolation of Pure Rare Earths

The work on the development of efficient methods of preparing high-purity light rare earths by an appropriate combination of available fractionation techniques will be continued. The essential raw material now supplied by the Department of Atomic Energy, which is also partly financing the project, is a nearly ceria-free mixture of the rare earths from monazite in the hydroxide or carbonate form. This will make the isolation of some of the rare earths beyond the first five (La, Ce, Pr, Nd, Sm) a more feasible proposition. There is also good probability of getting during the year experimental quantities of Indian minerals containing heavy rare earths for this latter work.

As a new complexing eluant for rare earths, sodium tripolyphosphate has given encouraging results, particularly in the isolation of samarium, which deserve further study. Some fundamental investigation of non-aqueous chemistry of the rare earths is also proposed to be undertaken this year from the laboratory stock of pure ceria, lanthana and praseodymia.

(b) Separation of Zirconium from Hafnium

This project, which comprises the twin projects of making reactor-grade zirconia and pure hafnia starting from Indian zircon (ZrO_2 65.1, HfO_2 1.7 per cent approx.), is to be continued. Grant-in-aid by the Department of Atomic Energy has substantially increased this year. It is proposed to set up and operate an integrated laboratory unit processing zirconium oxychloride to reactor-grade quality, collecting a hafnium concentrate and isolating pure hafnium oxide from the latter. In addition to the ion-exchange and partition chromatographic techniques involved in this work, some liquid-liquid extraction study will be taken up, in which a beginning has been made already.

(c) Separation of Niobium from Tantalum

A detailed flowsheet with basic laboratory data on the new process (Ind. Pat. Nos. 63736 and 67513) of obtaining pure niobium and tantalum oxides from Indian tantalocolumbites was drawn up, and is now with the Atomic Energy Establishment, which is understood to be examining the process for development. Further work will be taken up on the advice of the Establishment.

(2) LABORATORY PREPARATION OF SPECIAL CHEMICALS

The programme of work for the year will be similar to the previous year's programme. However, the following special items will be considered during the year:-

(a) Chromatographic alumina - The preparation and standardization of three types of alumina (neutral, acidic and alkaline) will be undertaken. A preparation unit of the capacity of about 7 kg/day will be put in operation.

(b) Active silica gel - Based on preliminary work on the preparation of active silica gel already carried out in this Laboratory, a small laboratory unit for its preparation will be set up.

(c) Sodium hydride - Using the modern high-speed stirring technique, sodium hydride will be prepared from sodium dispersion. Suitable methods for the preparation of sodium borohydride, sodium aluminium hydride and other substituted complex hydrides will be developed from this important starting material.

(d) Ultrapure materials for semiconductors - Materials of greater and greater purity are in demand in solid state research. For the thermistor project of this Laboratory, very pure oxides of Cu, Mn, Ni, Co, etc., are required in quantities of several hundred grams each. Special methods of preparation will have to be developed to make these materials from inferior grades which are readily available in the market.

(e) Solid luminescent materials - On a request from a Govt. Dept. for 10 lb. of fluorescent zinc sulphide, the preparation of this material is being carried out as a priority item. The preparation and characteristics of other solid luminescent materials will also be undertaken.

(f) Soda-lime - Special grade soda-lime required in hospitals for use in metabolic tests and oxygen therapy is wholly imported, involving a foreign exchange of over Rs 10,000, but a more important factor is the question of its ready availability at all times.

(3) BARIUM AND STRONTIUM CHEMICALS

Laboratory work carried out so far on the chlorination of Andhra barytes has established that a high degree of conversion efficiency of the mineral directly to barium chloride is obtainable with low losses of chlorine.

It is considered necessary to extend this work to a possible recovery of the sulphurous part of the mineral. The recovered sulphur will form an important byproduct of our process. A comparative study with alternative methods, for example, alkali fusion of the mineral in presence of catalyst, high temperature reduction, etc., will be necessary to establish our process on a sound basis. Further work on the purity of the barium chloride obtained, which depends not only upon the impurities in the mineral, but also on the composition of the reducing agent, will have to be done to prepare C.P. and better grades of barium salts.

In an analogous manner the preparation of strontium chemicals from Indian celestite will also be tried. The principal application of Sr. compounds includes pyrotechnics, lubricants, and driers. A Govt. Dept. expressed interest in the preparation of peroxide from indigenous sources. The preparation of this material as well as of other important strontium chemicals (hydroxide, oxide, nitrate) will be undertaken. Strontium greases (produced from strontium hydroxide and various soap stocks) are of special significance because of their superior heat resistance and protection against moisture or salt corrosion.

(4) OXIDATION OF SULPHUR DIOXIDE TO TRIOXIDE

On a request from a firm the problem of making an active vanadium catalyst from imported vanadium salts was examined and some laboratory work has been carried out since July 1959 with promising results. It is proposed to follow up this work during the coming year. The investigation will also include regeneration of the 'spent' catalyst from contact chambers by a suitable and simple process. A fundamental study of the catalytic activity of mixed oxides of other metals will also be made.

(5) ANALYTICAL RESEARCH AND CONTROL

This is a service-cum-research project involving the modification of known methods and development of new methods of analysis to meet the needs of several research projects of the Laboratory. Emission spectrography, spectrophotometry, chromatography including ion exchange, and electro-analysis including polarography are the different techniques employed in the development and standardization of new analytical methods. As examples of such methods already worked out may be mentioned (1) spectrographic methods for determining hafnium-zirconium in low quantities, and of tantalum and niobium, (2) spectrophotometric methods for titanium, niobium, germanium and phosphorus, and (3) a polarographic method for uranium. The need for the coming year is to estimate impurities of other rare earths in a prepared sample of rare earth oxide. Spectrographic methods for the determination of such traces of rare earth oxides will be standardized or developed as required.

In addition, some fundamental studies using ion-exchange and polarographic techniques will be carried out on some rare metal complexes of sodium tripolyphosphate, a new complexing agent of growing importance.

Following a suggestion to examine fly-ash samples of cement factories for the economic recovery of lithium, twelve samples of fly-dust from kilns and one from boilers from cement factories under A.C.C. Ltd. were examined for lithium content. Only in three samples, lithium was detectable (detection limit 0.01%), and was estimated to be less than 0.02%.

PHYSICAL CHEMISTRY

(1) SURFACE CHEMISTRY

(a) Prevention of Water Evaporation by Monomolecular Films

In the past year a few long chain compounds were synthesised, tested, and found to give encouraging results. It is now proposed to collect more extensive performance data of these compounds. Work will also be carried out on the (i) preparation and testing of mixtures of suitable alcohols and their derivatives prepared in the Laboratory; (ii) study of the film properties such as equilibrium film pressure and compressibility, 2-D viscosity, rate of spreading, orientation and phase behaviour of the potential compositions; (iii) further standardisation and (if necessary) modification of the techniques of measurement the film properties.

(b) Catalysis

The construction of the BET apparatus for surface area measurements and pore size determinations has been completed and the calibration of the apparatus is being undertaken. The construction of low and high temperature calorimeters for the determination of heats of adsorption and heat capacity is in progress. An apparatus for measurement of magnetic susceptibility of catalysts is proposed to be constructed. Work will be continued on: (i) the oxidation of important constituents of Indian turpentine oil; (ii) the recovery and reactivation of spent nickel catalyst obtained from hydrogenation of oils; (iii) the nature of the low temperature chemisorption of hydrogen and carbon monoxide on some oxide catalysts.

