# CHAPTER 2
# EXPERIMENTAL

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## 2.1 Introduction

From the view point of polymer chemistry, a polymer anchored transition metal complex catalyst is basically a functional polymer(1). It consists of two parts, a transition metal complex and a polymer. Preparation of polymer anchored catalysts involves three steps. i.e., Polymerization, attachment of a ligand by covalent bonding and finally co-ordination of metal ions to form a chelating compound. Polymerization may be performed initially, after bonding of a ligand group or after co-ordination of the metal ion as shown in Figure 2.1

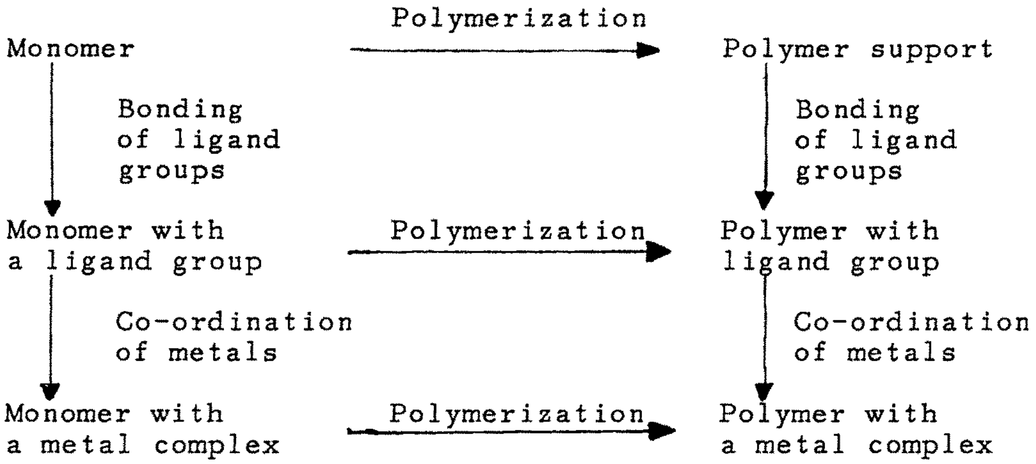


Figure 2.1 Fundamental steps in the preparation of polymer anchored catalysts.

Selection of a support is very important where specific effect of the polymer is required. Although unsubstituted styrene copolymers can bind certain transition metal complexes via $\pi$ bonding through the benzene ring (2), functionalized polymers containing ligand groups are more stable for the preparation of the anchored catalysts.

This chapter deals with the method of preparation of the macroporous polymer supports of different crosslinking, their functionalization and anchoring of the metal complexes as well as the synthesis of corresponding unbound homogeneous complexes. The methods of characterization of polymer anchored and the corresponding homogeneous catalysts using various experimental techniques such as chemical and thermal analyses, measurement of surface area by BET method, bulk density, moisture content and swelling studies have been described. The other characterization methods employed namely UV-Vis, far-IR, FTIR, NMR, EPR, ESCA and SEM have also been described. A detailed experimental set up and procedure have been given for carrying out the hydrogenation of cyclohexene and oxidation of cyclohexane at normal temperature and pressure. Kinetics of hydrogenation/oxidation has been studied by varying the parameters such as, temperature of the system, amount of the catalyst and concentration of substrate.

## 2.2 Preparation of Poly (Styrene-divinylbenzene) supports. (PS-DVB).

Polystyrene divinylbenzene has been widely used as a polymeric support because it can be prepared with different physical properties and easy functionalities. The cross linking (weight fraction of DVB) and the volume fraction Fm have a predominant effect on the texture of the porous polymers. Fm is the ratio of volume of monomer to the total volume of monomers and diluent. The pore structure and specific surface area depend mainly on the cross linking and Fm values. A series of porous polymers were prepared using suspension polymerization technique keeping Fm constant at 0.61 (3) with variation of degree of cross linking between 5 and 15.

### 2.2.1 **Synthesis of beads :**

All the chemicals used were of GR. grade except styrene (99.5%) obtained from S.D. Fine-Chem. Pvt. Ltd. Boisar (India) and divinylbenzene (DVB), 50% ethylvinylbenzene supplied by Fluka AG, Chemische Fabrik CH-9470 Buchs. The inhibitor present in styrene and divinylbenzene was removed by treating with 2% sodium hydroxide solution. It was then washed with distilled water to remove sodium hydroxide and finally dried under anhydrous sodium sulphate. Monomer mixture was prepared by adding appropriate quantities of styrene, DVB, heptane as well as benzoyl peroxide (1-15% of the monomer mixture) and was mixed thoroughly. The ethylvinylbenzene content in the divinylbenzene has been considered as styrene and appropriate corrections were made while calculating the crosslinking of polymers. This is the standard method used in the preparation of commercial ion exchange resins (4,5).

The aqueous layer of the reaction mixture was preapred by mixing (twice the amount of monomers), sodium chloride (3% salt solution and sodium laurylsulphate (0.3-0.4%) of aqueous layer. The polymerization was carried out in a four necked Quickfit polymer reaction vessel. The stirring was achieved using a motor driven stirrer fitted through a mercury seal. The temperature of the kettle was maintained in a thermostatic bath with an accuracy of $\pm$ 0.2°C using a mercury contact thermometer. The aqueous layer was initially taken into the reaction vessel and the temperature was raised to 50°C. Keeping the stirrer speed at about 20-25 rpm, the monomer mixture was run into the reaction vessel and the speed of the stirrer was adjusted to give the desired size of the beads, and the temperature was raised to 80°C. At the sticky stage which came nearly after 90 minutes, the stirring speed was raised slightly. The reaction was allowed to continue for 18 hours keeping the temperature constant at 80$\pm$2°C. After polymerization, the contents were cooled to room temperature, filtered, washed with distilled water, water-ethanol (1:1) mixture and finally with ethanol. To remove any adsorbed material soxhlet extraction was carried out for 10 hours using ethanol-benzene (1:1) mixture as solvent. The beads were finally dried at 70°C for 24 hours.

