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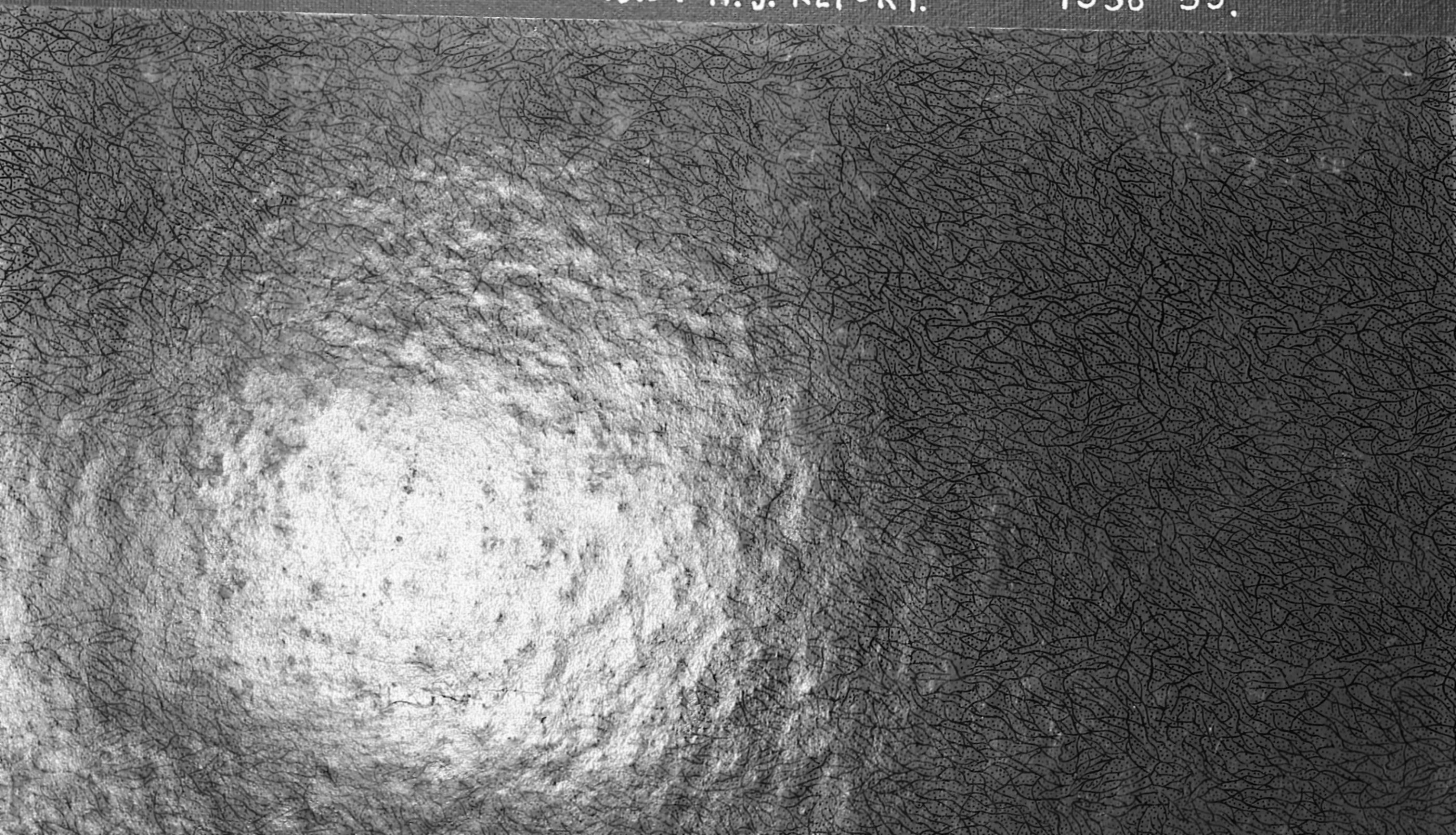

NATIONAL CHEMICAL LABORATORY

POONA.

HALF-YEARLY REPORT (April - September 1958)

C O N T E N T S

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BIOCHEMISTRY

STEROID SYNTHESIS

About 20 different strains of micro-organisms were examined with respect to their ability to oxidize the side chain of cholesterol. A fungus (isolate No. 17-10) was found to produce, among other steroid materials, a compound which resembled progesterone on chromatography with two different solvent systems. The characterisation of this compound is in progress.

The suitability of the steroidal alkaloids from "kurchi" bark for the synthesis of steroid hormones is under investigation. A study of the neutral steroids from the bark showed that the non-saponifiable fraction of the oil from kurchi bark contained at least four compounds giving positive Liebermann reaction. Of these two have been obtained in pure form and identified as lupeol and β-sitosterol.

The ability of some fungal isolates to oxidize terpenoid hydrocarbons, such as pinene, was investigated. It was found that pinene was rapidly metabolized by these organisms in shake cultures and yielded small quantities of sweet-smelling polar material.

SULPHUR METABOLISM

Experiments on the semi-continuous production of H_2S from inorganic sulphate, using sewage sludge, were continued and reproducible yields of sulphide were obtained. When half the total sulphate was supplied as sea-bitterns, the yield of sulphide was only 0.25% (w/v) daily.

Hydrogenase from D.desulphuricans was purified by ion-exchange and cellulose chromatography to a specific activity of about 12 x 10^6 ul. H_2 per mg. N per hour. The purified enzyme was free from cytochrom impurities and required iron salts for reduction of methylene blue, indicating that non-hemin iron is required for enzyme activity. The sulphate-activating enzyme was purified about ten-fold and obtained free from pyrophosphatase.

PECTIN METABOLISM

The isolation of uridine nucleotides, which are required for the preparation of UDP-galacturonic acid, was undertaken and about 75 mg. of mixed nucleotides were obtained from 5 kg of mung-bean seedlings. D-galactose-1-phosphate was synthesised from D-galactose and the preparation of D-galacturonic-1-phosphate is under investigation.

PROTEIN CHEMISTRY

The two peptide fractions obtained from the peptic digest of elastoidin were further purified by paper chromatography. They were found to have the following amino acid composition :

Fraction I - Asp_2, Thr_3, Ser_3, Glu_9, $Ileu_2$, L

Fraction II - Asp_3, Thr_2, Ser_3, Glu_9, Leu_1

Glutamidol, aspartidol, isoleucinol and leucinol were prepared for comparison with the corresponding compound obtained from the C-terminal residues.

NATIONAL COLLECTION OF INDUSTRIAL MICRO-ORGANISMS

(a) Maintenance

The routine subculturing and maintenance of yeasts, bacteria and fungi in the collection were carried out. Cultures were regularly made available for projects on the production of amylase, sorbose, riboflavin and for microbiological assays. During this period 87 cultures were despatched to various institutions in India and U.S.A. Eighteen cultures were added from foreign collections.

(b) Screening for new organisms

(i) Vitamins

Screening for high vitamin B_{12}-producing organisms was continued and 16 actinomycetes and 6 propioni-bacterium strains were selected for further study. Three cultures, which gave yields greater than 2.0 mg. B_{12} per liter, are being further investigated in shake flasks and fermentors for vitamin production and for the isolation of better strains of mutants.

Indigenous corn steep liquor was found to give the same yield of riboflavin as imported material. Supplementation with extracted groundnut cake, and continuous addition of carbohydrates during fermentation did not result in significantly higher yields of the vitamin.

(ii) Citric acid

The effect of the inoculum and depth of medium on acid yield was studied. Spores derived from different media and after storage at 0^{o} gave similar yields of citric acid, but lyophilization of spores had an adverse effect. Satisfactory yields were obtained at a medium depth of 2 to 3 cms. but not at greater depths. Several mutants and other strains of A.niger are being tested for their ability to ferment greater depths of media with satisfactory yields.

PILOT PLANT PROJECTS

BACTERIAL DIASTASE (in collaboration with the Chemical Engineering Division)

Eight fermentations were carried out in the 40-liter fermentor and the processing of the fermented medium was studied in order to standardise the procedure for the isolation of a stable amylase preparation. Recoveries of 85 to 90% of the enzyme from the clarified fermentated liquor were reproducibly obtained. A syatematic study of the fermentation conditions resulted in a reduction of the time of fermentation from 2 days to 1 day, replacement of peptone, which has to be imported from abroad, by indigenous materials, and an over-all increase in yield of the enzyme by 60 per cent. The yield of enzyme was the same in Sparger or Vortex types aeration. The loss in activity on storage of the enzyme preparations for four months at room temperature was found to be negligible. The stability of liquid concentrates with different preservatives is being studied.