Some fundamental studies on the influence of radiation induced defects in solid catalysts and of radiation itself on catalytic reactions will be undertaken.

(2) SOLID STATE CHEMISTRY

The structural, electrical and magnetic properties of some manganites and ferrites have been studied in this Laboratory. On the basis of these results 'thermistors' suitable for various applied devices were prepared on a pilot plant scale. The production is proposed to be continued for one more year. New compositions suitable for ceramic sintered oxide permanent magnets have been evolved, and these will now be used for preparing magnets on a pilot plant scale.

The laboratory investigations will be carried out on the following lines:

(a) Ferroelectric materials - Our investigations in the past two years have led to the synthesis of new ferroelectric compositions which show dielectric constants higher than barium titanate, high Curie temperatures, and well developed hysteresis loops. Further work will be continued to evolve suitable devices using these materials.

In addition to the above studies on polycrystalline materials, measurements of electrical, structural and optical properties of single crystals of a few useful phases will also be undertaken.

(b) Electroluminescence - A number of phosphors have been synthesised in the past year and some of them show good electroluminescence. It is now proposed to measure the spectral distribution of intensities and the efficiency in relation to the composition, crystal structure, doping and pretreatments of the phosphors.

(c) Crystal rectifiers and transistors - This industrially important field is of recent origin with wide scope for development. During this year we propose to begin work on (i) preparation of silicon of semiconductor grade from indigenous raw materials, (ii) zone refining and single crystal growth, and (iii) fabrication

of semiconductor devices.

(d) Solid state and molecular theory - The following theoretical investigations will be undertaken: (i) Magnetic exchange interaction and bonding in solids; (ii) Co-operative interactions and distortion in crystals; (iii) Electron-phonon interaction in solids; (iv) Behaviour of electron gas in the field of uniform positive charge as related to optical properties of molecules.

(e) Crystal and molecular structure studies of some important compounds will be continued.

(f) Corrosion - In recent years there has been great interest in the use of the rare earth metals and compounds as materials for reactor control and for preparation of compounds with high dielectric constants in solid state devices. It is planned to obtain a detailed knowledge of their oxidation behaviour and structure under different conditions. The kinetics of oxidation will be studied with the help of a Gulbransen type of microbalance, the designing and construction of which is already under way. This work is supported by the Atomic Energy Commission.

(g) Diffusion in the solid state - Studies on diffusion in solid state is stimulated by the rapidly growing interest in high temperature reactions over the wide area of applied science including nuclear technology. High temperature reactions in solids such as welding, sintering, corrosion, hardening, recrystallisation and formation of compounds in the solid state are closely connected with atomic mobility in solids. Radioactive isotopes will be used for the study of intermetallic diffusion and of self-diffusion in systems of applied interest.

(3) COLLOID-CHEMICAL AND SOLUTION PROPERTIES

(a) Cellulose. - Work was started to correlate some physical properties of pulps, obtained from different raw materials, with their suitability for rayon manufacture. The physico-chemical properties of the pulps, such as evaluation of molecular weight, rate of swelling, crystallite size, crystalline-amorphous ratio, and morphology of the treated fibre, will be investigated at all important steps in the xanthate process with a view to characterising their differences and correlating with the actual performance data under industrial conditions.

The light scattering and flow property studies of several rayon grade pulps dissolved in zinc ethylene diamine will be continued.

(b) Studies on the kinetics of the degradation of rubber which will throw light on the mechanism and also help in evaluating the energy of activation of the process under various conditions will be concluded this year.

(4) USE OF RADIOACTIVE ISOTOPES FOR MEDICAL AND OTHER RELATED RESEARCH

During the last few years, the NCL has collaborated with institutions such as the Armed Forces Medical College and the Virus Research Centre, Poona, in the investigation of various medical problems using radioactive isotopes. It is proposed to collaborate with the AFMC in investigating the extent of defective red blood cell production and iron utilisation due to various types of anaemias encountered in India.

(5) INSTRUMENTATION

Work on the nuclear magnetic resonance spectrometer which is in progress will be continued. When the staff has been recruited, work on the rapid scan infrared spectrometer will be undertaken.

Pilot Plant Project

THERMISTORS

Nature of the Project

In the past year a large number of thermistors of various types and desired characteristics have been prepared, mainly in the form of beads. The compositions used were those synthesised in the laboratory. It is proposed to continue this work of thermistor fabrication for one more year with a greater emphasis now on the rod and pellet types.

Background of the Project

In the past year a number of thermistors have been made to suit the special requirements of various organisations. The demand so far has been for a few pieces per party, but a greater demand is envisaged as the industries get familiarised with the multifarious applications of thermistors. The electrical characteristics, ageing tests, efficiency, etc., of different types of thermistors have been continuously examined with a view to improving the products.

Reasons for the Pilot Plant Development

It is proposed to continue the manufacture of thermistors for another year, as a large number of instrument industries will be benefited by our products. In addition to the bead type mainly undertaken in the last year it is proposed to make rod and pellet type thermistors in large quantities. The compositions suitable for these have already been successfully synthesised on a laboratory scale. For these types a great demand is expected from the radio, telephone and other allied industries. The Radio Research Committee estimates the requirements of thermistors for AC/DC radio sets alone at Rs 2 lakhs per year.

Estimated Duration of the Project - The project will continue for one more year. Sanction for two years was obtained last year and the staff was appointed only a few months ago.

Extra staff - Continuation of (i) Laboratory Asst.(Sr.) - One.
(ii)Laboratory Asst.(Jr.) - One.

Estimated expenditure

PP-1	Equipment	Nil
PP-2	Staff	Rs 4,000
PP-3	Raw materials	Rs 1,800
PP-4	Contingencies	Rs 800
	Total	Rs 6,600

Pilot Plant Project

PERMANENT MAGNETIC MATERIALS
(New Project)

Nature of the Project

In this project ceramic sintered oxide permanent magnets of various shapes and sizes will be prepared on a pilot plant scale using new compositions already synthesised in our laboratory.

Background of the Project

In the course of our studies of oxidic materials on their structural and magnetic properties suitable compositions for the preparation of sintered permanent magnets have been found and the technique for its preparation using these materials has been standardised. The equipment necessary for the fabrication and testing of the finished magnets is ready. The samples prepared on a laboratory scale have shown promising results. The fabrication as well as properties of this type of magnets have several advantages over the normal cast metal types.

Reasons for Pilot Plant Development

It is now proposed to prepare a large number of permanent magnets using indigenous bulk chemicals. If good characteristics and reproducibility are attainable, samples can be supplied to various instrument manufacturers. There is a great demand for permanent magnets for use in meters, telephones, microphones, loudspeakers, dynamos and several other electrical instruments, and their demand so far has been wholly met by import. After pilot plant production of our magnets it may be feasible to develop the process for industrial exploitation.

Estimated duration of the Project - Two years.

Extra staff required -

(i) Junior Scientific Officer - One.
(Will be responsible for looking after the thermistor pilot plant and others that may come up at a later date).

(ii) Laboratory Assistant (Sr.) - One.

Estimated total expenditure - Rs 20,500 for both the years.

