

CHAPTER 2

EXPERIMENTAL

2.1 MATERIALS

The quality and the source of procurement of each chemical used throughout the present work are given below :

- (i) Toluene, BDH (India), AnalaR grade, Chemicals division, Glaxo Laboratories (India) Ltd., Bombay - 25 (India).
- (ii) Ethylbenzene, MERCK-Schuchardt, Schuchardt, 8011 Hohenbrunn bel Munchen.
- (iii) Bromobenzene, E. Merck (India) Pvt. Ltd. Worli, Bombay - 400 018 (India).
- (iv) 1-Hexanol, BDH (England), AnalaR grade, BDH Chemicals Ltd., Poole, England.
- (v) 1-Octanol, Entzundlich, Gesundheitschadliche Dampfe, FERAK BERLIN, West Germany.
- (vi) Water

Water used was double distilled conductivity water. In order to attain required purity, ordinary laboratory distilled water was slowly redistilled after addition of alkaline potassium permanganate in a pyrex glass distillation assembly. The individual units of the distillation assembly were thoroughly cleaned with warm chromic acid, then rinsed several times with distilled water and finally dried in air oven free from dust particles. Necessary precautions were taken to keep

all the ground glass joints meticulously clean. In order to avoid any surface contamination, the first and the last fractions of the distillate were discarded.

The specific conductivity of the double distilled water, obtained as above, was measured by Toshniwal conductivity bridge type C₁₀ 1/01A, working at 50 C/S frequency and using a dipping cell with cell constant 0.971. Water having specific conductivity less than $1 \times 10^{-6} \text{ ohm}^{-1} \text{ cm}^{-1}$ at $30 \pm 0.1^\circ\text{C}$ was retained for use. The purity of water was further checked by measuring its refractive index and density at $30 \pm 0.1^\circ\text{C}$. Extra precautions were taken to store the conductivity water in clean and well stoppered Pyrex flasks. The purity of stored water was always checked by measuring its refractive index before use.

2.2 PURIFICATION OF THE LIQUIDS USED

In each case, the purity of liquids used was checked by the measurement of density, viscosity and refractive index at 25°C . Suitable purification operations involving fractional distillation and drying were performed for Toluene^{1(a)}, ethylbenzene^{1(b)}, bromobenzene^{1(c)}, 1-hexanol^{1(d)} and 1-octanol^{1(e)} and summarised as below.

<u>Chemical</u>	<u>Distilled fraction collected at</u>	<u>Drying agent</u>
toluene	110.6 \pm 0.5 $^\circ\text{C}$	P ₂ O ₅
ethylbenzene	136.2 \pm 0.5 $^\circ\text{C}$	anhydrous MgSO ₄
bromobenzene	156 \pm 0.5 $^\circ\text{C}$	P ₂ O ₅
1-hexanol	157.0 \pm 0.5 $^\circ\text{C}$	Aluminium amalgum
1-octanol	195.2 \pm 0.5 $^\circ\text{C}$	Boric anhydride

2.3 TEMPERATURE CONTROL

The temperature was controlled in a Toshniwal thermostat, Cat. No. GL 05 with the help of several boosters as well as small heaters depending upon wattage requirements. The booster heaters were controlled by the autoboster control point of the mercury regulator. When the bath was set, the temperature variations were limited to 0.1°C .

2.4 PREPARATION OF MIXED BINARY AND TERNARY SYSTEMS

Pure components to form a binary and a ternary system were taken separately in glass stoppered bottles and thermostated at the desired temperature for sufficient time. When the thermal equilibrium was ensured, the required volumes of each component were transferred and weighed in a different bottle which was already cleaned and dried thoroughly. It was then stoppered and the mixed contents were shaken well before use. While preparing different binaries and ternaries care was taken to ensure that the same procedure was adopted throughout the entire work.

2.5 MEASUREMENT OF DENSITY

The densities of selected pure liquids were determined at different temperatures following the standard experimental procedure^{2(a)} by measurement of the weight of liquid occupying a known volume in a Weld-type pycnometer, care being taken to prevent evaporation of the volatile liquid. The pycnometer was first cleaned, dried carefully, weighed and then filled with distilled water and placed in the thermostat set at required

temperature for 20 minutes. When temperature equilibrium was reached, any excess liquid on the tip of inserted plug was wiped off with a piece of filter paper, care being taken to avoid removing liquid from the plug capillary in the process. The pycnometer was then removed from the thermostat, wiped, dried with a cotton cloth and allowed to stand in the balance case for few minutes and finally weighed. In this fashion, repeat determinations were made of the weight of liquids and liquid mixtures. More than one pycnometer were used to advantage so that while one was weighed, the others were in the thermostat.

2.6 MEASUREMENT OF VISCOSITY

If the capillary-flow of a Newtonian incompressible fluid is laminar and steady then with certain assumptions the viscosity η is given by

$$\eta = \frac{\pi \Delta P r^4 t}{8 V' l} - \frac{\rho v'}{8 V' l t} \quad (2.1)$$

where ΔP is the driving pressure, r is the radius and l is the length of the tube and V' is the total volume which flows past any cross section in time t . Applied to the Ostwald Viscometer in which a liquid of density ρ flows through a height h , equation (2.1) becomes

$$\eta = A \rho t - B \frac{\rho}{t} \quad (2.2)$$

where $A = \frac{\pi r^4 g h}{8 V' l}$ and $B = \frac{V'}{8 \pi l}$

The values of viscometer constants A and B can be obtained by determining the flow time t experimentally for atleast two liquids whose viscosity values are known. However, with appropriate choice of capillary dimensions in Ostwald Viscometer, the term $B \frac{\rho}{t}$ can be made insignificant in view of the other experimental errors and assumptions involved, giving

$$\eta = A \rho t \quad (2.3)$$

Further if η_1, ρ_1, t_1 and η_2, ρ_2, t_2 are the viscosity, density and time of flow in the same Ostwald Viscometer for two liquids 1 and 2 respectively, eqn.(2.3) gives

$$\frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2} \quad (2.4)$$

Thus knowing viscosity, density and time of flow for a reference liquid, the viscosity of an unknown liquid or liquid mixture can be calculated provided the density and the time of flow for the latter is known.

Using eqn.(2.4) and following the standard procedure^{2(b)}, the viscosities of selected pure liquids as well as their binary and ternary mixtures were measured at different temperatures by Ostwald Viscometer. Before starting the actual experiment, the viscometer was thoroughly cleaned, dried and calibrated with double distilled conductivity water. The time of flow was found to be reproducible within 0.1 second for each measurement. The viscosity values obtained for liquids compared satisfactorily with their literature values.

NOMENCLATURE

A	constant eqn. (2.12)
B	constant eqn. (2.12)
h	height
l	length of the tube
ΔP	driving pressure eqn. (2.1)
r	radius of the tube, eqn.(2.1)
t	time of flow, eqn. (2.1)
v'	total volume which flows past any cross section in time t, eqn. (2.1)
η	coefficient of viscosity 'cP', eqn. (2.1)
ρ	density of the liquid

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