## 2.3 Chloromethylation (6)

The dried beads (10-20 mesh BSS) were taken for chloromethylation with 1,2 - dichloroethane, para formaldehyde using $AlCl_3$ as catalyst. 1,2-dichloroethane

(BDH) was distilled before use and aluminium chloride (anhydrous GR grade) was purified by sublimation. The commercially available styrene-divinylbenzene polymer beads (XAD-2 and XAD-8) were also chloromethylated using the procedure described below.

30gm of beads and 100 ml of 1,2-dichloroethane were kept in contact with each other in a three necked flask of 500 ml capacity fitted with a water cooled reflux condenser. A mixture of 18 gms of paraformaldehyde and 3 gms of $AlCl_3$ in 80 ml 1,2 - dichloroethane was added to the reaction mixture with constant stirring. The mixture was heated in an oil bath $\simeq 42^{\circ}C$. Dry HCl gas was bubbled through the system. The temperature was then raised to $80^{\circ}C$ and the reaction was allowed to continue for 20 hours. The beads were then washed sequentially with 50% aqueous dioxane, aqueous dioxane containing 10% HCl(v/v) and finally with dry dioxane until the washings were free from chloride ions.

### 2.3.1 Estimation of chlorine content

A standard gravimetric method is followed (7) to estimate the extent of chlorine content. The beads were fused with sodium peroxide and then estimating chloride as silver chloride by standard gravimetric method. A known amount of the polymer with twice the amount of sodium peroxide was fused in a platinum crucible over a gas flame, initially slowly followed by a strong heating to ensure the complete fusion of polymer with sodium peroxide. The crucible was cooled to room temperature and the mass containing sodium chloride and unreacted sodium peroxide were extracted with deionized water and filtered. The filtrate was collected in a 100ml volumetric flask and made upto the mark. A known amount of the filtrate was pipett'ed out into a beaker. The unreacted sodium peroxide present as sodium hydroxide was neutralized by the addition of dilute nitric acid. The pH of the solution was adjusted to $\simeq$ 6. The chlorine content was then determined gravimetrically as described by Vogel (7).

## 2.4 Ligand introduction

### 2.4.1 Ligand introduction on poly (Sty-DVB) supports

The chloromethylated polymer beads were kept in contact with ethanol for 30 minutes in a three necked 500 ml round bottom flask fitted with a condenser, mechanical stirrer and addition funnel. The amount of polymer, ethanol and ligand taken for the reaction are as given below.

| Crosslinking (%DVB) | Polymer (gm) | Ethanol (ml) | 1,2 diaminopropane (ml) |
|---|---|---|---|
| 5 | 20 | 150 | 10 |
| 15 | 20 | 150 | 10 |

1,2-diaminopropane was added dropwise into the reaction vessel over a period of 15 minutes using the addition funnel. The reaction mixture was refluxed for 4 hours with stirring. It was then kept for 48 hours with occasional stirring. The beads were then filtered and washed with 0.5N sodium hydroxide. It was finally washed with deionized water till the complete removal of sodium hydroxide and dried at 70°C for 24 hours.

The number of moles of 1,2-diaminopropane bound to the polymer beads were calculated from the results of microanalysis. These results were further confirmed by gravimetric estimation of chloride present as unreacted chloromethyl group on the polymer support.

### 2.4.2 **Ligand introduction on XAD-2 and XAD-8 resin**

In another series of experiments, XAD-2/XAD-8 resin supplied by Fluka AG was used as support. These beads were purified by soxhlet extraction with ethanol-benzene (1:1) mixture for a total period of about 48 hours (8) 1,2-diaminopropane was introduced onto the functionalized resin by the procedure mentioned earlier. The amount of the resin, ethanol, and the ligand taken for reaction are given below.

| XAD-resins (% DVB) | Amount (gm) | Ethanol (ml) | 1,2-diamino-propane (ml) | Refluxed time (h) |
|---|---|---|---|---|
| 2 | 25 | 180 | 10 | 8 |
| 8 | 25 | 180 | 10 | 8 |

The time of reflux was confirmed with trial experiments. The mixture was kept for 48 hours with occasional stirring. The beads were then filtered, washed with ethanol and finally with deionised water. It was then dried at 70°C for 24 hours.

## 2.5 Introduction of metal ions

### 2.5.1 Introduction of Ruthenium ions.

The functionalized polymer was kept in contact with ethanol for 30 minutes in a round bottom flask. A solution of $RuCl_3.3\ H_2O$ in ethanol taken in an addition funnel fitted to the reaction vessel was added over a period of 30 minutes. The reaction mixture was kept at room temperature for completion of reaction for 4-7 days. A change in colour of the supernatent liquid from dark orange to light orange and the beads turned to light grey is indicative of the formation of metal complex onto the polymeric matrix.

