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CHAPTER - II

EXPERIMENTAL TECHNIQUES

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CHAPTER - I I

EXPERIMENTAL TECHNIQUES

MATERIALS AND APPARATUS USED :

2.1 MATERIALS :

For all the conductivity and viscosity determinations, high purity of chemicals is required because small traces of impurities may have a large effect on these measurements. The purification of reagents used for experimental observations was done by taking following precautions.

WATER :

For conductivity and viscosity measurement, conductivity water was used. It was obtained by redistilling distilled water by using alkaline potassium permanganate in corning make quick fit distillating apparatus as described by Kraus and Dexter.<sup>95</sup> The precautions suggested by these authors were carefully observed. The conductivity water was protected from atmosphere by keeping it in a closed polythene can.

UREA :

The urea used for the experiment was of 'extra pure' grade from E.Merk Company with 98.5% purity.

LITHIUM CHLORIDE :

Unhydrous lithium chloride manufactured by SRL Company was used for experiment. Following the recommendations of Duval and Duval<sup>96</sup>, the salt was dried at 180-200°C for 6 hours and stored in a desiccator,

## 2.2 APPARATUS :

### THERMOSTAT :

A water thermostat precise to  $\pm 0.1^\circ\text{C}$  was constructed employing a mercury contact thermoregulator and electronic relay and with water capacity of about 30 liters. Thermostating of all the mixtures was normally done at least for ten minutes.

### PYKNOMETER :

The pyknometer of Ostwald modification of Sprengel<sup>97</sup> type having capacity 50 ml was used (Fig. 2.1.a). All the weights were taken on a sensitive double pan semimicro analytical balance.

### VISCOMETER :

For the measurement of viscosity, ubbelohde type viscometer<sup>98</sup> was used (Refer fig. 2.2). It is made up of glass having three bulbs A, B and C connected to 'U' shaped glass tubing 3. The bulb 'A' has two markings  $m_1$  and  $m_2$  above and below the bulb 'A'. A fine capillary is fixed between the bulb 'A' and 'C'. The flowing time of liquid depends upon the diameter of the capillary. For all the readings, the liquid was allowed to flow and time was recorded when the liquid level crosses the mark  $m_1$  and reaches mark  $m_2$ .

The bulb B was filled with liquid through tube 1 until the level of the liquid lies about between mark X and Y (not marked on the glass instrument) suction was applied to tube 2, while the top of tube 3 was closed by finger. The U tube capillary 4, and bulb A were thus filled with liquid as shown in figure 2.2 (left). When tubes 2 and 3

are opened, air passes through tube 3 into 'C' and immediately divides the liquid into two parts, as shown in figure 2.2 (right). In this way, suspended level was developed on the lower end of capillary. The liquid meanwhile begins to flow out of bulb A, through the capillary. However, it does not again fill 'C' but flows in a thin layer down its vertical wall and joins the liquid remaining in 'g' and B. The time was determined during which the meniscus of the liquid drops from mark  $m_1$  to mark  $m_2$ . A liquid of 10 ml was used in viscometer for each reading.

#### STOP WATCH :

A 'Racer' make stop watch reading to one tenth second was employed in measuring the time flow of the solution in the viscometer. The watch was wound up tightly and then allowed to run for a definite period before being used and was always handled in systematic manner.

#### GLASS APPARATUS :

Corning make glass ware was used such as burette, beakers of 250 ml and 100 ml capacity. Pipette and volumetric flasks of 250 ml capacity were also used.

### 2.3 METHODS AND PROCEDURES :

#### PREPARATION OF SOLUTIONS :

LiCl solutions were prepared at round concentrations of 0.1, 0.2, 0.3, 0.4, 0.5, 0.75, 1.0, 1.5 and 2.0 M in urea + water mixtures

at 0%, 5%, 10%, 15% and 20% by weight of urea solutions. The solutions were kept in stoppered bottles to prevent evaporation.

Lithium chloride is highly hygroscopic so its standard solution cannot be directly prepared. First round about concentration was made ( $\approx 2$  M). The exact concentration was calculated by titrating this solution with standard  $\text{AgNO}_3$  solution using  $\text{K}_2\text{CrO}_4$  as an indicator.  $\text{AgNO}_3$  was standardized by using standard  $\text{KCl}$  solution and using  $\text{K}_2\text{CrO}_4$  as indicator. Thus 2.0 M standard  $\text{LiCl}$  was prepared. The remaining concentrations (1.5 to .1) were prepared by diluting appropriate volume of standard (2M) solution. For dilution, mixture of urea + water was used as a solvent.