Studies on the chemical engineering aspects of the process for the production of bacterial diastase are in progress. Initially the work is being carried out on small bench units. Preliminary data on the filtration characteristics of the fermented liquid have been carried out. Arrangements are now being made for pilot plant work to collect engineering and process data for the design of a commercial plant.

TRANSFUSION GELATIN

The preparation of ten batches of transfusion gelatin, which was undertaken for the determination of the reproducibility of the process on a pilot plant scale, has been completed. During this period 5 more batches of ossein and 7 more batches of transfusion gelatin were prepared. A pooled lot prepared from the first three batches was tested at Armed Forees Medical College, Poona, on dogs, rabbits, rats and guinea pigs, and was found to cause no tissue damage or retention and deposition in the tissues. Clinical trials are in progress.

VITAMIN C SYNTHESIS (in collaboration with the Chemical Engineering Division)

About 100 lbs. more of sorbose were prepared by a modified procedure for the recovery of sorbose from the fermented medium, which resulted in increased recoveries. A detailed study of the oxidation of diacetone sorbose to diacetone ketogulonic acid was undertaken in order to define precisely the experimental conditions required for controlling the high exothermic reaction and obtaining reproducibly higher yields. Under optimum conditions the yields were increased by 10 to 12% on a laboratory scale.

In the Chemical Engineering Division work has been carried out in three directions : (1) detailed design of a commercial unit with a capacity of 3 cwts of vitamin C per day, (2) investigations on problems such as solvent recovery efficiencies, heat transfer data, crystallisation data, and experimental determinati of some physical data, and (3) modification of some steps in the process with a view to improve the yields in various stages. The design of most of the units has been completed.

CHEMICAL ENGINEERING

SEBACIC ACID AND OCTANOL-2 FROM CASTOR OIL
(in collaboration with the Organic Chemistry Division)

In continuation of the work done earlier in the Organic Chemistry Division, investigations have been taken up on the bench scale. A 3-gallon copper reactor with necessary fittings has been used for the preliminary work. A few runs have been made to study the effect of the composition of the charge, temperature, etc., on the yield of octanol and sebacic acid. Efficient methods for the recovery of sebacic acid from the solid residue and for the separation of 2-octanol from the liquid bye-products are being investigated. The bench unit reactor was later somewhat modified to increase the heat-input and to feed the superheated steam. Based on the preliminary experiments in the small reactor, a 10-gallon batch reactor has been designed for fabrication in the NCL workshop. A suitable heat transfer oil system for heating the large reactor is also being designed.

10-UNDECENOIC ACID AND HEPTALDEHYDE FROM CASTOR OIL (in collaboration with the Organic Chemistry Division)

A continuous thermal cracking unit has been designed, fabricated and erected. Five runs on the unit have shown that the design is generally satisfactory. Due to insufficient heat-input capacity the experiments on this unit could be carried out only for brief periods. Another reactor incorporating necessary improvements and with a large heat-input capacity is under fabrication. Some changes to improve the performance of the partial condensers are also being made. Designs of a high vacuum fractional distillation unit for separating the various fractions of the cracked product are being prepared.

ETHYL ACETOACETATE

Preliminary laboratory investigations are being carried out on the preparation of sodium ethoxide from sodium hydroxide and absolute alcohol, based on a German process using azeotropic distillation for the continuous removal of water of reaction. A bench unit and a larger pilot unit are being assembled for carrying out this operation.

ETHYLENEDIAMINE TETRACETIC ACID

Work on the preparation of ethylenediamine tetracetic acid (EDTA) from ethylenediamine and monochloracetic acid has been started. This route has been preferred since both the starting materials. can be manufactured from indigenous raw materials. After a few preliminary runs in a ½ liter batch glass reactor, investigations are now in progress on the preparation of EDTA in a small continuous stirred glass reactor. The effect of process variables such as temperature of reaction, residence time, etc., on the yield of EDTA is being studied using the reactants in molar proportions.

PILOT PLANT PROJECTS

POLYVINYL CHLORIDE

Further experimental data were collected on the ethylene pilot plant reactor (stage I) simultaneously with the production of ethylene for stage II. This data, coupled with the extensive data obtained earlier, will be used for reactor scale-up design work. The life of the catalysts in the fluid bed was observed to be shorter than with fixed bed as found out in earlier investigations in this Laboratory. Thermal efficiencies of the reactor, superheater and vaporizer were also experimentally determined.

Regular runs are in progress to determine the effect of variables such as the chlorine-ethylene ratio, the residence time and the submergence on the ethylene and chlorine efficiencies and the output from the reactor. The EDC obtained in these runs is being collected and will be processed for use in the next stage.

The pilot plant for the thermal dehydrochlorination of EDC to vinyl chloride (stage III) has been installed and is undergoing preliminary tests. Installation of the units for the polymerization (stage IV) is being taken up and will be completed on receipt of the polymerizers. Some laboratory scale research investigations relating to certain specific problems in the pilot plant project are being carried out. The thermodynamic charts of vinyl chloride prepared in the course of this work have been published.

RAYON GRADE PULP

The pilot plant staff was appointed during the period under review and the active organization of the project was started. Experimental investigations on a laboratory scale are in progress on (a) preliminary treatment of some raw materials such as bagasse, (b) small scale pulping studies, and (c) analysis of different indigenous raw materials. The depithing of bagasse is being tried in a hammer mill. The prehydrolysis method, followed by chemical digestion of the prehydrolysed materials, is being tried for obtaining pulps of acceptable chemical composition. Detailed analysis of six different samples of cashew nut shells and two other raw materials has been done. The intermediate products, such as the pre-hydrolysed material and the unbleached pulp from bagasse, are also being analysed. Two large digesters of the circulating kier type, already available in the NCL, are being overhauled and assembled for use for pilot plant work. Some equipment necessary for the batch post-digestion treatment are being fabricated in the NCL workshop to enable us to carry out preliminary work till the standard equipment is procured. Detailed specifications of the latter required for the pilot plant have been worked out. Orders for the purchase of the Emil Blaschke viscose and spinning units have been placed with the DGS&D. Necessary foreign exchange facilities and import licenses for other equipment are being finalised.

HEXACHLOROETHANE

Several trial runs carried out on the small laboratory copper reactor gave not only low yields, but also showed rapid corrosion of the reactor leading to leakages. Further work with this reactor was therefore discontinued. The results obtained during earlier investigations (1954-55) were however reconfirmed in a laboratory glass reactor, principally to check up the quality and activity of the recently received coconut shell charcoal as catalyst. Reproducible and good yields were obtained as before. Pending the arrival of the pilot plant reactor (with a capacity of 5 lbs of hexachloroethane per hour), attempts are being made to scale up the work as much as possible with a larger glass reactor. Nickel is about the most suitable material of construction as reported earlier. However, due to the great difficulty in procuring it, alternative materials of construction are also being tried under actual experimental conditions. A single tube monel reactor is being fabricated to test its suitability with reference to corrosion under processing conditions. A small bench reactor made of M.S. has also been nickel plated and will be tried.

Orders for the multi-tube pilot plant reactor made of monel have been placed. Attempts to procure a similar one but made of nickel are being made. Experiments are also in progress in the small (500 c.c.) glass reactor to find out better catalysts to produce higher yields per unit volume of the reactor or enable the process to be carried out at lower temperatures. Designs for the heat transfer oil system for heating the reactor are being worked out.

SORBITOL FROM GLUCOSE

Further hydrogenation runs were carried out in the rocking autoclave with imported catalysts as also with Raney nickel. The imported copper chromite and supported nickel catalysts gave poor conversions under conditions found favourable with Raney nickel. Larger scale experiments were then tried with a 5-litre vertical stirred autoclave, but very poor conversions were obtained and charring was also observed. The stirring mechanism was then considerably modified to increase the speed and practically 100% conversion was obtained under conditions used earlier in the rocking autoclave.

To obtain the process kinetic data for pilot plant scale-up, systematic investigations have been started initially with glucose and Raney nickel. A small vertical autoclave used for this purpose gave poor performance and necessary modifications are being tried by introducing suitable baffles, etc. The catalytic reduction of glucose to sorbitol at atmospheric pressure using other catalysts is also being tried. Use of ion exchangers for removal of traces of nickel has proved useful. An order for a 20 gallon medium pressure autoclave has been placed.

ETHYLENE OXIDE

Preliminary work on the production of ethylene chlorohydrin has been carried out with a glass reactor.

These experiments are on a semi-batch basis with continuous feeding of ethylene and chlorine, but not of water. Maximum possible gas feed rates, short of slugging, were found out with air for (1) batch and (2) continuous co-current runs, both without recirculation. The approximate data thus collected were useful in adjusting the maximum feed rates of the reactants.