The amount of polymer beads taken, quantity of ethanol used, amount of Ruthenium chloride and time of reaction are as given below.

| Polymer type* | Support (gm) | Ethanol (ml) | $Rucl_3\ 3H_2O$ (gm) | Time (Day) |
|---|---|---|---|---|
| 5PDAP | 20 | 150 | 0.6 | 4 |
| 15PDAP | 20 | 150 | 0.6 | 4 |
| 2XDAP | 20 | 150 | 0.6 | 7 |
| 8XDAP | 20 | 150 | 0.6 | 7 |

* Nomenclature : NPDAP, NXDAP

Where N = Percent crosslink
P = Copolymer of styrene divinylbenzene.
X = XAD, Copolymer of styrene divinylbenzene.
DAP = 1,2-diaminopropane.

### 2.5.2 Introduction of Palladium ions.

The functionalized polymer was kept in contact with ethanol for 30 minutes in a round bottom flask. A stock solution of $PdCl_2$ (1%) was prepared by dissolving $PdCl_2$ in minimum amount of HCl (AR) and then diluting with ethanol. Required amount of the stock solution was added to the reaction mixture through the addition funnel - and it was kept at room temperature for 4-7 days. The colour of the polymer beads becomes golden yellow, indicating the formation of metal complex on the polymer matrix.

The amount of polymer beads taken, quantity of ethanol and amount of metal ion used as well as the reaction time are as shown below.

| Polymer type * | Support (gm) | Ethanol (ml) | $PdCl_2$ (gm) | Reaction time(Day) |
|---|---|---|---|---|
| 5PDAP | 20 | 150 | 0.5 | 4 |
| 15PDAP | 20 | 150 | 0.5 | 6 |
| 2XDAP | 20 | 150 | 0.5 | 7 |
| 8XDAP | 20 | 150 | 0.5 | 7 |

* Nomenclature.

As in the case of Ruthenium ion.

The polymer beads were filtered and washed thoroughly with ethanol. The anchored catalysts thus synthesised were dried at 70°C for 24 hours and stored in vacuum desiccator.

The metal content in each catalysts was determined by refluxing a known amount of the catalyst with conc.HCl(AR) for 24 hours and then estimating the metal ion concentration by spectrophotometric method. Thus one gram of the catalyst was refluxed with concentrated HCl(AR) for 24 hours and filtered. All the washings collected were heated to a minimum volume of the solution and the pH of the solution was adjusted to 1.8. The concentration of the solution was measured at 510 nm by spectrophotometric method using 5 Nitroso 2,4,6 Triaminopyrimidine as metal indicator. The similar procedure has been followed by Singh et al. for estimating ruthenium ions (9). In the case of palladium the concentration of the test solution was measured at 490 nm using 1-Nitroso-2-Naphthol- 3,6 Disulphonic acid as metal indicator (10).

## 2.6 Synthesis of unbound complexes

In order to study the catalytic activity in homogeneous system, unbound complexes of Ru(III) and Pd(II) with 1,2-diaminopropane were synthesised. The catalytic activity of these catalysts was seen under the similar conditions at which the polymer supported catalysts were studied.

### 2.6.1 Synthesis of unbound ruthenium complex, [Ru(III) DAP$Cl_2$]Cl

$RuCl_3.3H_2O$, supplied by (Johnson Mathey Inc. New Jersey) was dissolved in a fixed quantity of ethanol placed in a round bottom flask. Distilled 1,2-diamoniopropane

(Sisco Chemical Industries, Bombay) was added to the reaction vessel by the use of addition funnel. Ruthenium-diaminopropane ratio was maintained as 1:1. The reaction was carried out at 35°C with constant stirring for four hours. The complex thus formed was filtered, washed repeatedly with distilled alcohol and dried at 70°C. Ruthenium content was estimated by the above mentioned method (9)

| | C. | H. | N. | Cl |
|---|---|---|---|---|
| Calculated (%wt) | 12.78 | 3.55 | 9.94 | 37.82 |
| Found (%wt) | 12.62 | 3.58 | 9.83 | 37.50 |

The complex is sparingly soluble in water and insoluble in common solvents. Conductivity measurement shows ionic nature of chloride. Due to its insoluble nature, it was planned to synthesize $[RuDAP\ Cl_2]Cl$ complex catalyst in solution by taking minimum concentration of $RuCl_3 .\ 3H_2O$ and 1,2-diaminopropane in methanol in such a way that the complex should not be precipitated out and remain in solution. Hence to synthesize the complex in the solution phase, $RuCl_3.3H_2O$ ($5x10^{-4}$ mole in 100 ml methanol) was placed in a round bottom flask and DAP ($5x10^{-4}$ mole in 100 ml methanol) was added through the addition funnel with constant stirring for 4 hours at 35°C. This complex was used to investigate the catalytic activity for the reactions under study.

### 2.6.2 Synthesis of unbound palladium complex, [Pd(II)DAPCl$_2$]

The method followed was the same as in the case of ruthenium complex (vide supra). The mole ratio of palladium and diaminopropane was maintained as 1:1. The complex thus obtained was filtered, washed with distilled alcohol and Pd content was estimated (10)

| Calculated (%wt) | C. | H. | N. | Cl |
|---|---|---|---|---|
| | 14.31 | 3.97 | 11.13 | 28.24 |
| Found (%wt) | 14.35 | 3.92 | 11.02 | 27.93 |

Chemical analysis for carbon, hydrogen and nitrogen was carried out in our laboratory using Coleman carbon and hydrogen analyser and for nitrogen Duma's method was followed.