#### DETERMINATION OF DENSITY :

The exact density was calculated by using pycnometer of Ostwald's modification of the Sprengel.<sup>97,99</sup> The pycnometer was cleaned by washing well with distilled water and then successively, with a small quantity of purified acetone. A current of dry air was drawn through the tube for drying purpose.

The weight of pycnometer was taken. For this purpose, it was suspended from the end of the balance beam by means of a double hook (Fig. 2.1 b) made of copper wire. The pycnometer (Fig. 2.1.a) was then filled with distilled water by attaching a piece of rubber tubing to the end B, and sucking gently while end A dips in water.

The pycnometer was then suspended in the bath by means of a wire hook and placed over bracket made from sheet of copper (Fig.2.1(c)).

In the sheet of metal a hole was cut, which allowed the body of the pyknometer to pass through, while the arms were resting against the ends of the hole. The length of the opening was such as to allow the pyknometer to pass so far through that the mark on the tube B of the pyknometer was just above the metal plate, and the water in the bath was adjusted to such a height that it just touched the underside of the plate. By means of such arrangement, the danger of water getting into the ends of the pyknometer tubes was avoided and the pyknometer was held in position more accurately than by hooks.

The pyknometer was then thermostated for about 10 minutes. The amount of water was adjusted so that it filled the pyknometer from the point A to the mark on B. (fig.2.1.a). If there was too little water, a rod or tube carrying a drop of water was placed against the end of the tube A, when water was drawn into the pyknometer by capillarity. If there was too much water, a piece of filter paper was carefully placed against the end of A, whereby water could be drawn from the pyknometer until the meniscus stood exactly to the mark on B.

The pyknometer was then removed from the bath and carefully dried by means of a cloth, taking care that none of the water was expelled from the pyknometer by heat of the hand or by the natural expansion of the liquid when the density was being determined at temperatures below that of room. When the pyknometer had taken the temperature of the balance case, it was weighed.

When the weight of pyknometer filled with water had been determined the pyknometer was emptied and dried and filled with the liquid,

the density of which was required. It was placed, as before, in the bath at constant temperature. The liquid was adjusted to the mark, the pyknometer dried with a cloth as described previously and weighed.

If the temperature at which the pyknometer is filled with water and with the other liquid is the same, then the relative density (uncorrected for the buoyancy of the air) can be calculated by the ratio of the weight of liquid ( $W'$ ), of the liquid compared with that of water at the same temperature.

$$d_t = W'/W$$

Densities of solutions were measured at 20°C, 25°C, 30°C and 35°C temperatures.

#### DETERMINATION OF VISCOSITY :

Viscosity determinations were carried out by making use of an ubbelohde type viscometer. The viscometer was rinsed with distilled water and dried with acetone. The interior glass surface was kept clean. 10 ml of the sample was employed for each measurement. After thermostating the solution in the viscometer itself for 10 minutes (fig. 2.3). Viscosity magnitudes of the samples were estimated using the density values in the following equation.

$$\eta_1 = \frac{\eta_0 d_1 t_1}{d_0 t_0}$$

in which  $\eta$  was the viscosity, 't' the time taken in seconds for the flow of equal volumes of experimental sample and water and suffixes 0 and 1 stood for the magnitudes of water and experimental sample respectively.

In the above expression, the density<sup>100</sup> and viscosity<sup>101</sup> of water at temperatures 20°C, 25°C, 30°C and 35°C used were as follows:

#### DENSITY AND VISCOSITY OF WATER

Temp °C	Density (kg m <sup>-3</sup> )	Viscosity (mNSm <sup>-2</sup> )
20	998.235	1.005
25	997.073	0.8937
30	995.674	0.8007
35	994.06	0.7225

#### 2.4 CONDUCTIVITY MEASUREMENTS :

Toshniwal 'Digital conductivity meter' Type CL01.10A was used for precision conductivity measurements, having high sensitivity.

#### CONDUCTIVITY CELL :

The conductivity meter can be used with different types of cells depending upon the use.

We used 'immersion on dip type cell'. It is constructed of glass with platinized platinum electrodes fused to glass strip for support. The cell constant was predetermined and marked on the cell. This is general purpose cell for immersion measurements in laboratory. The cell constant of the cell was 0.87.



## 2.5 OPERATING PROCEDURE :

The instrument was switched on and allowed to warm up for about 15 minutes for satisfactory operation of the instrument.