INORGANIC CHEMISTRY

TITANIUM DIOXIDE

The two parts of the project, (i) chlorination of the sludge and isolation of titanium tetrachloride, and (ii) hydrolysis of the purified tetrachloride to yield pigment grade titania, were studied concurrently. Removal of iron oxide from the sludge before chlorination was essential, and the method found most suitable so far was to calcine the sludge with carbon above 900°, cool and leach it with hydrochloric acid obtained from the subsequent step of hydrolysis of the tetrachloride. Laboratory results were reproducible with a batch of 25 lbs. of sludge, which on treatment gave a product containing Fe_2O_3 3, TiO_2 39 per cent from the original material containing Fe_2O_3 16.1, TiO_2 26.1 per cent (both calculated on ignited basis).

It was necessary to decolorise, at least partly, the reddish-coloured crude tetrachloride to obtain a good white titania on hydrolysis. It was observed that this could be done by cold contact with aluminium foil instead of the usual distillation procedure recommended for this corrosive, water-sensitive liquid.

For hydrolysis, it was considered an obvious advantage to lower, if possible, the temperature of hydrolysis from above 100° to around 60°, as in the latter case one might use rubber-lined mild steel tanks in place of costly equipment resistant to hot hydrochloric acid solutions. Direct hydrolysis at 60° (with usual seeding) proceeds to less than 70%. From detailed

studies on the nature of the seed used, the ratio Ti:Cl in the hydrolysing liquid and the effect of addition agents, the extent of hydrolysis (at 60°) could be increased up to 95%. This study was combined with one on the effect of conditioning agents on the final physical characteristics of the pigment after calcination so as to obtain a bright, soft and non-gritty product.

SYNTHETIC CRYOLITE

Quantitative laboratory studies on the selective extractability of the fluorine of silica-containing fluorspars with neutral complexing reagents such as aluminium sulphate showed that while the desired elimination of silica was achieved, the cryolite was heavily contaminated with alumina. The process, even on replacing aluminium sulphate with ferric sulphate and recovery of the latter, was found to be uneconomical.

Since much of the silica in impure fluorspar can be removed by ordinary mineral concentration methods, study is now restricted solely to extraction of the fluoride by alkali fusion, with subsequent processing to remove the small amount of silicate to the extent required by the aluminium industry. The presence of an oxidic catalyst was found necessary, and ilmenite was very effective, which confirms certain observations made by us in 1955. Optimum conditions are being worked out. Attempts to re-use the leached ilmenitic sludge as catalyst in the fusion of a fresh charge were unsuccessful.

PHOSPHATIC COMPOUNDS

The synergic effect which some condensed soluble phosphates produce on the detergency of soap and detergent compositions is known. Trisodium phosphate, now produced in India, is a suitable starting material, and a process was worked out in 1952 on making sodium tripoly-phosphate from it by reacting with phosphoric acid. A much cheaper method is now being worked out on a laboratory scale by replacing phosphoric with hydrochloric acid and removing the sodium chloride formed at the appropriate stage. Several variables affect the yield and purity of the product, the most common contaminant being pyrophosphate, which should be below 5%.

SEPARATION OF THE RARE EARTHS

Ceria precipitation cycles, using 2 kg rare earth mixed chlorides per cycle, were run regularly to (i) prepare ceria of over 99% purity in an average yield of 85%, and (ii) supply the necessary raw material for obtaining other light rare earths (La, Pr, Nd, Sm). The ceria-free mixture, after removal of the small quantity of 'heavy' earths, used to be bifurcated into a 'lanthana concentrate' and a 'didymia concentrate' in a cyclic two-stage hydrolytic process. After a detailed study of this step it has been found possible to simplify this into a single step hydrolytic operation. Further work on the recovery of pure lanthana by immobilization on a cation-exchanger, separation of the didymia-samaria concentrate into predominantly binary fractions prior to final separation, etc., is being continued. The project is partly supported by a grant from the Department of Atomic Energy.

PURE ZIRCONIA AND PURE HAFNIA FROM INDIAN ZIRCON

The laboratory process of making zirconium oxychloride through alkali fusion, reported earlier, was expanded to 30 kg. zircon per batch and useful process data are being collected in the processing of a total of 70 kg of the mineral. The crude oxychloride obtained through the usual stages is being recrystallized iron-free for use in the silica gel column to remove hafnium. Two columns, each capable of handling more than 1 kg of the oxychloride, have been set up and are being run intermittently to collect samples and working data. Besides giving pure zirconia containing less than 100 p.p.m. of hafnia, the column yields about four-fifths of the total hafnia in the form of a 30% concentrate, which is found to be very suitable material for the preparation of pure hafnia by an anion-exchange process being developed simultaneously. The project is partly supported by a grant from the Department of Atomic Energy.

PURE NIOBIUM AND TANTALUM OXIDES FROM INDIAN TANTALO-NIOBATES

The simultaneous recovery of the twin oxides of niobium and tantalum in a high stage of purity by a simple chemical extraction of the mineral followed by a liquid-liquid extraction step has been worked out and was reported earlier. Though the recovery of the (imported) solvent used for the purpose was about 80%, the possibility of using one indigenously available and cheap, even with a lower degree of recovery, is indicated by further work in the current period.

TITANATE/SILICATE AND OTHER INORGANIC ESTERS

The feasibility of replacing (imported) butyl alcohol with fractionated fusel oil, a by-product of the Indian alcohol industry, in the preparation of hydro-phobic-cum-coating compositions of titanate/silicate was reported earlier. Lately, several such fusel esters have been prepared by improvement or modification of the known methods for the corresponding butyl analogues. These include the titanate, phosphate, phathalate and acetate, on which comparative tests will be carried out in the several fields of their probable application. A new and simple method has also been found to remove the colour and offensive smell of the fusel oil fraction used for these preparations.

SPECIAL PREPARATIONS

Ceric ammonium nitrate and ceric sulphate tetrahydrate of analytical reagent grade have been prepared from 99% ceria obtained from the Division's rare earths project. Following an outside request for pure hydrazine chloride, the first step of preparing the sulphate has been standardized and the high yield of 45% has been made reproducible. It was observed that about two-thirds of the total ammonia used in the synthesis could be recovered in a laboratory - scale experiment.

ORGANIC CHEMISTRY

SYNTHETIC DRUGS

(a) Synthesis of pyridoxine (Vitamin B_6)

Work on the synthesis of pyridoxine hydrochloride (Vitamin B_6) has been carried out on a laboratory scale, starting from chloracetic acid. This method involves ten steps which have been investigated to get maximum yields. Reduction of the intermediate compound 2-methyl-3-nitro-4-ethoxymethyl-5-cyano-6-chloropyridine to 2-methyl-3-amino-4-ethoxymethyl-5-aminomethylpyridine was carried out initially using Pd/carbon catalyst, and the recovery of palladium from the spent catalyst was also studied. Now a cheaper catalyst has been successfully used for this reduction. Pilot plant work on the preparation of vitamin B_6 is being undertaken shortly on the basis of these results.

(b) Synthesis of stilbestrol and its analogues

(i) Diethylstilbestrol

Homoanisic acid, an intermediate for the preparation of deoxyanisoin, the starting material for the preparation of stilbestrol by Dodd's procedure, was prepared by a modified method. The standard methods of preparation mentioned in the literature were repeated and comparative data on the yields were collected. It was observed that the method developed in this Laboratory gave the highest yields (about 70%).

(ii) Dimethylstilbestrol

α-Methyldeoxyanisoin, an intermediate for the preparation of dimethylstilbestrol, was prepared from deoxyanisoin by the action of methyl iodide in the presence of sodium ethoxide. 2:3-Dianisylbutane-2-ol was prepared

from -methyldeoxyanisoin by the Grignard reaction with methyl magnesium iodide. 2:3-Dianisylbutane-2-ol has been dehydrated by Dodds to dimethylstilbestrol dimethyl ether by the action of phosphorus tribromide. Better yields are obtained by using phosphorus oxychloride.

(iii) α-Dienestrol

Preliminary experiments were carried out for the preparation of dienestrol by the following route:- Phenol → p-hydroxypropiophenone → mixture of pinacols (α and β) → α-pinacol diacetate (separation) → dienestrol diacetate → α-Dienestrol. An analytically pure sample of α-dienestrol was prepared Experiments on the standardization of the intermediates are in progress.

(c) 4-Hydroxycoumarins

The new method using malonic acid has been extended to the synthesis of other 4-hydroxycoumarins including 1-thio-4-hydroxycoumarin, 3-phenyl-4-hydroxy-coumarin and 3-ethyl-4-hydroxycoumarin.

(d) Preparation of m-xylohydroquinone

The preparation of m-xylohydroquinone, which has been reported to be promising as an oral contraceptiv is being investigated. The known procedures starting from m-xylenol have been repeated and confirmed, and a new route starting from p-nitrophenol is being explored.