The catalysts thus synthesised were named as NPML and NXML.

Where, N = Percent crosslinking
P = Co-polymer of styrene divinylbenzene
X = XAD-2/XAD-8 polymer
L = Ligand.

Let us define

Catalyst A = 5PRu (III) DAP

Catalyst B = 15PRu (III) DAP

Catalyst C = 5PPd (II) DAP

Catalyst D = 15PPd (II) DAP

Catalyst E = 2 X Ru (III) DAP

Catalyst F = 8 X Ru (II) DAP

Catalyst G = 2 X Pd (II) DAP

Catalyst H = 8 X Pd (II) DAP

Catalyst I = [Ru(III) DAPCl$_2$]Cl
(Homogeneous)

Catalyst J = [Pd(II) DAPCl$_2$]
(Homogeneous)

The surface area and pore volume of all the supports and catalysts were measured using BET method at liquid nitrogen temperature using Carlo-Erba Sorptiomatic Series 1800 Model SO-19 (Italy) instrument.

## 2.7 Apparent bulk density

Apparent bulk density is an important practical parameter because it indicates the mass of catalyst which will pack into a reactor of specified volume. It depends on the efficiency with which a working catalyst such as grains, tablets or beads pack in space. It can be easily evaluated by the ratio of direct measurement of volume of dried catalyst in a standard graduated measuring cylinder to its weight as given below :

$$d(Abd) = \frac{\text{Weight of the catalyst}}{\text{Volume of catalyst bed}} \quad \mathrm{gml}^{-1}$$

In a typical experiment 1.0 gm of dried polymer bound catalyst was taken in a graduated corning measuring cylinder of capacity 10ml and the volume was measured when it remained constant after tapping. The experiment was repeated for five different weights and the mean was taken as apparent bulk density.

It is an important factor in the case of rigid supports but it is of little importance where the volume of catalyst bed changes when it comes in contact with the reactants / solvents.

## 2.8 Moisture content of the catalyst

In order to determine the moisture content, the catalysts were dried in an oven at $70 \pm 1^{\circ}C$ to a constant weight. Five different weights of catalysts were heated at a time and from the average of the five, the percentage of moisture content was calculated by the use of following equation.

$$\% \text{ Moisture} = \frac{A-B}{A} \times 100$$

Where A = Weight of the catalyst before drying

B = Weight of the catalyst after drying

## 2.9 Swelling studies

A fixed quantity of the catalyst (0.5gm) was taken in a glass filter tube and immersed in the solvent under study for 30 minutes at room temperature. The tube was then inserted into a 15 ml centrifuge tube closed with a teflon cap and centrifuged for fifteen minutes at 1200 rpm. The tube was weighed immediately and dried for overnight at 100°C to a constant weight. The mole percent of swelling was calculated by the use of the following equation.

$$\text{Swelling (S) (mole \%)} = \frac{A-B}{W \times M} \times 100$$

Where A = Weight of the catalyst after centrifugation

B = Weight of the catalyst after drying

W = Weight of the catalyst taken for study.

M = Molecular weight of the solvent under study.

## 2.10 Spectral characterization :

UV-Vis reflectance spectra of the solid catalysts were recorded on a Shimadzu UV-240 spectrophotometer with reference to a non absorbing standard, $BaSO_4$. The electronic spectra of the unbound complex was recorded in solution with 1cm matched quartz cells using methanol as a reference solvent.

The IR spectra of the polymer bound catalysts were recorded in the range of 250-4000cm$^{-1}$ using KBr pellets on a Perkin Elmer 883 spectrometer. FTIR of the catalysts were recorded on Perkin Elmer 1720X(USA) instrument. The NMR spectra of unbound complex $[RuDAPCl_2]Cl$ were recorded on 90 MHz Perkin-Elmer R-32 instrument.

Scanning electron micrographs (SEM) were taken on a Jeol, JSM-T300 scanning electron microscope. In order to study the SEM, the catalysts were sprayed on a double adhesive tape mounted on a brass stud and were coated with gold.

Electron spectroscopy for chemical analysis (ESCA) which is an important tool for characterization of metal complex catalysts was recorded on VG model ESCA-3 mark II (England) with the $Alk_{\alpha}$ and $MgK_{\alpha}$ as radiation sources.

Electron paramagnetic resonance (EPR) spectrum was scanned on Breaker ESP-300, X-band spectrometer using a 100 KHz field modulation.

EPR experiments were conducted on powder samples at $298^{\circ}K$ in an atmosphere of nitrogen.