		1960-61	1961-62
PP-1	Equipment Rs	Nil	Nil
PP-2	Staff	7,000	7,500
PP-3	Raw materials	2,000	2,000
PP-4	Contingencies	1,000	1,000
		10,000	10,500

ORGANIC CHEMISTRY

(1) CHEMISTRY OF INDIGENOUS NATURAL ORGANIC PRODUCTS

(a) Steroids

The total steroids from the sugar-cane wax will be studied in detail and after standardising the separation procedures and identification of all the constituents, their conversion into physiologically active steroids will be undertaken.

(b) Terpenoids*

Study of the following forest materials of our country will be initiated with a view to characterising their constituents and, if feasible, converting them into potentially useful derivatives:

(i) Oleoresins from Dipterocarpaceae, Hardwickia pinnata, Pinus species, Ailanthus malabaricum, etc.

(ii) Woods of Cedrela Toona, Thuja species, Erythroxylon monogynum, Lansium annamalyanum, Cedrus Deodara, Cinnamomum cecidodaphne, etc.

(iii) Zingiber zerumbet, Cyperus rotundus, etc.

(iv) Utilisation of Indian turpentine oil : Part I. Chemistry of carenes*

Carenes constitute about 40 per cent of the turpentine oil from Pinus longifolia. Work leading to the conversion of carene into menthol or other useful industrial products will be carried out.

(v) Utilisation of Indian turpentine oil: Part II. Chemistry of longifolene*

Reactions of longifolene will be investigated with the ultimate aim of utilising it as an industrial raw material

---

*Essential Oils Research Committee - schemes shifted from Bangalore.

(vi) Camphor, borneol and related products*

It is planned to work out more modern methods for the conversion of pinenes into these products.

(vii) Menthol from Ajwan oil**

(viii) Chemistry of the essential oil of Artemisia pallens**

(c) Fatty Acids***

(i) Conversion of hydnocarpic and chaulmoogric acids into bicyclic ring systems.

(ii) Conversion of kamlolenic acid into ω-hydroxy acids and dicarboxylic acids of different chain lengths and their conversion into monocyclic systems in which the trans-annular effect will be studied.

(iii) Mechanism of pyrolysis of ricinoleic acid and its esters.

(iv) Utilisation of inedible fatty oils****

Work on the preparation of fatty alcohols from inedible oils and the preparation of quaternary ammonium compounds from these alcohols will be undertaken. The quaternary ammonium compounds will be of interest from the point of view of their bactericidal properties.

(v) Waxes

Work on the chemical composition of sisal wax will be continued.

(vi) Alkaloids

(a) Quinidine from cinchona alkaloids: Work on the conversion of quinine into epimeric quinidine and the development of methods for the separation of quinidine from cinchona febrifuge, will be continued.

*Essential Oils Research Committee - schemes shifted from Bangalore

**To be sponsored by the EORC.

***New project.

****ICOC - CSIR project.

(b) Economic isolation of morphine and other alkaloids from Indian opium.

(vii) Nimbin and nimbidin

Work leading to their structure elucidation is being carried out. These compounds have been shown to possess certain useful pharmacological properties.

(viii) Cashew shell liquid

Attempts are being made to prepare wetting agents, detergents, etc., and to use anacardol and anacardic acid as raw materials for synthesis.

(2) ORGANIC REACTIONS

(a) Biochemical Transformation of Simple and Complex Organic Compounds*

This field, though quite old, has received a tremendous impetus as a result of the potentialities of the micro-organisms discovered in connection with the chemistry of steroids. It is planned to undertake systematic work on this subject with special reference to the effect of the conformation of the substrate on the selectivity of the reaction.

(b) Polyphosphoric Acid in Organic Chemistry**

Work on the action of polyphosphoric acid on γ-, δ- and higher lactones, isomerisation with polyphosphoric acid, polyphosphoric acid induced 1,2-shifts, intermolecular acylation, etc., will be initiated.

(c) Borohydrides

The study of sodium borohydride and other complex metal hydrides as selective reducing agents in organic chemistry will be continued.

(d) Chemistry of Azulenes***

Certain fundamental aspects of the chemistry of azulenes and the use of azulene as a starting material for several new types of organic compounds will be studied

---

*New project.
**New project - Continuation of work initiated at Bangalore.
***Essential Oils Research Committee - Schemes shifted from Bangalore.

(3) ORGANIC CHEMICALS INCLUDING SYNTHETIC DRUGS

(a) Synthetic Drugs

Non-technical notes on 4-hydroxycoumarin and useful compounds derived from it, diethyl stilbestrol, and hexylresorcinol have been submitted to the NRDC. During the next year work on a few other synthetic drugs such as aminophylline and menadione (vitamin K analogue) on which some work has already been carried out will be continued.

(b) Work on the following problems referred to the NCL by a firm will be continued during the year:- (i) Conversion of dichlorobenzene mixture to 1,2,4-trichlorobenzene; (ii) Upgrading the quality of o-dichlorobenzene; and (iii) Ethyl chloride from waste gases.

(4) NATURAL AND SYNTHETIC COLOURING MATTERS

(Most of this work, which is a continuation of the work in progress in the Director's laboratory in Bombay, is being carried out with the aid of research fellows and students).

The programme is broadly identical with that outlined last year and will consist of a continuation of the following problems:- (a) The chemistry of lac dye (sponsored by the Indian Lac Cess Committee). (b) The syntheses of various naturally occurring anthraquinone colouring matters. (c) The constitution of the colouring matters of Garcinia morella. (d) The colouring matters present in the heartwood of Artocarpus integrifolia and other Artocarpus species. (e) Synthetical experiments in the flavone and isoflavone series. (f) The synthesis, constitution, and dyeing properties of anthraquinonoid vat dyes; dyes derived from cyanuric chloride; the constitution of new azoic coupling components.

Pilot Plant Project

ISOLATION OF STEROLS FROM SUGAR-CANE WAX.

(Project approved during 1958-59, but work not commenced)

For several reasons work on the pilot plant extraction of sterols from sugar-cane wax could not be started. It is hoped that within six months or so, we shall be able to reach a stage to start pilot plant work. The following staff was sanctioned for 1959-60 and sanction for it will be necessary during 1960-61:-

Junior Scientific Assistant	- 1
Laboratory Bearer	- 1

Estimated Expenditure during 1960-61

PP-1 Equipment	-	Rs 3,000
PP-2 Staff	-	Rs 4,000
PP-3 Raw materials	-	Rs 2,000
PP-4 Contingencies	-	Rs 500
		Rs 9,500

60-151-

ESSENTIAL OILS

(1) NATURAL ESSENTIAL OILS

(a) Vetiver Oil (Vetiveria zizanioides Stapf).

It is proposed to investigate vetiver oils from five regions with a view to studying the chemistry of their constituents.

Two varieties of this oil are under investigation and several new and interesting compounds have been isolated and characterised. One of these is an eudalenic alcohol with wrong configuration at all centres.

The programme of this year will consist of the investigation of the other components and the examination of the remaining varieties.

All arrangements regarding raw materials, etc., have been completed.

(b) Costus Root Oil (Saussurea lappa Clarke)

The objective is to develop a method for isolating the oil from the roots under as mild conditions as possible and study the constituents and to find out uses for them.

(i) A new method (patented in India and abroad) of extraction of the oil rich in lactones has been standardised.