STEROLS

(a) Progesterone from bile acids

Experiments were continued on the synthesis of progesterone from bile acids obtained from Bombay. The several stages involved in the synthesis have been satisfactorily completed on a laboratory scale. Larger scale experiments are being taken up.

(b) Sterols from sugarcane wax

The new method evolved for the extraction of sterols from sugarcane wax has been worked out in detail, and will be shortly tried on a pilot plant scale.

ORGANOBORANES

A systematic study of the synthesis of organoboranes containing functional groups has been initiated. Standardisation of conditions under which a double bond in an olefinic compound can be hydrated or shifted to another position by thermal isomerisation will be useful in synthetic organic chemistry.

Borohydride reductions

A study of the reducing action of the complex metal hydrides and diborane on substituted anthraquinones has been started.

Utilisation of cashewshell liquid

Anacardol, prepared from cashewshell liquid, has been hydrogenated, etherified and sulphonated. Its possible use as a detergent is under examination. Complete hydrogenation of anacardol at 260-270° and 200 atms. pressure yields secondary alcohol as a colorless wax. A ketone is obtained by oxidation of this alcohol, and both these products are under further examination.

N-methyltaurine

A method for the production of this important intermediate for wetting agents and stabilized diazo compounds is being worked out using locally available ethylene chlorohydrin.

NEW SYNTHESIS OF 2:4-DIHYDROXYQUINOLINES

A new and simple synthesis of 2:4-dihydroxyquinoline involves the use of zinc chloride and phosphorus oxychloride as condensing agents. The process utilises easily available materials and has certain advantages over the known processes. The general applicability of the method has been established by the preparation of a number of 2:4-dihydroxyquinolines.

PREPARATION OF ISOPROPYL ALCOHOL

At the request of the Defency Ministry work on the preparation of isopropyl alcohol from acetone has been undertaken. Apparatus has been fabricated and preliminary experiments have been carried out.

WAXES

(a) Sugarcane wax

Cane wax which was left after the extraction of sterols was oxidised thrice by sodium dichromate and sulphuric acid. This oxidized wax was comparable with the oxidized wax from total cane wax. Experiments were carried out for improving the solvent take up of nitric acid bleached wax; products with higher melting points have been obtained, but the solvent take-up property remained unsatisfactory.

Basic chromium sulphate has been prepared from chrome liquor, a by-product in the process of refining and modification of sugarcane wax.

(b) Sisal wax

Extraction of sisal wax from sisal waste

Waste material, left after the separation of fibre from sisal leaves, was used for this purpose. Two types of wastes are available, one obtained by the retting method and the other by the mechanical method, wax was extracted from these waste materials by hot toluene. Four extractions could remove almost all the wax. The waste from the retting method gives a very high percentage of wax (12-13%), which is of a good quality. The other waste gives about 6% of wax. Some of the above wax samples are comparable with carnauba wax as regards solvent take-up property. In larger scale experiments about 14 lbs. of waste material was extracted in one batch.

The sisal wax samples were tested by preparing standard creams and comparing their hardness with those prepared from carnauba wax. The sisal wax creams are heat resistant.

Composition of sisal wax

Work on the composition of sisal wax has been undertaken.

VEGETABLE OILS WITH SPECIAL REFERENCE TO INEDIBLE OILS

(a) Pilot plant work on nim oil

Crude nim oil (total 426 gallons) was extracted with dilute alcohol in about 40 gallons batch unit in mild steel or stainless steel drums fitted with baffles. Alcoholic extractive varied between 7 to 11% of the oil. The optimum conditions of alcohol extraction and various data in regard to loss of oil, loss of alcohol (5-8%), etc., were determined. Refining and bleaching experiments with the alcohol extracted oil have been carried out in a De Laval unit (2 gals. capacity) and optimum conditions of temperature, strength and amount of alkali, etc., determined, and refining loss (about 18-20%) estimated and accounted for. When 'acid oil' from refining foot is taken into consideration, refining loss including the loss due to alcohol extraction works out to 10-13%, which is reasonable considering the characteristics of the raw oil and also the quality (pale yellow with very faint characteristic odour) of the resultant oil (2 to 3 Y+0.2 to 0.5 R; Lovibond, 1 cm.) which could be hydrogenated with the use of 25-30% extra catalyst.

The pilot plant work on refining-bleaching, deodorization and hydrogenation-has subsequently been carried out in the Hindustan Levers (Bombay) factory pilot plant unit (600-1000 lbs. capacity), and data on each step were collected. The resultant oil and fat are completely free from odour and taste. The alcohol extracted nim oil was supplied to a factory for experiments on splitting by high pressure steam in an autoclave. The mixed fatty acid thus obtained was greyish white in colour after bleaching with earth and was free from disagreeable odour and taste. While quality soap as well

as high grade fatty acids can be produced with the purified, refined and hydrogenated nim fat, the alcohol extracted oil itself can also be used for making soap or fatty acids.

(b) Processing of alcoholic extractive of nim oil

An appreciable quantity of alcoholic extractive (ca. 10 kilos) obtained from the pilot plant work on the alcohol extraction of nim oil has been processed and about 700 gms. of nimbidin (T) prepared. This is being used for preparation of sodium nimbidinate required for pharmacological studies and clinical trials.

(c) Nimbidinates

Various methods of hydrolysis of nimbidin as well as 'total bitters' are being tried in order to standardise the method for preparation of sodium nimbidinate of uniform physiological activity.

(d) *Denaturants for alcohol*

Methods have been evolved for quantitative estimation of the denaturants, pyronimin as well as 'total bitters' in denatured spirits. A method has been developed for quantitative estimations of the denaturants, based on the colour produced by them with sulphuric acid. The colour developed can be compared against standard iodine solution in a Helige-Dubosco type colorimeter.

(e) Some experiments on refining and hydrogenation of nim oil

Bitters in nim oil could be removed by treatment with 60°Be' sulphuric acid, but the sulphur compounds responsible for the unpleasent odour could not be completely removed by this treatment. Oxidation with potassium permanganate, potassium dichromate or hydrogen peroxide could not deodourise the nim oil. Treatment of the oil

with acetic acid, oxalic acid, citric acid was also found to be unsatisfactory. Reduction with sodium sulphide or sodium bisulphite was found to have no action on the sulphur compounds. Treatment of the refined nim oil with about 1% Raney nickel deodourised; treatment with zinc and acetic acid or hydrochloric acid was found to be fairly satisfactory, but these methods are not economical.

(f) Extraction of nim oil (in collaboration with the Chemical Engg. Divn

Further experiments with the centrifugal Podbielaniak extractor showed that the particular model is not suitable for the nim oil-alcohol system. The output was too low and an undesirable temperature rise was taking place in spite of making many modifications suggested by the firm. Hence the batch tank extraction method was finally adopted with 30 gal. of oil per batch. Repeated extractions (6 to 7) were carried out. Approximate rate studies indicated that extraction for 3 hours was sufficient. An additional stirred extraction tank was fitted up. About 3 tons of raw nim oil have been processed. The extracts were distilled, the first and second under vacuum. A total of 3780 lbs of finally processed oil (3440 lbs of oil 340 lbs of absorbed alcohol) was obtained. About one ton of this was sent to a firm for further treatment and hydrogenation trials. Pending the receipt of further quantities of raw nim oil and Karanja oil, preliminary studies on the continuous countercurrent extraction of these oils in a small rotary disccontactor have been started.

PREPARATION OF FATTY ALCOHOLS, SURFACE-ACTIVE AGENTS AND DETERGENTS FROM VEGETABLE OILS

Pisa seed oil and phulwara butter (Madhuca butyraceae) were reduced to lauryl alcohol and a mixture of cetyl alcohol and oleyl alcohol respectively (yield 91%). Cetyl alcohol was separated from oleyl alcohol by fractional distillation under reduced pressure (190-195°/15 mm.). Similarily nim oil has been reduced to a mixture of alcohols from which a fraction consisting of cetyl and stearyl alcohols (I:1) is obtained by fractional distillation at 180-185°/5 mm. These alcohols have been supplied to the Physical Chemistry Division for use in its water evaporation studies. A few typical and well-established detergents and germicides are being prepared and submitted to tests in the Physical Chemistry Division to enable a choice of materials and methods to be made for economical production.

Preparation of isopropyl myristate

Isopropyl myristate is used in cosmetic formulations. Khakan fat (Salvadora oleoides or S. persica) contains about 28% myristic acid and is a suitable raw material for the production of isopropyl myristate. Isopropyl myristate was prepared by the following procedure : Khakan fat → methyl esters → Methyl myristate by fraction of methyl esters → myristic acid → isopropyl myristate. About 30% isopropyl myristate could be obtained from Khakan fat. It was observed that isopropyl esters can be directly prepared from Khakan fat by esterification with isopropyl alcohol.