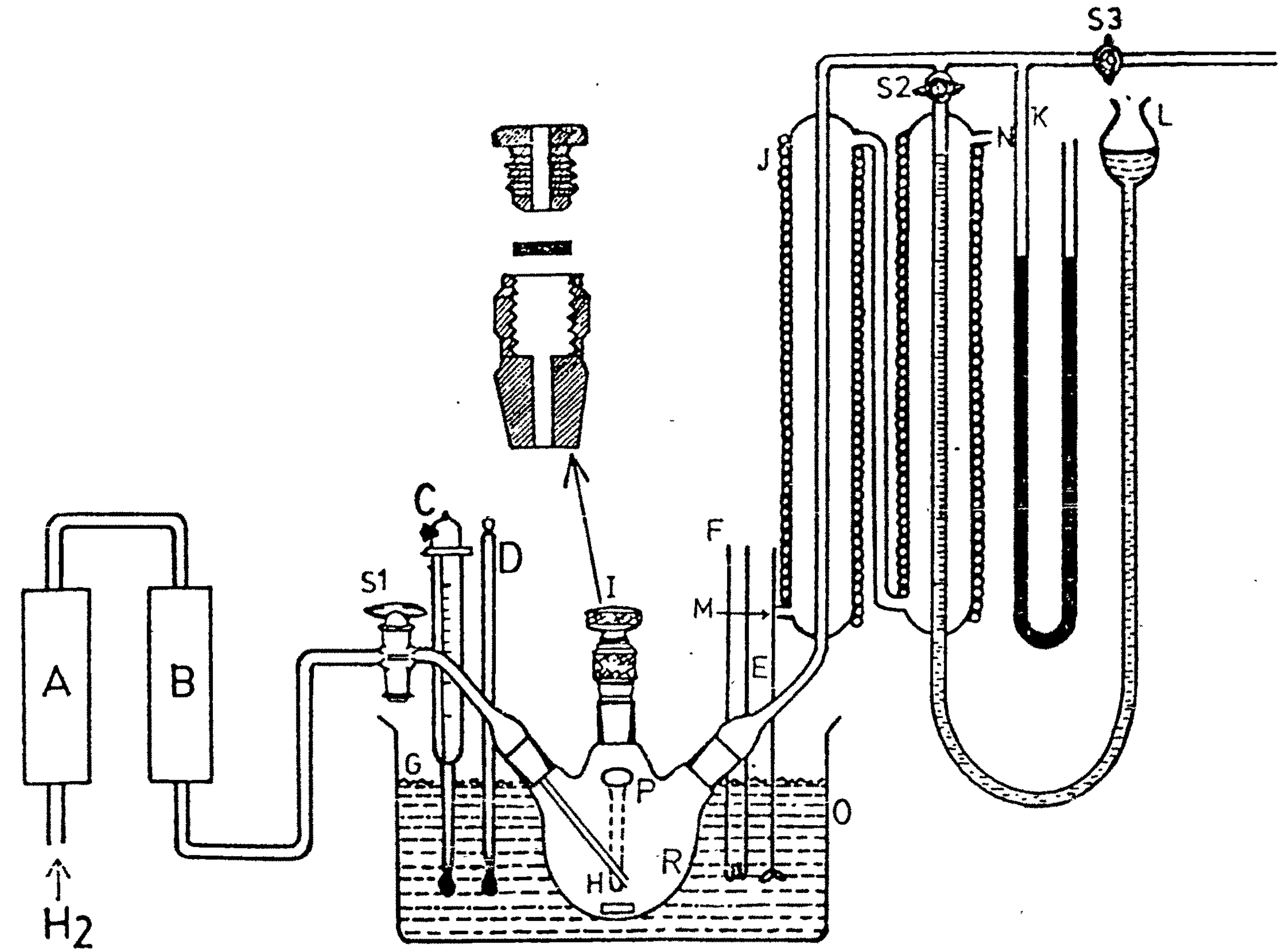
### 2.11 Thermal analysis

Thermal stability and phase change were studied in the range of $30-500^{\circ}C$ by thermogravimetry (TG) and differential thermal analysis (DTA) on Shimadzu-DT-30 (Japan) using alpha-alumina as a reference. Sample weight was recorded by programming the temperature upto $500^{\circ}C$ at a rate of $10^{\circ}C$ per minute in nitrogen atmosphere.

## 2.12 Kinetics of catalytic hydrogenation reaction

### 2.12.1 Experimental procedure

The kinetics of hydrogenation reaction was carried out at atmospheric pressure in a megnetically stirred three necked semi batch glass reactor using methanol as the reaction medium. The experimental set up is shown in Fig.2.2. A known amount of the catalyst with methanol (freshly distilled, passed through activated alumina and saturated with hydrogen at the reaction temperature) was kept in the reaction vessel. The air in the system was removed by applying vacuum, opening the stopcock $S_3$ and closing the stopcock S1 and S2. The reaction vessel was flushed with $H_2$ gas 2 to 3 times by opening stopcock S1. The air in the gas burette was removed by raising the paraffin reservoir (L) to the maximum before evacuation Adequate amount of hydrogen gas was stored in the gas burette by carefully opening the stopcock S2, when there was positive pressure in the mercury manometer (K) keeping S3 closed. By passing molecular hydrogen, the system was made saturated at the reaction temperature by starting the magnetic stirrer (H) keeping stopcocks S1,S3 closed. After saturation, stirring was stopped and the pressure in the system was adjusted to atmospheric pressure by opening stopcock $S_2$ and adjusting the level of reservoir (L). A known amount of cyclohexene was injected into the reaction vessel through the injection port (I) using hypodermic syringe. The arrangement was made in such a way that the temperature of the reactor as well as the



A) Deoxo trap B) Molecular sieve trap C) Contact thermometer D) Thermometer E) Mechanical stirrer F) Heating coil

G) Thermocole H) Magnetic bar I) Injection port J) Asbestus rope K) Manometer L) Liquid reservoir

M) $H_2O$ from thermostat N) $H_2O$ to thermostat O) China clay vessel P) Thermowell R) Reaction vessel

S1, S2, S3 - Quick fit stop cocks

Figure 2.2 HYDROGENATION/OXIDATION APPARATUS jacketed burette was maintained at 35±0.1°C. The hydrogenation reaction was initiated by starting the stirrer. The progress of the reaction at atmospheric pressure was followed by adjusting the level of reservoir (L) at various intervals of time and measuring the hydrogen uptake. Necessary corrections were applied for the vapour pressure of the solution.