### STANDARDIZATION OF THE INSTRUMENT :

Before use, the conductivity cell was thoroughly cleaned with conductivity water and was dipped into the conductivity water for about 24 hours.

A standard solution whose conductivity is accurately known at measuring temperature was taken. 0.1 M KCl solution was taken. The conductivity of this solution was 1408.8 micromhos/cm at 25°C.

The 200 Hz Oscillator frequency was selected for conductivity measurements.

The instrument was standardized with this standard solution and the instrument thus standardized was used to determining the conductivities of unknown solutions.

### MEASUREMENT OF UNKNOWN CONDUCTIVITY :

The cell wires were connected to the 'CELL' terminals and the cell was dipped into the solution under measurement. With proper range selection, the conductivity of unknown solution was determined at that temperature.

## 2.6 PREPARATION OF STANDARD SOLUTION :

The standard solution of KCl was prepared by weighing accurately 0.745526 gms. of KCl crystals (Analar grade) and dissolved in 1000 ml conductivity water.

The conductivity of this solution was 1408.8 micromhos/cm at 25°C.

Following table gives the conductivity in mhos per centimeter of KCl solution for standardization.

0.74556 gms of KCl per 1000 ml of water

Temp °C	Conductivity ( mhos/cm )
20	0.0012731
25	0.0014088
30	0.0015466
35	0.0016425