Kokum butter

The hydrogenation of kokum butter was carried out using Rufert nickel catalyst and the iodine value was brought down to less than unity.

Chrysalis oil

A sample was sent by the Kashmir Government. The oil was dark reddish brown in colour with a strong disagreeable odour. Its acid value was 104. The oil cannot be refined by conventional methods. The dilute alcoholic extract showed the presence of di-palmitin (m.p. 64-55°) as reported in the literature.

Jatropa-curcas oil

A sample was sent by the Bombay Village Industries Board for ascertaining its suitability for soap making. The Jatropa nuts are reported to be very toxic to cattle and they are classified with the castor species. The sample of oil supplied was optically inactive and ricinoleic acid was absent. The oil being a soft one its suitability for soap making will have to be further investigated.

(e) Khakan and pisa fats

These two myristic and lauric acid-rich fats could be purified and refined to colourless fats with practically no disagreeable odour by the methods developed in this Laboratory for processing inedible oils such as nim. Systematic chemical examination of the non-fatty constituents is in progress.

Effect of vitamin A acetate on Boudouin test of vanaspati

The tests on the effect of vitamin A acetate on the Boudouin test of vanaspati were concluded and a report submitted to the Government of India and for publication in the Jour. Sci. Industr. Res.

PAINTS AND VARNISHES *

(a) Air drying wrinkle finish coating compositions

Air drying wrinkle finish coating compositions of the type formerly developed in the Laboratory were prepared using kamala seed oil, tung oil and stand linseed oil. Ester gum and maleic anhydride modified resins for the costing compositions were also prepared in the Laboratory. Paints from these varnishes were prepared using different types of pigments. Films of the varnishes and paint were applied on glass, tin, aluminium and hard board surfaces and the performance of the films were tested by standard methods. Sample panels were prepared from these varnishes and paints and supplied to Regional Research Laboratory, Hyderabad.

(b) Kamala seed oil

Kamala seeds (200 lbs) were cleaned by the method developed in the Laboratory. The cleaned seeds were extracted in the cold with benzene, and 11 lbs of oil were supplied to N.R.D.C., England, at their request.

Keeping qualities of kamala seed oil

Kamala seed oil was stored without adding any antioxidant or preservative in transparent glass containers exposed to light, glass containers kept in the dark, and in galvanized tins, at room temperature. Weekly determination of iodine values and kamlolenic acid content of the fatty acid did not show any change during two months, but the oil had thickened slightly.

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* Except for problems arising from the programme of the Polymer Chemistry Division, work on this field is being transferred to the RRL, Hyderabad.

Keeping qualities of kamlolenic acid

Kamlolenic acid was kept in the dark as such, under petrolium ether at room temperature at 15° and at 0°, and in methyl alcohol, ethyl alcohol and methyl ethyl ketone at room temperature. The results showed that kamlolenic acid as such deteriorates rapidly. Under petrolium ether at room temperature it deteriorates after a week at 15° under petrolium ether the deterioration distinctly sets in only after a month; while at 0° it kept well for six weeks. In the remaining three solvents, there is a definite fall of a few units in the kamlolenic acid content after a month.

THE CHEMISTRY OF COMMERCIAL DYES

The main constituent of nitrated dibenzanthrone, which dyes green developed to a black by hypochloride oxidation, is 16-nitrodibenzanthrone. The compound has been isolated in crystalline and analytically pure form. Work on the constitution of the black dye produced on the fibre and on the chemistry of the so-called Vat Direct Blacks is in progress. Some years ago Dr. N.V. Talavdekar submitted a Ph.D. thesis on "Cyanurated and other azoic dyes". In view of the recent interest in dyes derived from cyanuric chloride as representing a new class of dyes combining directly with cellulose, work in this field has been resumed. Work on the chemistry of anthrimides and carbazoles is being continued. The thesis of N.B. Desai recently approved for the Ph.D. of the University of Bombay includes a section on the colour of 16:17-dimethoxydibenzanthrone in relation to its structure and stereochemistry. 16:17-dimethoxy dibenzanthrene has been prepared, but as partially anticipated it has not been possible to resolve this compound and demonstrate its optical activity. 16:17-dimethyldibenzanthrene, which may be easier to resolve, is being prepared, as well as similar compounds containing acid groups which may therefore be combined with optically active bases.

THE CHEMISTRY OF NATURAL COLOURING MATTERS AND OTHER PLANT PRODUCTS

1:3:8-Trihydroxy-2-hydroxymethylanthraquinone has been synthesised and found to be different from versicolorin isolated by Hatsuda from the metabolites of Aspergillus versicolor. Damnacanthol and damnacanthal, two colouring matters occuring in a Japanese plant, have been synthesised by a new method. The synthesis of endocrocin, which is a colouring matter of great interest occurring as a mould metabolite and in a lichen, is nearing completion. In view of the reported antitubercular activity of aloe-emodin and rhein, a series of anthraquinone derivatives are being synthesised and examined. At the request of two physicians in Bombay, who have found by clinical trials that the fruits of Capparis monii(Rudanti) has powerful antitubercular activity, the chemistry of the constituents is being examined. Work on the constituents of the heartwood of Artocarpus integrifolia and the colouring matters of Garcinia morella is being continued.

ESSENTIAL OILS

SYNTHETIC PERFUMERY MATERIALS

(a) Macrocyclic compounds

(i) Civetone

Starting from oleic acid, 250 gms of pure cis-civetone and 6 kg of the intermediate, civetone dicarboxylic acid, have been prepared. In this synthesis, 99.3% pure pelargonic aldehyde, of which 12 lbs have been collected, is obtained as a by-product which meets a major portion of the processing cost in the civetone synthesis.

(ii) Dihydrocivetone

About 1 lb of dihydrocivetone has been prepared starting from kamala oil. Benzophenone and 1:1-diphenylethylene are obtained as by-products, which find some use in cheap perfumes.

(iii) Macrocyclic compounds from shellac

The preparation of pentadecane-1:15-dicarboxylic acid, the intermediate for dihydrocivetone, from 500 g. of aleuritic acid isolated from shellac was completed. The yields are almost quantitative at every stage. From an evaluation of the comparative cost-data worked out in consultation with the Secretary, NRDC, this method appears to be superior to that employing kamala oil.

(iv) New routes for the preparation of macrocyclic compounds

Preliminary work is in progress on the reaction of derivatives of undecylenic acid and cyclo-alkanones.

(b) Methyl heptene carbonate

It is widely used in high-grade perfumery preparations. Experiments have been carried out to standardise the conditions for preparing it from heptaldehyde.

(c) Dihydrojasmone

Dihydrojasmone (500 g.) was prepared in one batch through the Stobbe condensation of octanone-2 with diethyl succinate. The quantity of polyphosphoric acid employed at the last stage of the synthesis has been reduced to one-third of the quantity employed in the earlier trials.

(d) Dihydroisojasmone

Another trial for the preparation of dihydro-isojasmone starting from undecylenic acid (300 g.) was undertaken. The product was obtained in yield of 33% which could not be further improved as the rest of the product undergoes polymerization during the reaction.

(e) Preparation of menthol

Menthol is an important aromatic chemical which is imported to the value of about Rs.70 lakhs. Preliminary investigations on the preparation of menthol from Java citronella oil have therefore been undertaken.

ESSENTIAL OILS

(a) Costus root oil

Costus roots (1000 lbs) were extracted to yield 62 lbs. of costus root oil. Experiments were simultaneously conducted to collect data on the effect of particle size and stirring on the efficiency of oil extraction (in collaboration with the Chemical Engineering Division). The oil has been subjected to a refining tfeatment which considerably improved the quality of the oil. Studies on the constituents of costus root oil were continued. A tentative structure for the lactone costunolide has been proposed. Its structure is somewhat unusual, containing a 10-membered carbocyclic system. The hydrocarbon components have been isolated by fractional distillation from larger quantities of the oil for further studies. During chromatographic purification of the components, it has been found that the fractions contain, besides hydrocarbons, oxygenated bodies. The hydrocarbons and the oxygenated bodies have the same boiling point and cannot be separated by fractional distillation.

(b) Agar wood oil

A new sesquiterpene alcohol $C_{15}H_{16}O$ belonging to the eudalene series has been isolated from agar wood oil and its structure elucidated. The alcohol, hitherto unknown, has been named as 'Agarol' to indicate its relation with agar. Eight other components have been isolated so far from the oil.