## 2.13 Kinetics of oxidation reaction

The oxidation of cyclohexane was carried out using molecular oxygen. The apparatus used for oxidation reaction is the same as in hydrogenation reaction. The detailed experimental procedure is described above. The system was flushed with medicinal grade oxygen gas before injecting the substrate and the progress of oxidation reaction was followed by measuring the oxygen uptake at different intervals of time.

The products after hydrogenation and oxidation were analysed by Shimadzu GC-7AG Chromatopac CE 1B chromatograph using FID detector. For the analysis 15% carbowax on 20M chromasorb-W column was used. The typical chromatograms are shown in Figures 2.3 to 2.6 for hydrogenation. In the case of oxidation reaction, the products formed (in terms of %) calculated from the chromatograms are given in Table 2.1. A good material balance with only 1-2% deviation between $H_2/O_2$ consumed (measured experimentally) and that required according to the stoichiometry of the hydrogenation / oxidation reaction (i.e., One mole of $H_2/O_2$ required for the hydrogenation / oxidation of one mole of cyclohexene / cyclohexane) was obtained.

Table 2.1

Percentage of products formed for oxidation of cyclohexane

| Name of Catalysts | % of products<br>Cyclohexanone | Cyclohexanol |
|---|---|---|
| 15PRu(III)DAP | 11.2 | 20.4 |
| 15PPd(II)DAP | 16.7 | - |
| 8XRu(III)DAP | 12.3 | 19.8 |
| 8XPd(II)DAP | 14.3 | - |

```
CHART SPEED  0.0 CM/MIN
ATTEN:   1   ZERO:  5%

CS  3
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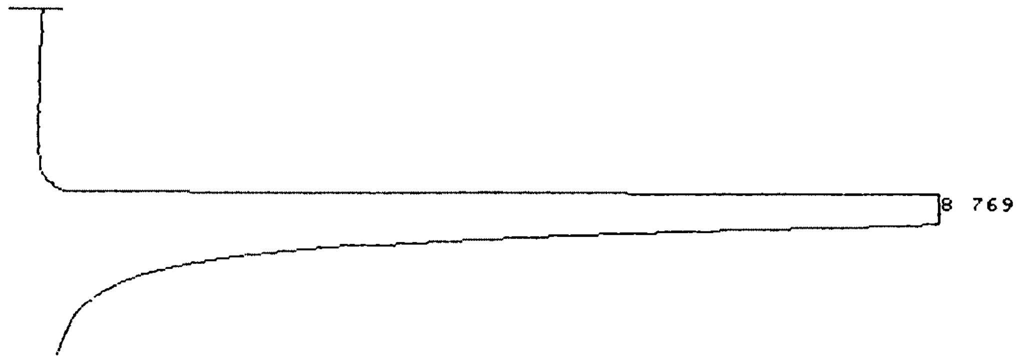