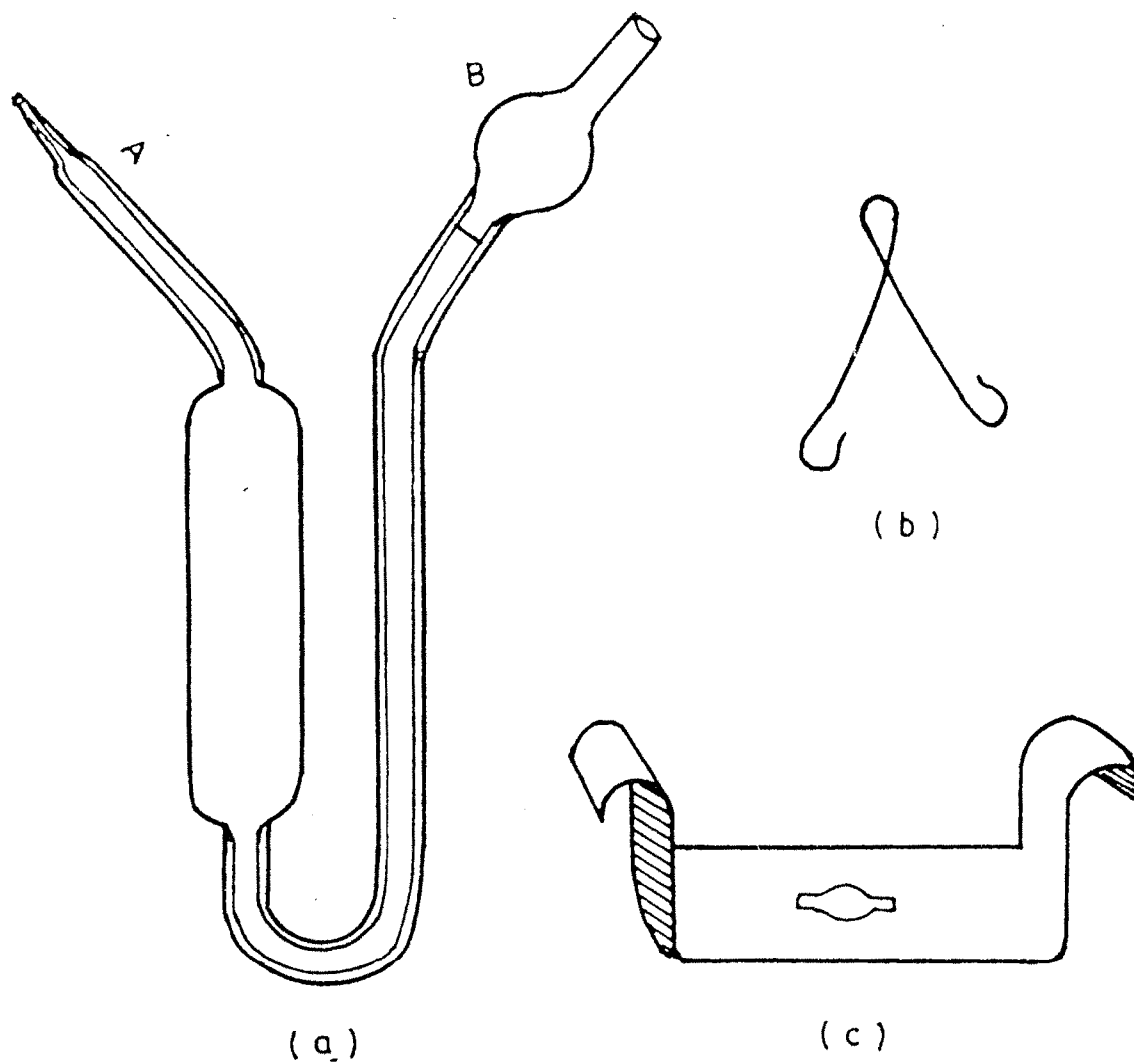


FIG.2.1 - PYKNOMETER FOR LIQUIDS .

(a) PYKNOMETER VESSEL , (b) WIRE SUPPORT  
FOR WEIGHING , (c) BRACKET FOR SUPPORTING  
PYKNOMETER IN THERMOSTAT .

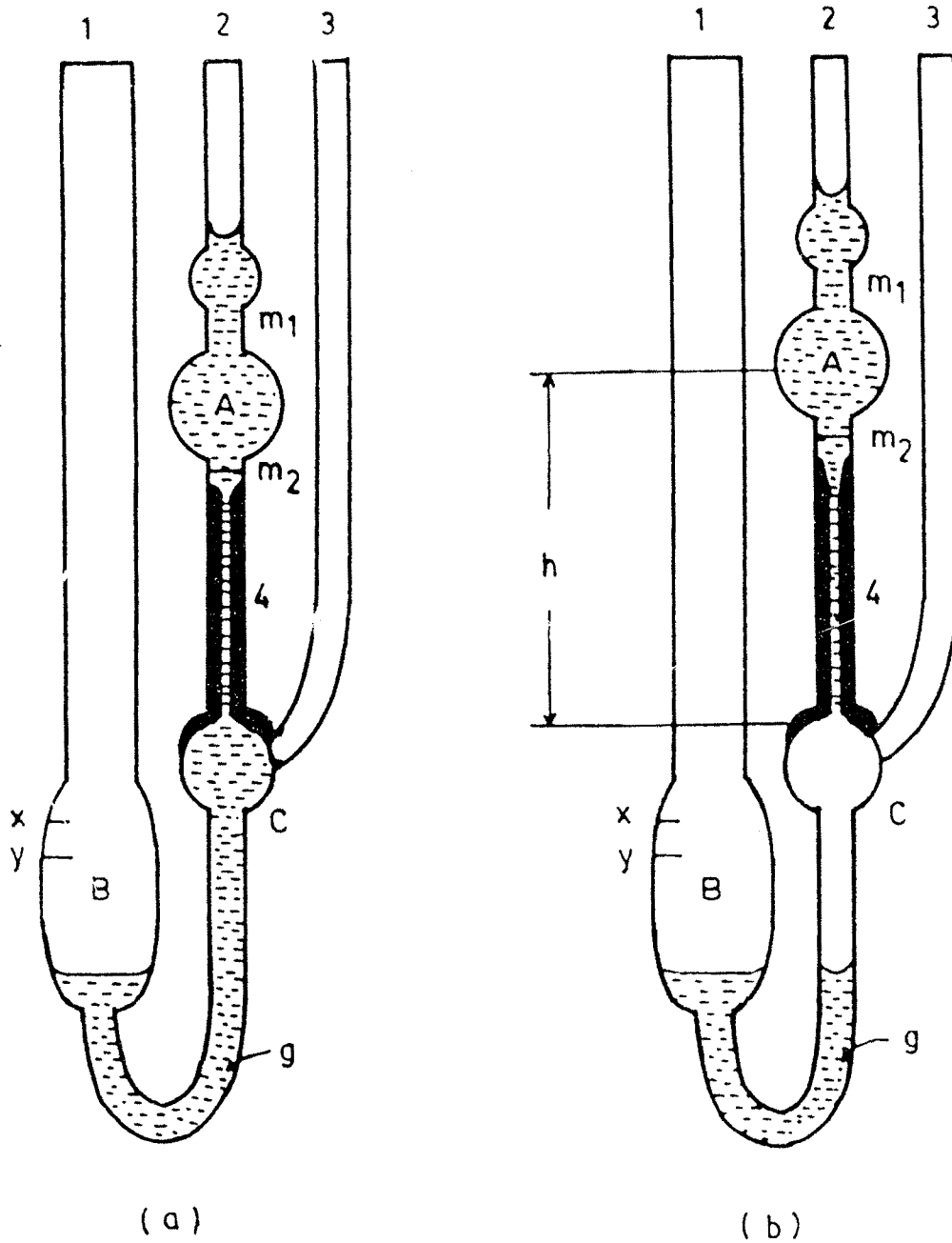


FIG. 2·2 — UBBELOHDE TYPE VISCOMETER .