(ii) Many new compounds have been isolated and some have been characterised.

(iii) Delactonised oil prepared from the whole oil has been found to be acceptable to industry. A foreign order is being finalised.

(iv) Pilot plant extraction of the oil has been completed.

Programme of work for the year 1960-61

(i) Characterisation of the remaining constituents.

(ii) Finding uses for the lactones obtained in high yields.

(iii) Extraction of fresh quantities of oil for further investigation.

(iv) Initiating more foreign consumers to this 'new type oil.

(c) Agarwood Oil (Aquilaria Agallocha Roxb.)

The objective is to extract the oil under very mild conditions so as to preserve the original nature of the constituents and study the components.

Solvent extraction procedure has been standardised. Agarol, the main constituent of the oil, which is responsible for the odour has been completely characterised. Another constituent, a keto-alcohol, has also been characterised.

The programme of this year will be characterisation of other components and synthesis of agarol from easily available raw materials.

Raw materials and chemicals for further investigation are in hand. Work on the synthesis of agarol has been started.

(d) Ginger Oil from Different Varieties

The objective is to study the constituents of different varieties of ginger oil. Work on wild ginger oil (Zingiber zerumbet Smith) has been recently completed.

This year, examination of oils from other varieties will be investigated.

Some of the raw materials and chemicals for this purpose have been collected.

(e) Indian Valerian Root Oil (Valeriana wallichi)

The objective is to study the constituents of this commercially important oil. Samples of the oil have been obtained by solvent extraction under mild conditions.

Systematic examination and characterisation of the components of the oil will be initiated this year.

Further quantities of roots with and without rootlets are being collected.

(f) Morpankhi Oil (Thuia orientalis L.)

The objective is to examine the oil with a view to developing a suitable method for detecting its presence in sandalwood oil,

as it is used as an adulterant. About 4 lb. of this oil have been supplied by the Marketing Officer, Govt. of India. The Agricultural Ministry is interested in its investigation.

Critical examination with the above objective in view will be taken up this year. Initial work will involve characterisation of the constituents.

(g) Oil of Cyperus rotundus and other Varieties

Some work has been done on the oil of Cyperus scariosus partly in collaboration with the H.B.T.I., Kanpur, who sent one of their staff members to the NCL for that purpose.

This year the characterisation of the constituents of other varieties will be initiated.

(h) Indian Nard (Nardostachys Jatamansi DC) - Different Varieties

Jatamansi is well known for its medicinal properties. Objective is to characterise the constituents of its oil. Raw materials are being collected.

(i) Some Oils from the Essential Oil Plantations in the Madras Area

Many commercially important essential oils are being produced in the Cinchona Plantations in Madras in the Annamalai Hills. Since many of these have not been studied systematically, the EORC felt that it would be desirable to investigate their chemistry and entrusted this work to us.

Some raw materials have been already procured.

The Assistant Director, Essential Oils Division, is going to tour the Annamalai Hills area for further information in the near future.

(2) RESIN AND RESIN OILS

Shal (*Shorea robusta*), black dammar (*Canarium strictum* Roxb.), white dammar (*Vateria indica* L.) and *Boswellia serrata* Roxb.

Work on shal resin oil, commonly known as "Chua" oil, is at a sufficiently advanced stage; some of the components have been isolated and characterised. Investigation of the resin also has been initiated.

The work is to be extended to cover the other resins. Genuine samples of all the resins have been collected.

(3) SYNTHETIC AROMATIC CHEMICALS

(a) Macrocyclic Compounds

Muscone, ambrettolide and other related products.

The objective is to develop methods of synthesis of these compounds from easily available Indian raw materials.

Work on civetone and dihydrocivetone, including pilot plant trials, have been completed.

Exaltone and exaltolide are under pilot plant trial.

Laboratory investigation on isoambrettolide and dihydro-ambrettolide has been completed.

Some progress in our work on muscone and ambrettolide has already been made. The work will be further extended during the year.

Some of the results so far achieved on macrocyclic compounds have been covered with patents in India and abroad. The NRDC is considering commercial exploitation of these products.

(b) Menthol from Citronellal

Citronellal, through isopulegol, has been converted to a menthol mixture from which *l*-menthol has been isolated through suitable derivatives.

Further extension of the work for isolation of *l*-menthol in high yields and direct cyclisation of citronellal to isopulegol will be investigated.

Top priority is being given to this problem.

(c) Irone from Turpentine Oil

The object is to synthesise this important perfumery product from pinonic acid.

Conversion of pinonic acid to irone will be investigated.

Substantial quantities of pinonic acid have been prepared by oxidation of pinene obtained from turpentine oil.

(d) Alkyl-γ-lactones

Coconut Aldehyde (γ-Nonalactone)

This is used as a food flavouring agent. Conditions have been found to get the β-γ-unsaturated product by condensing heptaldehyde with malonic acid and its subsequent conversion to γ-nonalactone.

This year the work will be extended further. Raw materials for the same are easily available.

(e) Utilisation of Sandalwood Oil Hydrocarbons

Substantial quantities of α- and β-santalenes have been separated by distillation. Preliminary experiments have been carried out for conversion of santalenes into new useful oxygenated bodies through Prin's reaction and by rupturing the cyclopropane ring in α-santalene.

(f) Other Synthetic Aromatic Chemicals

The Essential Oils Research Committee has assigned to us the syntheses of the following compounds. These are listed below in order of priority assigned by the Committee:-

(i) Phenyl ethyl alcohol
(ii) Nitro musks
(iii) Indole
(iv) Hydroxycitronellal to be synthesised from citronellal using entirely new set of reactions.
(v) Methyl anthranilate from o-nitrotoluene likely to be available from the High Explosive Factory, Kirkee, Poona.
(vi) Geraniol from α-pinene
(vii) Coumarin, heliotropine
(viii) Aldehyde C_{12} (lauric) from Pisa seed oil as the starting material.
(ix) Aldehyde C_{12} (M.N.A.)
(x) Skalole
and (xi) Aldehyde C_{16}.

Work on these will be undertaken in the above sequence, depending, however, on the availability of material, staff, etc.

Pilot Plant Project

EXALTONE - EXALTOLIDE

This project was sanctioned last year. Due to various reasons necessary staff could not be recruited. The work was partly managed with difficulty with the existing staff and hence is at present in an incomplete state. Steps have been taken for recruitment of the staff. Its renewal during 1960-61 will be necessary.

Financial Requirements

Pay (including allowances)	
2 Junior Scientific Assistants 290 x 12 x 2	- Rs 6,960
1 Senior Laboratory Assistant 205 x 12	- Rs 2,460
	Rs 9,420
Chemicals and contingencies	- Rs 4,000
	Rs 13,420

Pilot Plant Project

DIHYDROJASMONE - ISOJASMONE

This project was also sanctioned last year, but due to non-availability of staff it could not be completed. Two Laboratory Assistants have now been appointed. A post of Junior Scientific Assistant is still lying vacant and is expected to be filled up in the near future, for which adequate steps are being taken.

Renewal of the project during this year would be necessary.

Financial Requirements

Pay (including allowances)	
1 Junior Scientific Assistant 290 x 12	Rs 3,480
1 Senior Laboratory Assistant 205 x 12	Rs 2,460
	Rs 5,940
Chemicals and contingencies	Rs 1,500
	Rs 7,440

PROVISIONAL APPROVAL OF PROJECTS

(1) It may be necessary for us to extract one ton of costus root on a pilot plant scale again during the current year.