(c) Vetiver oil

The sesquiterpene hydrocarbon, b.p. $94^{o}/2$ mm., isolated from the South Indian variety has been examined. The experimental evidence collected so far indicates that the hydrocarbon has the cadalenic carbon skeleton containing two double bonds.

(d) Wild ginger oil

Investigations on the elucidation of the structure of zerumbone were continued. On the basis of the evidence so far collected, a tentative structure for tetrahydrozerumbone has been proposed. The hydrocarbon fractions obtained from the oil were chromatographed over alumina and the different fractions were examined. It was found that these fractions were composed entirely of one hydrocarbon which on further purification was found to be identical with humulene. The alcoholic fraction has been purified by refractionation followed by chromatography. Further work for the elucidation of the structure of these components is in progress.

(e) Resin oil from Shorea Robusta

The resin exuded by Shorea robusta tree is commonly known as 'Sal resin' (Dhup). About 15 lbs of the sal resin were subjected to destructive distillation at high temperatures under vacuum. The oil which distill out was obtained in a yield of about 68%. The crude oil was totally distilled in vacuum and the resin oil so obtained was separated into the acidic, phenolic and neutral portions. The neutral portion has been repeatedl fractionated and the various fractions further purified by chromatography. One of the fractions appears to be a hydrocarbon $C_{10}H_{16}$ and is probably related to ocimene. Some of the components show unusually high refractive index. One of the components appears to be pure blue azulene. Further investigations for the characterization of these components are in progress.

PHYSICAL CHEMISTRY

PREPARATION OF WATER-DISPERSIBLE DDT

(a) Spray atomization

As a first step in the preparation of water-dispersible DDT powder, experiments on the micronization of DDT(technical and partially purified) by spray crystallisation of their melts on the newly fabricated high speed spinning top type atomizer were carried out. The micronized powder yield of DDT was, however, much less than that of other substances, such as paraffin wax, stearic acid, Lindane (99% γ-HCH) which also were similarly micronized. Consequent on the termination of the I.C.M.R. scheme, experimental work on this project has been suspended.

(b) Study of supercooled systems

To supply fundamental data as an aid in the above process, the linear velocity of crystallisation of a number of compounds was measured on a specially constructed microscope stage at different supercooling temperatures. Apparatus for the determination of viscosity, density and surface tension of supercooled melts was calibrated.

SURFACE CHEMISTRY

(a) Prevention of water evaporation

A comparison was made of the differences in loss of water due to evaporation from water surfaces covered with films of mixtures of long chain fatty alcohols prepared from various indigenous oils. The evaporation losses were found to be the least in the case of films of purified cetyl alcohol obtained by the fractionation of commercial cetyl alcohol.

An apparatus has been fabricated for the determination of two-dimensional viscosity of surface films.

(b) Electro-deposition and vapour-phase deposition

A study of the structure and growth of cuprous iodide formed from vapour phase on (100) face of NaCl, (0001) face of mica and (101) face of calcite showed that the deposit formed at room temperature was always crystalline with {111} orientation, while at about 300°, the halide developed 2-degree orientation on NaCl. The growth of silver on PbS showed normally developed {111} orientation changing gradually to {100} orientation with increase in the substrate temperature.

Weak photovoltaic effect was observed in CdTe deposits condensed obliquely on glass from vapour in vacu

Thin metallic films deposited in vacuo on nylon fabrics were found to be fairly adherent. Experiments are being extended to various metal films as well as other fabrics.

SOLID STATE CHEMISTRY

Thermistors are much in use in electrical and electronic circuit as a control. The electrical conductivi of binary systems of manganites was studied to find the variation of the activation energy as a function of composition. These results enable us to select suitable thermistor materials for different requirements. A process has been developed for making number of thermistor beads in one run in a simple way. Sealing techniques are being developed.

(a) Dielectric materials

With a view to producing good dielectric material, the properties of crystals having the general formula $A^{I}_{\frac{1}{2}}B^{III}_{\frac{1}{2}}Ti^{IV}O_3$ were studied. In the $La_{\frac{1}{2}}Li_{\frac{1}{2}}TiO_3$ - $BaTiO_3$ system the dielectric constant showed a maximum at 8% $BaTiO_3$. The ferroelectric property of $BaTiO_3$ was destroyed by addition of more than 2% $La_{\frac{1}{2}}Li_{\frac{1}{2}}TiO_3$. Work is in progress to decrease the dissipation factor of these compounds by preparing and sintering the compounds in an oxygen atmosphere.

(b) Electroluminescence

The effect of impurities on the brightness of different ZnS type phosphors in an A.C. field is being studied.

(c) Magnetic susceptibility

A magnetic balance has been set up and the working range is being extended down to liquid air temperatures.

(d) Corrosion

Corrosion of alloys under atmospheric conditions is being studied.

(e) Structural studies

A relation between temperature and the cation distribution in spinels has been theoretically deduced. This gives a simple X-ray method of evaluating he site preference energy which was hitherto undetermined. The experimental results obtained for $MgMn_2O_4$ conform to the above deduction.

The structures of solids synthesised for the dielectric studies were determined.

The $\alpha \rightarrow \beta$ ZnS transformation at high temperatures is being studied by X-ray diffraction.

Thermoelectric powers of tellurium doped with different impurities were studied as a function of temperature.

(f) Photoconductivity

Materials using CdS doped with Cu and halogens were prepared and used in experimental cells.

CATALYSIS.

Raney alloys were prepared under various conditions, and one was found to compare favourably with the American product as regards fineness, composition, pyrophoric nature and X-ray diffraction pattern. Hydrogen absorption of cinnamic acid and desulphurisation of anthraquinone-1:8-disulphonic acid showed the laboratory product to be as good as the imported variety.

Copper bronze prepared in the form of bronze flakes was in the size range 5-30 μ, and it effected decarboxylation of anthraquinone-β-carboxylic acid in a yield of 55%.

An elaborate calorimetric and adsorption apparatus is being constructed for accurate measurements of heat capacity and heats of adsorption for the prediction of the catalytic activity of various substances.

Preliminary investigations are in progress for the preparation of p-cymene from Δ^3-carene obtained from Indian turpentine.

SOLUTION PROPERTIES OF COLLOIDS AND MACROMOLECULES

Examining known solvents for cellulose in connection with the rayon grade pulp project, it was found that zinc ethylenediamine and chloride ions in a narrow range of composition exhibited the property of dissolving cellulose. Measurement of the change of viscosity with time indicated that there was no further appreciable degradation of cellulose with time.

Light scattering and viscosity studies on the thermal degradation of sol rubber were made at 60° and at room temperature in three solvents and at three concentrations.

Theoretical expressions for the light scattered by thin polymer films under stress and various conditions of polarisation were obtained as functions of stress Young's modulus, Poisson's ratio and polar and azimuthal angles of observation.

An expression for the second virial coefficient A_2 for the solutions of surface active agents in water has been obtained by theoretical study. Also, an expression for the excluded volume was obtained as a function of surface potential, absolute temperature, and Debye's reciprocal distance of the ionic atmosphere.

Surface active agents

In industrial and technological applications, there is a growing need to evolve simple and quick methods for testing the suitability of various surface active agents. Instruments and apparatus have been fabricated for the evaluation of known and new surface active agents now being prepared in this Laboratory from

indigenous raw materials in terms of their foaming power, wetting of textile fibres, dispersing power, interfacial tension, contact angle, etc.

The effect of additives on the critical micelle concentration, foam stability, etc., is being investigated.

INFRARED ABSORPTION SPECTRA STUDIES

Infrared structural studies were made on various natural products and synthetic dyes.

Quantitative estimation of squalene present in a number of fish liver oils by infrared absorption were completed for the Government Oil Factory, Kozhikode. Tentative spectro-structural correlations were made of homologous series of n-alkylmalonic acids and their diethyl esters. The absorption bands for the hydrocarbon chains in the acid molecule suggest a triclinic packing.

RADIOACTIVE TRACER TECHNIQUE

The decay rates of human red blood cells in patients having various forms of cancer are being investigated by radioactive tracer techniques. A method of finding blood volume and its rate of production was worked out. A pilot study of anaemia using cobalt-labelled vitamin B_{12} has been completed. The data collected by tests on normals were utilized for diagnosis of the various types of anaemia in a large number of patients. (in collaboration with the Armed Forces Medical College).

A tracer method for the determination of thickness of different fibres and the sizing efficiency of various compositions has been developed for the Ahmedabad Textile Industry's Research Association.