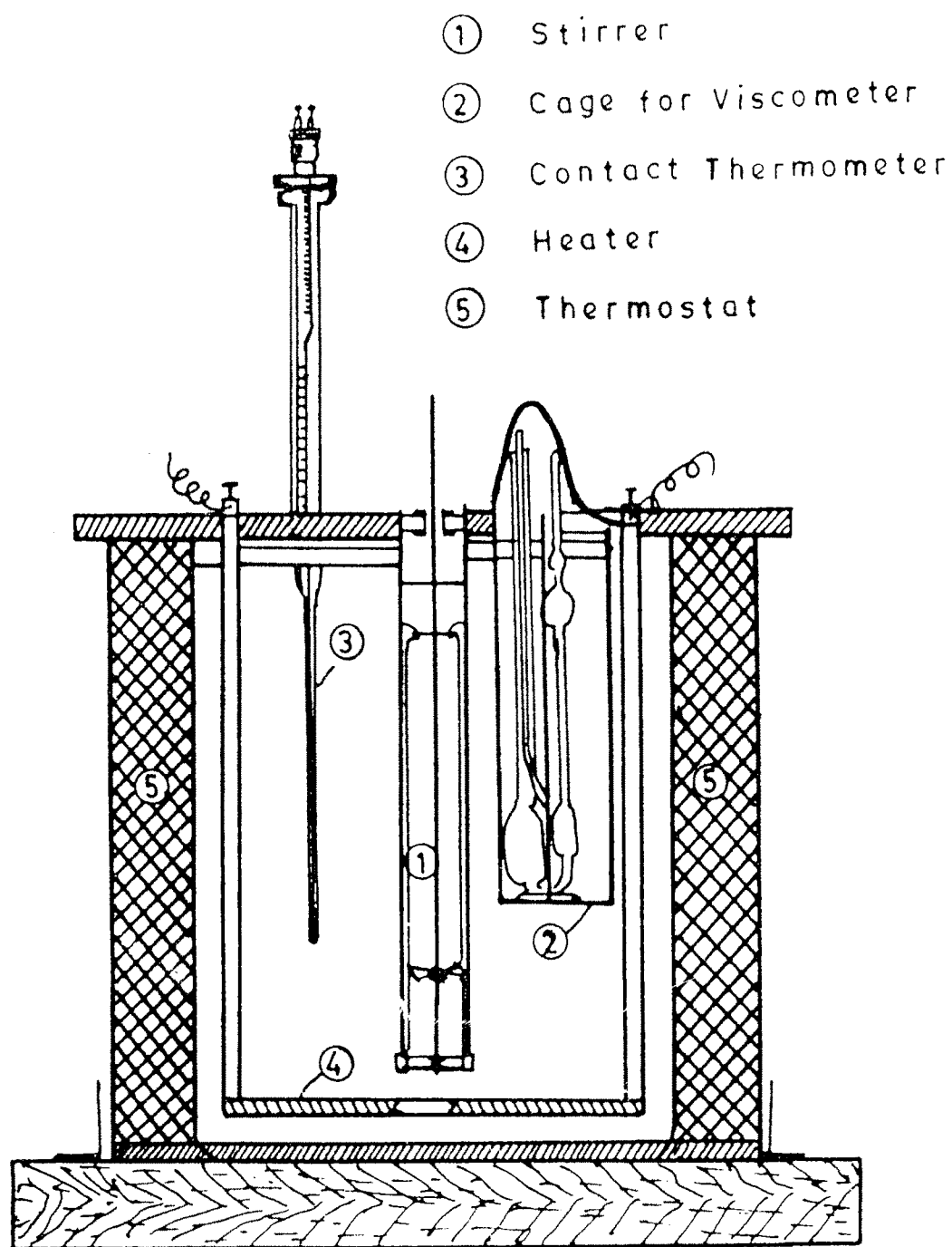


FIG. 2-3 — EXPERIMENTAL ARRANGEMENT.