Financial Requirements

Pay (including allowances)

1 Junior Scientific Assistant 290 x 12	- Rs	3,480
2 daily wage labourers for 200 days 2x200x2	-Rs	800
	Rs	4,280

Chemicals and Raw Materials

Costus root one ton at the rate of Rs 350/- per maund	- Rs	9,800
Solvent pet. ether 600 gallons at the rate of Rs 6/- per gallon	- Rs	3,600
	Rs	17,680
Chemicals and contingencies	Rs	500
Total		18,180

(2) It may also be desirable to carry the synthesis of dihydroambrettolide and isoambrettolide using aleuritic acid through pilot plant stages. Provision for staff and contingencies may be made in this regard.

Financial Requirements

Pay (including allowances)

1 Junior Scientific Assistant 300 x 12	Rs	3,600
1 Senior Laboratory Assistant 180 x 12	Rs	2,160
	Rs	5,760
Apparatus and equipment -	Rs	1,000
Chemicals and contingencies -	Rs	5,000
Total	Rs	11,760

BIOCHEMISTRY

(1) STEROID SYNTHESIS

The objective of this work is the production of aldosterone and other clinically important steroid hormones. The chief line of investigation is the conversion of steroidal alkaloids to aldosterone by chemical and microbiological methods.

(2) BIOSYNTHESIS AND METABOLISM

This project will consist of fundamental work on the biosynthesis and metabolism of biochemically important materials such as the pectic substances and proteins. The mechanism of pectin synthesis and the enzymes which degrade pectin have not hitherto been systematically investigated. A study of the biochemistry of these compounds will be of fundamental interest and will also be of practical value, as in the production of pectinases.

(3) MICROBIOLOGY

(a) National Collection of Industrial Micro-organisms

The culture collection contains about 1000 yeasts, bacteria and fungi of importance for research and industry, which are supplied to institutions in India and abroad.

(b) Microbiological Production of Sulphur

This is a long-term applied research project for the production of sulphur by the microbiological reduction of sulphates. Laboratory scale work on a semi-continuous process using sewage sludge was completed last year and studies on a 50-litre fermenter will be undertaken.

(c) Screening and Mutation Work and New Fermentations

The programme of work consists of screening of new isolates and mutant strains of industrially useful micro-organisms, especially those for the production of enzymes, organic acids and terpenes, and studies on their nutrition and metabolism. Work is in progress on the production of protease and the microbiological transformation of terpenes. The isolation and cultivation of plant cells by the submerged technique and the study of their metabolism has been undertaken in order to study the production of industrially useful substances by this technique.

POLYMER CHEMISTRY

(1) INDUSTRIAL PROBLEMS

(a) Ethylene Polymers of Low Molecular Weight

In order to develop a suitable catalyst system for polymerising ethylene mainly to hexamer, various catalysts including Friedel-Crafts type have been tried and some more are yet to be studied. The liquid polymers obtained will be used to alkylate benzene prior to its sulphonation to yield surfactants.

(b) Expanded Polystyrene Plastics

Preliminary experiments on the preparation of expandable polystyrene, using low boiling non-solvents as a blowing agent, were completed. The extension of this work, employing the suspension polymerisation process under pressure has been held up for want of a suitable pressure kettle. This has since been fabricated and is under test. The technique of fabrication, foaming the expandable polystyrene beads thus produced into sheets and other expanded articles, will be developed.

(c) Polyurethanes

A suitable foam-producing machine has been designed, fabricated and successfully used for making 12" x 12" x .2" sheets of polyester-urethane flexible foams. These studies will now be extended to the use of castor oil. The technical implications and other related matters for the economic production of flexible foams in the country will be examined.

With certain modifications this machine will be used for making large size polyester-urethane rigid foams. The technique for making polyurethane base printer's rollers will also be investigated.

(d) Polymerisation of Vinyl Chloride (PVC Project)
(in collaboration with Chem. Eng. Div.)

Systematic work on this project will be carried out as soon as the monomer becomes available from the pilot-plant now under assembly in the Chemical Engineering Division.

(e) Resins for Low Pressure Laminates
(New Project)

The manufacture of glass fabric reinforced laminates using imported resins has been recently licensed in the country. Such low pressure laminates are finding increasing uses in the fabrication of automobile bodies, boats, rifle furniture and other armament accessories, etc. It is proposed to initiate studies on the preparation of suitable polyester resins for this particular application.

(2) FUNDAMENTAL STUDIES IN POLYMERS

(a) Polymerisation and Copolymerisation

(i) The investigation of hydrazine initiated vinyl polymerisation in aqueous medium under high vacuum conditions has provided evidence regarding the nature of the initiation reaction. This investigation will be further pursued in order to evaluate the kinetic and Arrhenius parameters.

*(ii) It has earlier been postulated that the influence of the dielectric nature of the medium is discernible on copolymerisation reactions, as shown by certain binary systems with one of the components as a strongly polar monomer. This fact will be experimentally verified through study of composition and reaction rates of systems with polar monomers.

(iii). The graft-polymerisation of methyl methacrylate with natural rubber latex will be further studied in order to determine the relative contribution of the reaction of initiator with rubber as well as the reactions of polymer radicals with rubber.

(iv) A study of the kinetics and mechanism of polymerisation of olefins using Ziegler type catalysts based mainly on zirconium compounds and metal alkyls will be undertaken.

(b) Molecular Structure and Physical Properties of Polymers

(i) The studies on the anomalous behaviour of polymer solutions at high dilutions will be continued.

*(ii) A new method proposed for determining the molecular weight of polymers from viscosity measurements will be verified from extensive experimental data.

---

*New project.

(3) ION-EXCHANGE

(a) Syntheses

(i) Cation-exchange Resins - Preliminary work on the preparation of a sulphonated polystyrene cation-exchange resin has been carried out. Further studies to determine the optimum conditions for sulphonation of the polymer will be carried out. Resins of various degrees of cross-linking will be prepared and their physico-chemical behaviour studied.

(ii) Anion-exchange Resins - From melamine, formaldehyde and hexamine a promising anion-exchange resin has been prepared in small lots. It is planned to extend this to a larger scale.

Reaction of polyvinyl chloride with pyridine will be further studied, and besides pyridine other amines will also be investigated.

(iii) Membranes - These membranes could not be synthesised due to pressure of other work. It is now proposed to prepare both cation and anion type membranes with rubber base as per our earlier patent specification.

(b) Application of Ion-exchange Technique to Alkaloids

No work has been carried out due to priority of other work. The possibility of using the ion-exchange technique for the recovery and/or fractionation of various alkaloids will be examined.

(c) Electrodialysis - Electrodialysis cells will be constructed with imported and locally fabricated membranes and the following studies will be conducted:-

Demineralisation of saline solutions. Earlier work will be extended to other membrane systems also.

Fundamental studies involving transport number determination and other physico-chemical properties.

Purifications effected by using one type of membrane only.

(4) RUBBER

(a) The following projects approved by the Indian Rubber Manufacturers' Research Association (IRMRA) will be continued:

(i) Rubber-base Adhesives

To make indigenous multi-purpose adhesives like pliobond, Bostike, etc.