```
CHANNEL: 1B - 1    TITLE:                              16:46

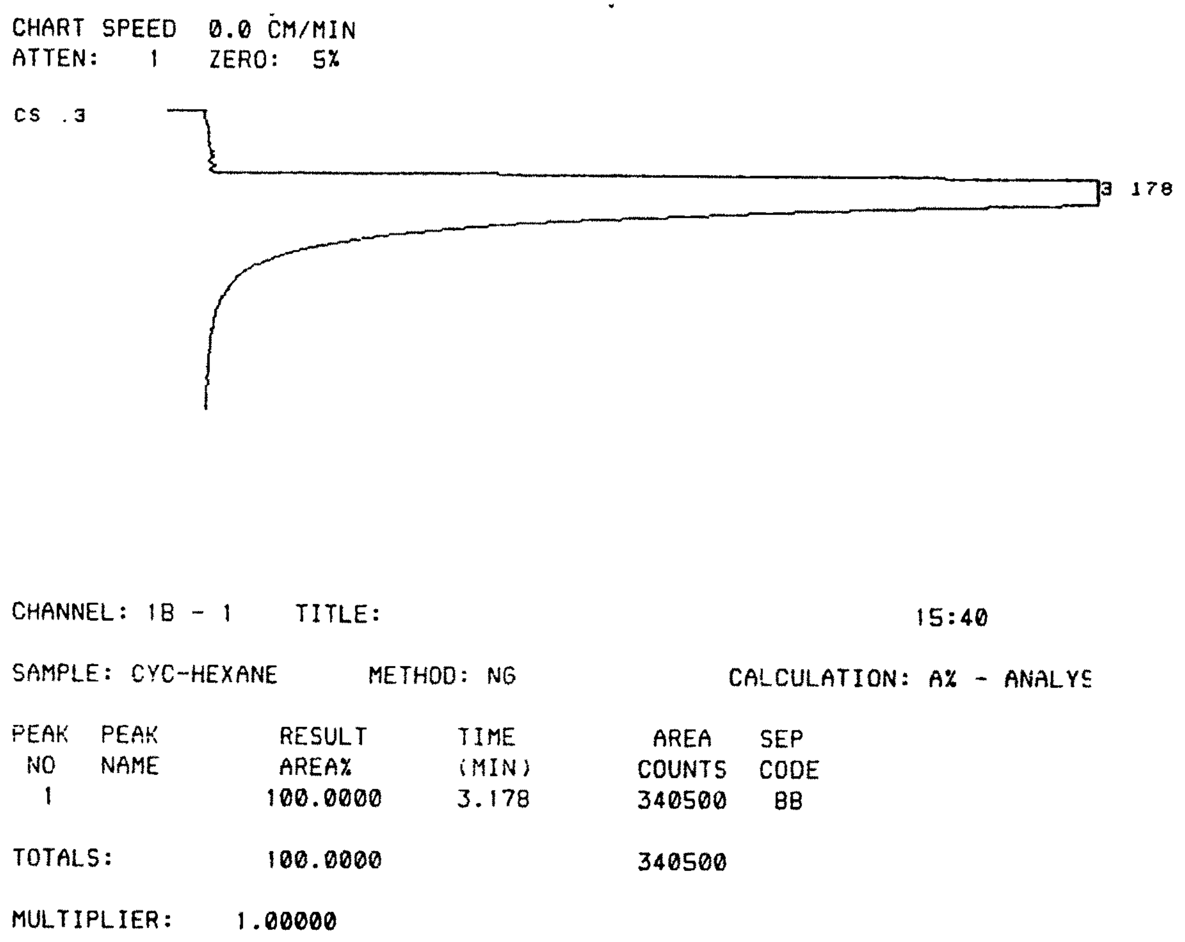
SAMPLE: METHANOL        METHOD: NG              CALCULATION: A% - ANALYS

PEAK  PEAK        RESULT      TIME        AREA    SEP
 NO   NAME        AREA%       (MIN)       COUNTS  CODE
. 1               100.0000    8.769       239036   BB

TOTALS:           100.0000                239036

MULTIPLIER:    1.00000
```

Fig. 2.3 GC Chromatogram of methanol



```
CHART SPEED  0.0 CM/MIN
ATTEN:   1   ZERO:  5%

CS .3
```

```
CHANNEL: 1B - 1    TITLE:                          15:40

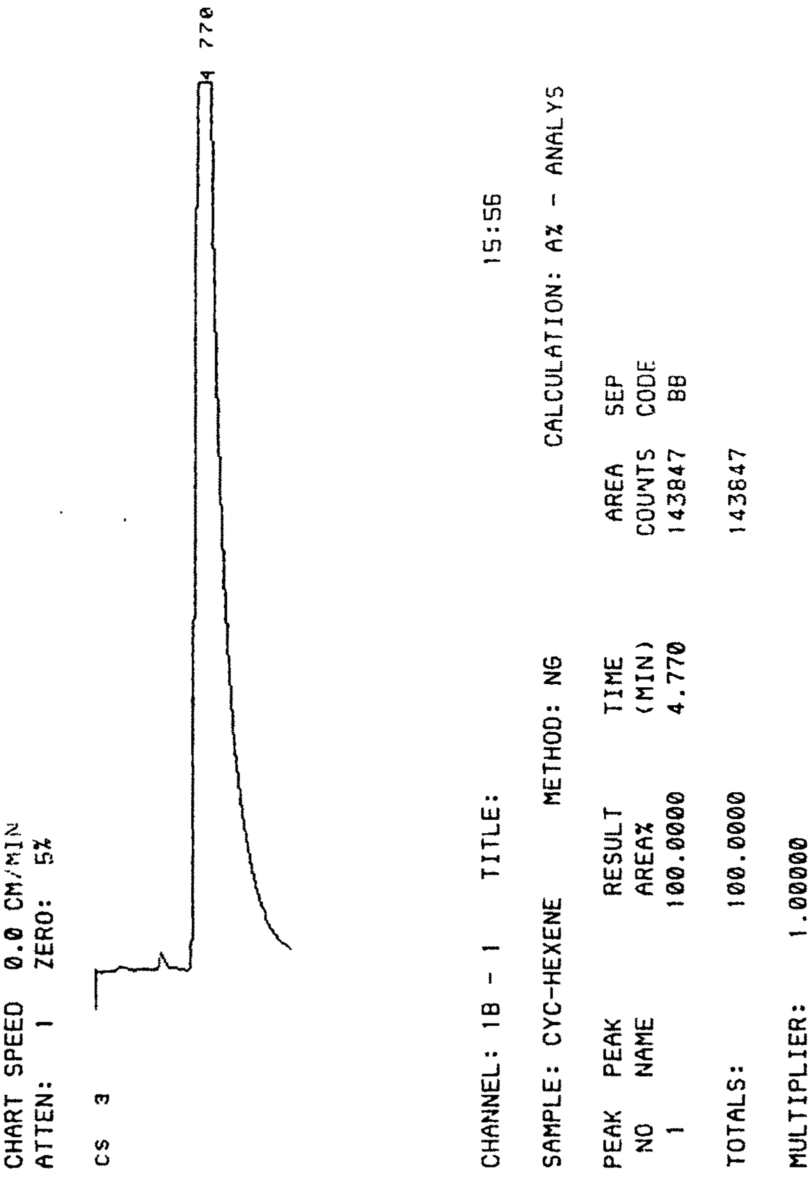
SAMPLE: CYC-HEXANE      METHOD: NG          CALCULATION: A% - ANALYS

PEAK  PEAK        RESULT      TIME        AREA    SEP
 NO   NAME        AREA%       (MIN)       COUNTS  CODE
  1               100.0000    3.178       340500   BB