INSTRUMENTATIONS

This Section attended to a large number of repair and fabrication jobs of which the following are examples: (1) Construction of a large electro-magnet (for use in a Nuclear Magnetic Resonance Spectrometer) dissipating 1.5. kW and capable of 10 kilogauss in a 2" gap over 6" dia. pole pieces. (2) Instrument for the measurement of thermal conductivity of rubbers and plastics (for the Polymer Chemistry Division), (3) Servicing and recalibration of the infrared spectroscope using different prisms, (4) Design and construction of a large number of exhibits and working models for the India 1958 Exhibition.

POLYMER CHEMISTRY

POLYMERISATION STUDIES

(a) The polymerisation of methyl methacrylate in aqueous solution initiated by hydrazine hydrate was investigated under high vacuum conditions. The experiments were designed to establish the following: (i) the role of oxygen, (ii) the mechanism of production of free radicals in the absence of oxygen, (iii) the role of metal ions on the reaction. Evidence obtained so far points to the possibility of production of free radicals by oxidation of hydrazine in alkaline solution of metal ions.

(b) N-nitrosoacetanilide was studied as an initiator in the polymerisation of methyl methacrylate at 30°. While the rate of polymerisation falls appreciably with time even with 5-10% conversion, probably due to rapid wasteful decomposition of the initiator, the initial rate follows a regular square root relationship to the concentration of the initiator. Another notable feature is the low molecular weights of the products obtained, much lower than expected for the same rates in bulk polymerisation.

(c) Experiments on the polymerisation of methyl acrylate using an amine-peroxide initiating system failed to yield reproducible results. The reason was investigated and found to be the low purity (45%) of the available sample of monomer freed from inhibitors by alkali washing and fractional distillation.

(d) Experiments towards obtaining liquid polymers of ethylene (C_{10}-C_{18} hydrocarbons) suitable as alkyl components in the preparation of detergents of the alkyl-aryl-sulphonate class were undertaken. With the available facilities for work under normal pressure, efforts were made to utilise $AlCl_3$, the most active among ionic catalysts in the polymerisation of ethylene, in the presence of alkyl halide co-catalysts. Ethylene dichloride prepared in the Laboratory has been used as the solvent-co-catalyst, with $AlCl_3$ and also $TiCl_4$, the liquid polymers obtained being stored under nitrogen. Another line of approach has been to use the metal halides $AlCl_3$, $TiCl_4$, $ZrCl_4$ in the presence of finely divided metals, e.g., aluminium which can reduce the transition halides to a lower valency. In the presence of alkyl halide a reactive catalyst would be expected to form <u>in situ</u> analogous to the Ziegler system and capable of functioning at atmospheric pressure. The combinations, $AlCl_3$/Al/ethylene dichloride, $TiCl_4$/$AlCl_3$/Al/ethylene dichloride have been used and small amounts of liquid polymers prepared.

<u>Solution properties</u>

i. Experiments with dilute solutions of sol rubber and polystyrene suggested that the observed anomalous behaviour of polymer solutions at high dilutions results from the configurational changes of the macromolecules in solution. Further evidence to this effect was obtained from the viscosity behaviour of the tertiary system polymer-A, polymer-B, and solvent. The viscosity behaviour of polymer-A, was examined as a function of the concentration of the polymer-B in the

mixed solvent (polymer B + solvent). A break in the viscosity-conc. curve at a point corresponding to the minimum in the usual viscosity-conc. curves was observed suggesting that the maxima-minima effects observed in the viscosity behaviour of polymer solutions at high dilutions results from the configurational changes of macromolecules in solution.

ii. Polymethyl acrylate obtained by polymerising the monomer at 40 ± 0.1 to about 25% conversion is being fractionated from a 2% solution in benzene using methanol as a precipitant. These fractions will be utilised for studying the effect of shear on the viscosities of their dilute solutions.

POLYSTYRENE

i. Using a 10 gallon-kettle a few large scale trials with the suspension technique (process being patented) were completed. The product, was, however, not quite clear due to contamination with grease, etc., from the reaction kettle.

ii. Work on expandable polystyrene beads using low boiling non-solvents as blowing agents has been taken up.

iii. To develop useful flame-proofing agents for expanded polystyrene, synthesis of the bromo derivatives of diallyl phthallate and triallyl phosphate has been undertaken. These will be used during the polymerisation of styrene.

iv. Cation-exchange resins based on polystyrene and with different degrees of cross-linking (by means of divinyl benzene) were prepared and their capacities evaluated. The resins compare well with standard imported products.

ION-EXCHANGE

(a) Pilot plant project

i. Preparation of cation exchange resin from cashewnut shell liquid (CNSL):- In the period under review 1,355 lbs of the resin were prepared. Conditions have been standardised for large scale polymerisation of CNSL using a kettle fabricated in the Laboratory; 70 lbs. of CNSL per batch can be polymerised and 750 lbs. of the polymer were prepared. The strength of the sulphonating acid must be maintained at 98% for obtaining resins with a good exchange capacity.

ii. Water softening :- With improvement in water supply the 2.5 cu.ft. unit was put into operation and studies on repeated regenerations are being continued. The water softening plant installed at a factory using 500 lbs. of our CNSL resin is being regularly inspected and necessary data on its performance gathered.

(b) Studies on the CNSL cation-exchange resin

i. The values of the equilibrium quotients of the resin in the hydrogen form for exchange with Li, NH_4, K, Ag, Ni and Co ions determined at 0.1 N ionic concentration are found to be 0.663, 1.295, 2.058, 3.877, 2.828 and 4.366 respectively. The column kinetics for the above ions have also been studied.

ii. Calcium-hydrogen cycle was studied using 0.1 N and 0.01 N calcium chloride solutions. The total and break through capacities are 14 and 12-12.5 kg/cu.ft. as $CaCO_3$ respectively.

(c) Synthesis

i. Cation-exchange resin: - with a view to synthesis exchangers with variable cross-linkings, work

on naphthalene and its derivatives was taken up, and resins with relatively high capacity (3.5 to 4.3 meq/g.) were prepared. An earlier preparation, however, exhibited about 73% efficiency for purification as compared to about 84% efficiency for a polystyrene-based exchanger.

Experiments in the polymerisation of styrene in presence of naphthalene, phenanthrene, anthracene and cardanol did not yield any useful results.

ii. Anion-exchange resins :- Use of commercial CNSL for preparing anion-exchange resin did not prove successful and hence other starting materials were considered. Melamine formaldehyde resins are being studied for preparation of suitable anion-exchange resins.

(d) Electrodialysis

Using a pair of permaplex cation and anion-exchange membranes a three compartment electrodialysis cell was constructed. Desalting of sodium chloride solution is being studied by varying the rate of flow under constant applied voltage.

(e) Catalysis

The catalytic constants for three exchangers were estimated and their reproducibility checked.

RUBBER

(a) Rubber-base adhesives

For a further study of the effect of compositions of resin-rubber mixes, over 20 adhesives were prepared and tried for bonding strips of aluminium and steel. Shear strengths from 600 to 950 lbs. psi were obtained.

(b) Thermally conductive rubber

Copper coils previously tested were replaced by steel coils, and heat transfer measurements were continued. A systematic study of chemical resistance of eight such mixes to 53 chemicals (b.p. above 110°) has been made at room temperature and at 60°

(c) High impact ebonite

In continuation of previous work, eleven mixes were made from natural rubber. Curing time continued to be high (15-60 minutes) and impact strengths up to 1.5 ft.lb. were obtained.

(d) Micro-cellular rubber

Blowing agents based on carbamide were prepared and tested in press-cures. Only those chemicals that have not yet been mentioned in literature are being investigated. The blowing capacities of these chemicals compare well with those of foreign proprietory materials in use.

(e) Fire-proof and flame-proof rubber

Paraffin wax in 1 lb. lots was chlorinated and tested on cotton-braided cables according to ISS:434; it passed the 10 and 30 seconds tests.

(f) Transparent rubber

The use of transparent rubber for clear tubing, pharmaceutical goods, etc. is common. To make such products, hitherto imported, a start has been made from latex, and a few samples made are fairly transparent and stand hot water treatment without discolouring. Further work is in progress.

POLYURETHANES

Flexible foams were prepared from polyesters chiefly made from sebacic acid and ethylene glycol and diethylene glycol. Castor oil alone was tried for preparing flexible foams, but without success. Blending of castor oil with polyesters, polyethers and waxes helped in giving good flexible foams.

Rigid foams were prepared from polyesters of succinic and sebacic acids, phthalic anhydride and glycerine. It was found that the use of ethyl cellulose considerably improved the pore structure of semi-rigid foams obtained from castor oil. Further work is in progress.

The pre-polymer of castor oil and toluene di-isocyanate in certain proportions gave good flexible films and coatings which have good electrical properties.

(a) Surface coatings and U-F resins

Attempts were made to improve the hardness of wrinkle finishes based on tobacco modified alkyds, but without success.