(ii) Blowing Agents for Microcellular Rubber

New indigenous blowing agents will be formulated and tried in sponge mixes, particularly for closed cell structure.

(iii) Identification and Estimation of Antioxidants in Rubber

To evolve rapid, accurate methods for the purpose mentioned in the title.

(iv) Evaluation of Indigenous China Clay and Whiting for Rubber Use

To make available to the Indian rubber industry standard grades of hard and soft clay and whiting comparable to imported brands.

(b) Work on the following new projects already approved by the IRMRA will be taken up:

*(i) Block Polymers from Solid Natural Rubber

To modify the natural rubber molecule chemically for achieving better physical and chemical properties (e.g. higher resistance to abrasion, chemicals, solvents, ageing, etc.).

*(ii) Studies in Crystallinity of Transparent Rubber

Determination of the extent of crystallinity in transparent rubber, unstrain and strained, will be tried by the light scattering method, in collaboration with the Physical Chemistry Division.

*(iii) Lignin and Wood Flour in Rubber Compounding

To study the effects of various grades of lignin and wood flour available as industrial wastes in rubber compounding.

---

*New project.

CHEMICAL ENGINEERING

(1) CHEMICALS FROM CASTOR OIL

(Partly financed by the ICOC; started - Dec. 1957; continued from 1959-60)

A 10-gallon bench unit reactor was designed, fabricated and installed. Investigations covering 12 experiments with 20 lb. oil per batch have been carried out, trying many modifications in the process. Sebacic acid (98 per cent pure) has been obtained in 58-59 per cent theoretical yield. The octanol, though obtained in similar yields, however contained appreciable amounts of octanone, and this aspect is now being intensively investigated. In addition to the above, 14 runs with 2 lb. oil per batch were also carried out in the smaller 3-gallon reactor.

Work on the following aspects will be carried out:-

(a) Reasons for high octanone yield in the octanol and process conditions to minimize the octanone content.

(b) Investigation on the recovery and utilization of various byproducts.

(c) A detailed process study of the reaction of the alkali scission of technical ricinoleic acid with catalysts, various kinds of diluents, etc., and the effect of the reaction.

(d) Development of a continuous system for the alkali scission of castor oil, if possible.

(2) PROCESS STUDIES OF CATALYTIC HYDROGENOLYSIS WITH SPECIAL REFERENCE TO PRODUCTION OF CETYL AND OCTADECYL ALCOHOL

(Continued from 1959-60; started- July 1959)

Extensive plans for suppression of water evaporation from large reservoirs by use of suitable reagents forming monomolecular films are under consideration in India. One such substance used is the 50:50 cetyl-stearyl alcohol mixture. Preliminary investigations on the process for its production from cottonseed oil have been already started as a part of this problem. Work conducted so far related mainly to high pressure hydrogenolysis of the complete mixture of the cottonseed oil fatty acid esters. The results obtained indicate a fairly good conversion of esters to the corresponding saturated fatty alcohols, as evidenced by the saponification, iodine and hydroxyl values. Further work on the following lines will be carried out:-

(a) Processing and upgrading the feedstock in order to obtain an enhanced output of the desired product per unit volume of the high pressure reactor. (b) Detailed investigations on the hydrogenolysis of various feedstocks in available batch pressure reactors of varying capacities and designs up to a maximum of 5 litre total volumetric capacity. (c) The treatment of the crude product obtained after hydrogenolysis to finally yield the desired 50:50 mixture of C_{16} and C_{13} alcohols. (d) Investigation of the hydrogenolysis reaction on the continuous high-pressure system after the equipment is received.

The bench scale investigations are aimed at obtaining optimum conditions for larger scale trials. It is proposed to work this process as a pilot plant project after the receipt of the 75-litre high pressure autoclave from overseas, the necessary procurement action for which has been initiated.

(3) ETHYL ACETOACETATE

(Started - September 1958)

Ethyl acetoacetate is one of the important intermediates for dyes. Its manufacturing process consists of two steps: (a) preparation of sodium ethoxide and (b) condensation of ethyl acetate in presence of sodium ethoxide.

Work carried out during 1959-60

Experiments have been carried out to obtain sodium ethoxide in lots of 1½ lb. from sodium hydroxide and ethanol. The conditions for the conversion of sodium hydroxide to sodium ethoxide up to 90-93 per cent yield have been set up. Experiments to produce ethyl acetoacetate have been also carried out. The yield of ethyl acetoacetate obtained so far is about 65 per cent based on sodium hydroxide.

Work proposed during the year

(a) Experiments will be conducted to improve the yield of ethyl acetoacetate. (b) Study of recovery, purification and re-utilization of the excess ethyl acetate recovered during the condensation will be carried out. (c) Further study of the design of the reactor system to prepare sodium ethoxide by using azeotropic distillation will be conducted. (d) Experiments to prepare ethoxyacetic acid required in the vitamin B_6 pilot plant project will be carried out.

(4) CHLORINATION OF ILMENITE AND PRODUCTION OF RUTILE TITANIA PIGMENT

A laboratory process has been evolved in which the bulk of the iron present in ilmenite is first removed by the byproduct hydrochloric acid generated at a later stage, and the upgraded ore chlorinated in a static bed in an electrically heated silica tube (3" dia.) under established conditions. On hydrolysis followed by calcination of the titanium tetrachloride under conditions worked out, pure rutile type titania of pigment quality is obtained. The acid obtained on hydrolysis is used directly for the extraction of iron from a second batch of ilmenite of equal size.

The process will be examined with a view to pilot plant investigation in two stages, following a suggestion from the Secretary, CSIR, to undertake the investigation at the laboratory's own cost.

The use of titanium dioxide as a superior, non-toxic white pigment is now well established in the country, both of the anatase and rutile types, the latter being sometimes preferred owing to its better covering power and non-chalking characteristics. Large quantities of titania white are imported every year, though one plant in the country is producing anatase titania (1,800 tons/year) by a sulphuric acid process. Imports of rutile titania white alone amounted to 5608 and 7085 cwt. in 1957 and 1958, valued at Rs 8.3 and Rs 9.6 lakhs. Consumer paint industries have written to the Government of India expressing their preference for the rutile type titania pigment.

An intermediate product of the process, titanium tetrachloride, finds use as a defence chemical for the production of smoke screens, and is also a raw material used for the manufacture of titanium metal and titanium organics.

(5) A STUDY OF THE RATE CONTROLLING STEPS AND MECHANISM OF THE KINETIC DEHYDRATION OF ETHANOL

(New problem)

Kinetic dehydration of ethyl alcohol to produce ether and ethylene is an important reaction, the mechanism of which has not been conclusively established yet. Due to the difficulties in the analysis of the product gases, which contain the highly volatile ethyl ether, the process kinetics of this catalytic reaction has not yet been thoroughly investigated. In investigations carried out in India and abroad, either higher temperatures have been used to avoid the formation of ether or higher alcohols have been used for similar studies.

Improved methods of chemical analysis have been recently suggested and using either these methods or with the help of vapour phase chromatography, it should be possible to investigate the problem in detail. An integral reactor with suitable alumina as catalyst will be used to study the effect of temperature, space velocity, feed rate and length to diameter ratio of the reactor on conversion. Attempts will be made to elucidate the mechanism with the help of this experimental data.