TOTALS:           100.0000                340500

MULTIPLIER:    1.00000
```

Fig. 2.4 GC Chromatogram of cyclohexane



```
CHART SPEED  0.0 CM/MIN
ATTEN:  1    ZERO:  5%

CS 3

CHANNEL: 1B - 1     TITLE:                          15:56

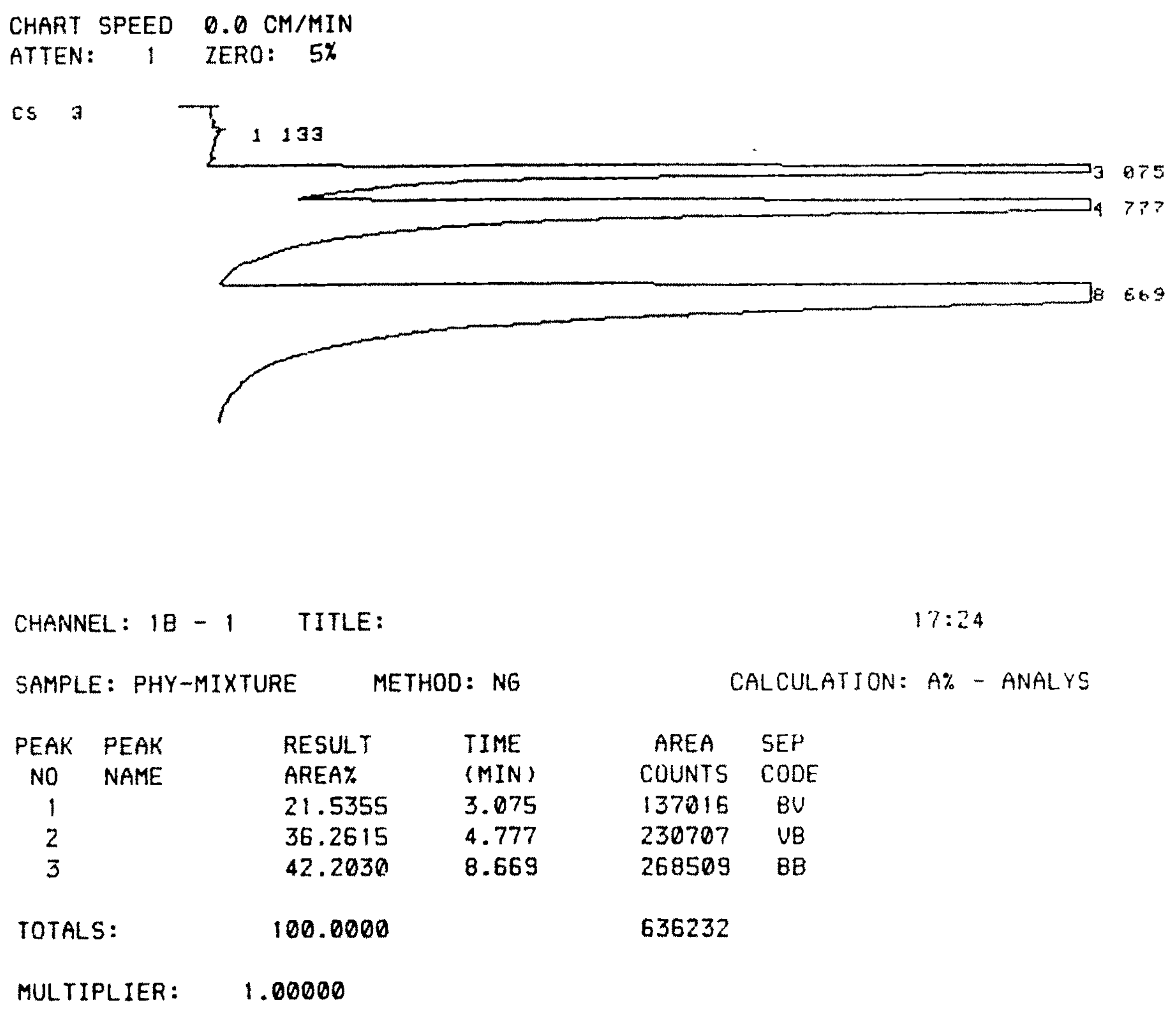
SAMPLE: CYC-HEXENE      METHOD: NG          CALCULATION: A% - ANALYS

PEAK  PEAK       RESULT      TIME      AREA    SEP
 NO   NAME       AREA%       (MIN)     COUNTS  CODE
  1              100.0000    4.770     143847   BB

TOTALS:          100.0000              143847

MULTIPLIER:      1.00000
```

Fig. 2.5 GC Chromatogram of cyclohexene



```
CHART SPEED  0.0 CM/MIN
ATTEN:   1   ZERO:  5%

CHANNEL: 1B - 1    TITLE:                                17:24

SAMPLE: PHY-MIXTURE     METHOD: NG           CALCULATION: A% - ANALYS

PEAK  PEAK         RESULT      TIME        AREA   SEP
 NO   NAME         AREA%       (MIN)      COUNTS  CODE
  1                21.5355     3.075      137016   BV
  2                36.2615     4.777      230707   VB
  3                42.2030     8.669      268509   BB

TOTALS:           100.0000                636232

MULTIPLIER:     1.00000
```

Fig. 2.6 GC chromatogram of cyclohexene hydrogenated products

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