Textile bobbins coated with formulations based on modified urea resin and modified alkyds were tested by outside parties and satisfactory reports on their performance have been obtained. The large-scale preparation of the resins is being taken up to gather data for preparing a non-technical note.

(b) Utilisation of castor oil residues

Preliminary experiments suggested that the residue obtained from alkali fusion of castor oil can be used for making alkyds. The residue obtained in the cracking of castor oil was modified to obtain a good paint oil. The films obtained dried in about three hours and were colourless, non-tacky and water resistant. Both the residues will be further examined when they become available in large quantities of uniform quality.

OTHER PROJECTS

(a) Rigid porous filters

120 feet length of tube-well filters were given a finishing coat and despatched to a firm for actual field tests by their installation in a full size tube-well. These tests had to be postponed due to heavy rains and are now expected to be carried out before the end of the year.

(b) Can sealing composition

Five gallons of the can sealing composition according to the improved method were prepared and sent to two outside parties for evaluation. These were tried on high speed automatic machines and satisfactory test reports were obtained. Further quantities of the composition are now being prepared for trials by a few more interested parties. Several other tests have been conducted in the Laboratory to determine the keeping qualities of the composition under various conditions. Similar comparative tests were conducted with imported products.

(c) Foundry core oil

Work was undertaken on modification of the core oil with other fatty oils and different combinations of gum. The various samples made are being tested with regard to tensile strength, drying time and green strength.

SURVEY AND INFORMATION

REPORTING ON THE WORK CONDUCTED IN THE NCL

Reports were prepared and supplied as usual to the Secretary, CSIR, and to the Publications Directorate.

COMMERCIAL INFORMATION SERVICE

(a) Information regarding the following was collected and furnished to members of the staff : Castor seed, castor oil and castor cake; hydroxylamine hydrochloride and hydrazine hydrate; dehydrocholic acid; 2:6-ditert-butyl p-cresol; waxes; guar gum; liquid glucose and dextrose; kamala dye; sodium metal; glutamic acid and monosodium glutamate; stearic acid; PVC moulding resins and powders; gelatine; heptaldehyde and undecylenic acid; bauxite sludge; sebacic acid and octanol; core oil; tung oil; ethyl acetate.

(b) New surveys regarding prices, consumption, and availability were carried out for the following : nylon; sisal waste; fusel oil; castor oil chemicals; core oil; pepsin and rennin; lauric acid and its derivatives.

(c) Patent search was carried out for the following : vitamin C; sorbitol.

(d) Information regarding manufacturers of apparatus and equipment in India was compiled and card indexed.

(e) Import data regarding chemicals connected with the projects which are in progress in the Laboratory were compiled from daily import lists.

TECHNICAL INFORMATION SERVICE

The number of enquiries on technical problems received and attended to in consultation with other Divisions of the Laboratory was 899.

COORDINATION

Coordination of work conducted in other Divisions was continued by way of studying projects; helping in calculation of cost of manufacture in the case of the processes developed in the Laboratory; carrying out market survey; and (when required) literature survey in connection with the research projects proposed to be taken up in other Divisions.

OTHER SERVICES

(a) The Division continued to serve other Divisions by preparing photographs and drawings as required for Publications.

(b) The number of visitors shown round the Laboratory during the period under review was 1330.

II. LIBRARY

The additions made during the period were 825 books, 161 patent specifications, 37 microfilms, 16 photocopies and 4 translations. Current periodicals, numbering 718, were received regularly. The United States Book Exchange again supplied several back volumes of periodicals and serials as gifts.

Indian Patents were received in the NCL Library Patent Inspection Centre.

III. WORKSHOP

The number of job cards completed during the half-year was 1524. The following items of apparatus were fabricated: various models for the India 1958 Exhibition; sieving machine; flask gum shaker; air circulating pump similar to Borosite '0' type; furnace casing for making activated alumina, Herbig type testing apparatus; M.S. Reactor; small high-pressure stainless steel autoclave with rocking arrangement; exhaust blower similar to Gallenkamp's, capacity 1150 c.ft. per minute; shop-lifter for workshop (capacity 500 lbs.); fractionating column with condenser; magnetic stirrer; components of electro-magnet; ball mill; portable stirrer; cold water circulating unit.

Services and erection

Transfer of ice plant to a new location; services to open air Laboratory (Essential Oils Project); extension of liquid air plant; lay-out services to Room No.137 of gas, water and electricity; constant-temperature room with necessary refrigeration units.

IV. SCIENTIFIC WORK CARRIED OUT FOR OTHER INSTITUTIONS

Organic Chemistry

Microanalysis of 160 organic compounds was carried out for Universities and other Institutions. This service will have to be suspended in future because of the demands from NCL workers.

Essential Oils

On behalf of M/s. Tata Oil Mills Co., Ltd., who maintain a jasmine plantation in the NCL Estate, jasmine concrete was extracted from jasmine flowers (about 300 lbs.).

Physical Chemistry

The preparation of facsimile (electrosensitive) paper was investigated for the Meteorological Department, Government of India, Poona, and a suitable formulation was suggested.

A member of the staff of the Railway Testing & Research Centre, Lucknow, was trained in physico-chemical techniques, such as X-ray diffraction, DTA, and sorption kinetics, for studying the engineering properties of soils. The properties of a few selected soils were also investigated.

Chemically treated brass surfaces were examined by electron diffraction on behalf of the Institute of Science, Bombay.

Electron micrographs of fibres prepared under a variety of conditions were taken for the Ahmedabad Textile Industry's Research Association, Ahmedabad.

Electrical conductivity measurements of solutions of phosphor-buffers used for electrophoresis were carried out for Hindustan Antibiotics Private Ltd., Poona.

Polymer Chemistry

On a request from the Indian Standards Institution, New Delhi, a survey of the quality of indigenous phenol-formaldehyde moulding powders, was undertaken. Sixteen samples of moulding powders, both of Indian and foreign make, were examined for their electrical and mechanical properties. This work involved moulding of about 400 feet pieces of tensile strength, impact strength, surface resistivity, volume resistivity, electrical strength and power factor. A report on the values of their impact and tensile strength was forwarded to the Indian Standards Institution. This work was completed in eight weeks.

Provisional draft specifications for polystyrene moulding compositions were also prepared on behalf of the Indian Standards Institution.

Technical advice on the following was given to 24 outside parties :

Urea formaldehyde and phenol formaldehyde moulding powders; identification of nylon samples; plastics in the preparation of artificial limbs; isolation of proteins from oil cakes; surgical, insulating and pressure sensitive tapes; and vulcanisation and accelerators for rubbers.

V. NEW EQUIPMENT RECEIVED

Chemical Engineering

Rotary vacuum pump; Foster automatic temperature controller; briquetting machine; lead-lined reactors, 15 gals. and 50 gals. ; Hartwin Ram pump; Birlec arc furnace; S.S. filter press (Jacketed).

Essential Oils

Baskerville high pressure autoclave and hydrogenation equipment operating upto 300 atm./300°.

II. PATENTS

PATENTS FILED

Organic Chemistry

1. Ind. Pat. No.64958 : Improvements in or relating to polishing composition - Shah,S.M., Hinghe, V.K., Mhaskar, V.V., and Shah, R.C.

2. Ind. Pat. No.65440 : A process for the extraction of wax from sisal waste - Shah, S.M., Hinghe, V.K., Mhaskar, V.V., and Shah, R.C.

Essential Oils

3. Ind. Pat. No.64959 : A process for the preparation of dihydrojasmone - Amin, H. and Bhattacharyya, S.C.

4. Ind. Pat. No.65543 : A process for the preparation of α,ω-dicarboxylic acids and ω-hydroxy acids suitable for conversion to macrocyclic compounds, using aleuritic acid as the starting material - Mathur, H.H. and Bhattacharyya, S.C.

PATENTS SEALED

Polymer Chemistry

1. Ind. Pat. No.59608 : Porous Rigid Filters - Pandya, R.N. and Kapur, S.L.

PATENTS ACCEPTED

Polymer Chemistry

1. Ind. Pat. No.59497 : Production of porous polymer suitable for preparing cation-exchange resins - Govindan, K.P., Pandya, R.N. and Krishmaswamy, N.

2. Ind. Pat. No.59606 : Preparation of cation-exchange resin from porous cashewnut shell liquid polymer - Krishnaswamy, N., Pandya, R.N., and Govindan, K.P.

Inorganic Chemistry

3. Ind. Pat. No.63736 : On 'A liquid-liquid extraction procedure for the separation of niobium and tantalum oxide mixtures' - Sarma B. and Gupta, J.

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5. Goswami, A., "Structure of iron and chromium deposited on copper single crystals"; Trans. Faraday Soc., 54, (1958), 821.

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