(6) STUDIES ON RELATIVE RESISTANCES IN MASS TRANSFER OPERATIONS
(CSIR Junior Research Fellow)

(New problem)

In a two-phase system, movement of material from one phase to the other was until recently generally based on the theory of two quiescent films of the two fluids with a common interface. It was further assumed that only the two films offered the resistance in the mass transfer and that the concentrations in the two films were such that there was equilibrium at the interface. Recent work done elsewhere, however, indicates that the amount of material actually transferred is somewhat different from that given by the two-film theory, thus indicating a probable additional resistance of some form perhaps at the interface. There is conflicting opinion on this aspect in the recent literature. Fundamental investigations will be carried out to gather further evidence and to determine whether any such significant interface resistance occurs and, if so, its relative magnitude under various circumstances.

(1) UTILIZATION OF INDIGENOUS CELLULOSIC RAW MATERIALS FOR THE PRODUCTION OF RAYON GRADE PULP

(Project started - August 1958; continued from 1959-60)

Nature of the Project

This project is a comprehensive investigation for the production of rayon grade pulps from indigenous cellulosic raw materials with attention to both the applied and fundamental aspects.

Present State of Development

Dendrocalamus strictus bamboo was investigated for pulping by the prehydrolysis-sulphate method and the experimental pulps were evaluated by chemical analysis and viscose making. Thirty-six experiments, comprising water-prehydrolysis and sulphate cooking followed by multi-stage bleaching, were carried out on a laboratory scale using 3 kg. of bamboo chips per batch. Yields of pulps, conforming to the best average analysis (95 per cent alpha-cellulose, 2 per cent pentosans and 900 degree of polymerization), averaged to about 28 per cent. However, silica and iron contaminants of these pulps were high. Some of these runs will be tried under optimum conditions in a stainless steel 20 cu. ft. kier. Investigations on the process "post-chlorination hydrolysis..." and on the role of parenchyma cells of bamboo in pulping and viscose production have been started. Pulping investigations on bagasse were deferred presently as mentioned in the annual report for 1958-59.

Units for laboratory processing of about 600 g. pulp per batch to viscose were fabricated in the NCL Workshop to enable preliminary investigations and evaluation of viscose. Some runs on viscose preparation have been made. A survey, by chemical analysis, of the various waste cellulosic raw materials, is being continued and three raw materials have been analysed.

Practically all the major items of equipment for the pilot plant have been indented.

Proposed Investigations for 1960-61

A detailed laboratory study of water and acid prehydrolysis followed by sulphate cooking of two species of bamboo with a view to obtaining maximum alpha-cellulose and optimum filterability of the viscose will be continued. The role of parenchyma in pulping and xanthation will also be studied. Other pulping techniques to lower pentosan and silica will be taken up for study.

The laboratory results of pulping will be reproduced on a pilot plant scale and pulps will be processed to viscose and further to the yarn stage in the Emil Blaschke plant expected in March 1960. In addition, work on the next raw material - bagasse - will be started. Scouting experiments on some other raw materials may be done.

A survey of the various raw materials by chemical analysis will be continued.

Expected Duration of the Project - Five years at least.

Staff Requirements

	Sanctioned	Recruited
Senior Scientific Officers	2	2
Junior Scientific Officers	2	2
Senior Scientific Assistants	1	Nil
Junior Scientific Assistants	6	4
Mechanic	1	1
Lab. Attendants	6	4

After installation of the pilot plant, full pilot plant staff will be recruited.

Estimated Expenditure during 1960-61

PP-1 Equipment	-	Rs 1,00,000
PP-2 Staff	-	Rs 61,500
PP-3 Raw materials	-	Rs 20,000
PP-4 Contingencies	-	Rs 5,000
		Rs 1,86,500

(2) POLYVINYL CHLORIDE FROM ALCOHOL AND CHLORINE

(Date of commencement- January 1957; continued from 1959-60)

Nature of the Project

A process for producing PVC from alcohol and chlorine, already worked out on a bench scale, is being tried on a pilot plant of capacity 8-10 lb. of V.C.M./hr. and about 100 lb. of PVC/batch.

Present State of Development

Design equations for enabling scale-up of the fluidized reactor to produce ethylene from alcohol using a particular bauxite as catalyst were worked out from the extensive data gathered from the pilot plant.

The performance of the high capacity reactor to produce ethylene dichloride has been studied in detail without using a catalyst. The effect of catalyst on conversion is being presently investigated. A patent for the process has been filed.

Preliminary tuning-in runs were carried out on stage III to test the pilot plant assembly to produce vinyl chloride which was further processed and liquefied. On the basis of these runs, some changes in the design of reactor and furnace are being made. The equipment for stage IV for purification and polymerization of the monomer has been installed and tested for leakages under pressure.

Programme for 1960-61

Detailed and systematic investigations on the performance of the vinyl chloride reactor will be carried out with a view to arriving at a suitable design equation. The effects of space velocity, temperature and temperature-gradient inside the reactor will be studied with special reference to carbon deposition inside the reactor tubes. One more experimental reactor of the conventional heat exchanger type will be set up for comparative study of its performance.

The monomer produced will be liquefied and distilled under pressure and the purified monomer will be polymerized according to the conditions standardized in bench scale work. Studies on polymerization will be gradually raised from 5 gallon to 50 gallon scale per batch. Conditions for obtaining polymers of various specifications will be standardized.

Duration of the Project

It is hoped to make considerable progress in stages III and IV during 1960-61.

Staff

As ethylene dichloride will have to be produced in large quantities from time to time for its consumer-acceptance tests, the appointment of one more Laboratory Assistant (Sr.), in addition to the present staff, is necessary.

The continuation of the existing staff already appointed in 1957-58 is also required. The total staff required is as follows:

Senior Scientific Assistant	- 1
Laboratory Assistant (Sr.)	- 3 (2 existing and 1 to be appointed)
Lab. Attendants	- 2

Estimated Expenditure

	Rs
PP-1 Equipment	- 10,000
PP-2 Staff	12,250
PP-3 Raw materials	5,000
PP-4 Contingencies	5,000
	32,250

(3) CARBON TETRACHLORIDE AND CHLOROFORM FROM SEWAGE GAS METHANE AND CHLORINE

(Started - December 1958; continued 1959-60)

Nature of the Project

To study and standardize the process conditions on a pilot plant with a capacity of 5 lb./hr. of combined carbon tetrachloride and chloroform from the chlorination of methane in a fluidized bed reactor.

Background of the Project

Work on a bench scale unit was carried out from Dec. 1952 to Nov. 1954. The proposal to put up a pilot plant was presented to the Executive Council in 1958-59. The pilot plant work was started in December 1958.

Present State of Development

The pilot plant with a 4" dia. reactor has been installed and a number of successful runs have been taken with 70-80 per cent yields of carbon tetrachloride product. The output of the continuous unit is being gradually increased by suitable adjustment of variables.

A bench scale unit has also been installed for detailed studies on some process variables as a guide for selecting the optimum range for investigations on the pilot unit.

Proposed Work in 1960-61

Attempts will be made to run the pilot plant for the production of both carbon tetrachloride and chloroform under the most suitable conditions indicated from the studies carried out on the bench scale unit. The externally heated reactor will also be installed and put into operation. Studies on the process variables for the production of carbon tetrachloride and chloroform on a bench scale will be continued.

Expected Duration of Project

The project is expected to be completed by about March 1961, unless a production unit has to be operated for special purposes.

Staff

Senior Scientific Assistant	-	1
Senior Lab. Assistant	-	1
Lab. Attendant	-	1

Estimated Expenditure for 1960-61

		Rs
PP-1 Equipment	-	15,000
PP-2 Staff	-	6,800
PP-3 Raw materials	-	3,500
PP-4 Contingencies	-	2,000
		27,300

(4) ETHYLENE OXIDE AND ITS DERIVATIVES
(Started - March 1958; continued from 1959-60)

Nature of the Project

The production of ethylene oxide by the chlorohydrin route and pilot plant investigations.

Background of the Project

A large number of very useful chemicals manufactured from ethylene oxide are being imported. The relative merits of the two routes to ethylene oxide have already been indicated. Since ethylene chlorohydrin has wide applications by itself, as is evident from the enquiries made by certain commercial firms in Bombay, this route has been chosen for study.

Present State of Development

Bench scale investigations on the production of ethylene chlorohydrin (ECH) on a 1" single column reactor were continued and have been completed. Experiments were also carried out on a larger 2½" dia. reactor and the results obtained were in conformity with those on the 1" unit. The chlorohydrin solutions so obtained were concentrated by fractionation and supplied to the Organic Chemistry Division for the synthesis of N-methyltaurine and to Bombay.

A pilot plant for producing 10 lb. of ECH per hr. was designed and its fabrication is nearing completion in our Workshop. A small bench unit for ethylene oxide is being assembled.

Programme for 1960-61

Investigations on the preparation of ethylene oxide on the bench unit and completion of the integrated pilot plant for anh. ECH and ethylene oxide. Also investigation for the production of one or two derivatives.

Staff

The following staff will be required.

Junior Scientific Assistant	- 1	(will be appointed this year)
Lab. Assistant (Sr.)	- 1	(already appointed)
Lab. Attendant	- 1	

Estimated expenditure

		Rs
PP-1	Equipment	- 15,000
PP-2	Staff	- 6,500
PP-3	Raw materials	- 5,000
PP-4	Contingencies	- 2,000
		28,500

(5) HEXACHLOROETHANE FROM ETHYLENE AND CHLORINE

(Continued from 1959-60; date of commencement - June 1958)

Nature of the Project

To study the process conditions on a pilot plant with a capacity of 5-6 lb. per hour of HCE in a static bed.

Background of the Project

About 100 tons of hexachloroethane are imported yearly. The object is to establish the manufacture of hexachloroethane on a commercial basis using Indian raw materials to attain self-sufficiency.

Present State of Development

Work on a bench pilot plant, both in a glass reactor and single monel tube reactor, has been completed satisfactorily. The monel metal was found to be practically unaffected even after 100 hrs. of run. The products obtained were tested and were found acceptable by the TDE (ME), Kirkee. The active life of catalysts has been determined.

The pilot plant work could not be started for the following reasons:-

(a) The nickel reactor is on order and is being fabricated by a Calcutta firm.

(b) Activated coconut shell charcoal, on which our work was based, was not made available to us by the Cordite Factory, Aruvankadu, till late in 1959.

Proposed Work for 1960-61

The following staff will be required for the runs.

Lab. Assistant (Sr.) - 2

Lab. Attendants - 2

The Lab. Attendants and the Lab. Assistants will have to be appointed for six months.

Estimated Expenditure during 1960-61

		Rs
PP-1 Equipment	-	5,000
PP-2 Staff	-	3,500
PP-3 Raw materials	-	2,000
PP-4 Contingencies	-	1,000
		11,500

(6) VITAMIN B_6

(Started - August 1958; continued from 1959-60)

Nature of the Project

The object of the work is to undertake pilot plant production of vitamin B_6 based on the process worked out on a laboratory scale in the Organic Chemistry Division. This process consists of ten primary stages, each of which is to be studied by carrying out several batch preparations based on the production of 250 g. of the final product.

Background of the Project

Pyridoxine hydrochloride (vitamin B_6) forms an important constituent of the vitamin B complex used widely for therapeutic purposes. The present demand in India for this valuable product is totally met by import which amounts to about 2000 lb. and worth nearly Rs 8.5 lakhs (1958). The design, costing and economic data required for erection of such a unit can only be obtained by pilot plant investigation of the process.

Present State of the Problem

The first four stages in the production of vitamin B_6, which consist of the preparation of (a) ethoxyacetic acid, (b) ethoxyethyl acetate, (c) ethoxyacetylacetone, and (d) cyanacetamide have been satisfactorily carried out on a pilot plant scale, and the necessary data have been collected. Work on several batches was carried out in each stage in order to establish the reproducibility of the results.

Proposed Programme for 1960-61

The remaining six stages of the process will be carried out on a pilot plant scale and the required process and design data will be collected.

A plant for the production of 10 lb. of vitamin B_6 per day will be designed on the basis of the data collected during the pilot plant investigation.

Duration of the Project

This project is likely to be completed in 1960-61 provided the glass-lined autoclave, for use in stages 8 and 9 and on order, is received in time from Germany.

Staff

Senior Scientific Assistant	- 1
Junior Scientific Assistant	- 1
Senior Laboratory Assistant	- 1
Lab. Bearer (Skilled)	- 1

Estimated Expenditure

PP-1 Equipment	-	Rs 15,000
PP-2 Staff	-	Rs 9,800
PP-3 Raw materials	-	Rs 8,000
PP-4 Contingencies	-	Rs 4,000
		Rs 36,800

(7) ISOPROPYL ALCOHOL FROM ACETONE AND HYDROGEN

(Date of commencement - April 1959;
continued from 1959-60)

Nature of the Project

A suitable catalyst for the production of isopropyl alcohol from acetone and hydrogen has been developed and process conditions for its use found out on a small bench scale unit in the Organic Chemistry Division. The pilot plant investigations, now being carried out, will deal with the design of the reactor, and of the various other accessories such as cooler-condenser to enable the integrated design of a full-size unit and also produce larger quantities of isopropanol for further tests.

Present State of Development

Experiments have been carried out initially on a bench unit to decide on the choice between the fluidized or fixed bed reactor. Subsequently a pilot plant using the fixed bed (capacity - about 5 lb. of isopropyl alcohol/hr) has been designed, fabricated and installed. Preliminary experiments have been carried out and some modifications of the unit have since been done. Systematic investigations are now being carried out.

Programme for 1960-61

Regular runs will be carried out to investigate the effect of different process variables, and later continuous runs will be done to test the life of the catalyst. Work on the effect of shape of the catalyst will be carried out. Data on the other auxiliary units used will be collected, whenever necessary, for design purposes.

Duration of the Project

Till 31st December 1960.

Staff

The following staff sanctioned for 1959-60 is required for 1960-61:-

Junior Scientific Assistant	- 1 (to be appointed
Lab. Assistant (Sr.)	- 1 (joined in early Jan. 1960
Lab. Bearer (Skilled)	- 1 (existing)

Estimated Expenditure

		Rs
PP-1 Equipment	-	12,000
PP-2 Staff	-	2,600
PP-3 Raw materials	-	3,000
PP-4 Contingencies	-	1,500
		19,100