3.1 Equilibrium Still

A modified version of the equilibrium still with circulating vapour phase described by Rao and coworkers [219] was used to obtain equilibrium vapour and liquid samples. Figure 1 shows schematically the details of the equilibrium still along with its accessories. Figure 2 is an overall photograph of the equilibrium still and the associated equipment. Essentially the still consists of the boiler, B, with a nozzle through which the recirculating vapours enter the boiler thereby
FIG. 1 SCHEMATIC DIAGRAM OF THE EXPERIMENTAL SET UP

(SCALE, 3:10)
Fig. 2 Overall Photograph of the Equilibrium Still and Associated Equipment
providing thorough agitation to the boiling liquid, entrainment trap, E, the condensate receiver, R, with a nozzle arrangement similar to that of the boiler providing agitation to the condensate, the condensate return leg, L, and the condensate total vapouriser, V. \( K_1 \) and \( K_2 \) are the sampling cocks for the liquid and condensed vapour samples respectively. C is the condenser and F is the feed mixture inlet. An air jacket is provided over the vapour space of the boiler with a 125 watt compensating heater to minimise heat losses preventing refluxing in the boiler tube. Figures 3 and 4 show close-up photographs of the equilibrium still (shown without the insulation material) and the condensate receiver respectively. 125 watt heating cords with glass fibre insulating sleeves were used to supply the energy to the boiler and total vapouriser. The energy supply is controlled separately for each heater by the use of autotransformers and 'Sunvic' energy regulators. A voltage stabiliser was used to ensure steady 230 volt supply from the mains. A combination of glass wool and asbestos cloth was used for insulating the main parts of the still like the boiler, vapouriser, air jacket and entrainment separator. The boiling tube, vapouriser and condensate receiver were filled with 3 mm ceramic insulation beads to two-thirds of their volumes. In order to ensure accurate measurements of temperatures, minimising surface heating of mercury from the neighbouring heater element turns, the thermometer pockets were constructed with double walls.
Fig. 3 Close-up Photograph of the Equilibrium Still
Fig. 4 Close-up Photograph of the Condensate Receiver
(cf Figure 1). The capacity of the boiler is about 80 ml while that of the condensate receiver is about 20 ml. A coiled type condenser was used to condense the vapours. The vapour space of the condenser was connected to a train of U-tubes containing anhydrous calcium chloride to avoid moisture absorption by the system.

3.2 Measurement and Control

Anschütz short range thermometers graduated to 0.1°C were used for measuring the equilibrium temperatures. These thermometers were calibrated against a precision mercury thermometer standardized against ice point and normal boiling point of distilled water. The accuracy of the temperature measurements was of the order of ± 0.1°C. The calibration and the accuracy of these thermometers were checked at intervals during the period of experimental runs. Stem corrections were applied to temperature readings [88].

For each experimental run, the atmospheric pressure variation was recorded with a standard mercury barometer with an accuracy of ± 2 mm of Hg. Temperature corrections for the expansion of the mercury and the scale of the barometer were applied to the barometric readings [88]. Data on all the systems, except the acetic acid - ethylbenzene and acetic acid - p-xylene, were obtained at 760 ± 2 mm of Hg pressure.

Air for maintaining the pressure in the still was supplied by a fractional horse power boiler. Two bubblers, H, (Fig.1) containing concentrated sulphuric acid along with two U-tubes with anhydrous calcium chloride were used in the line to absorb moisture.
from the air entering the system. The air pressure was adjusted to 760 mm of Hg by means of pinch cock PC on a rubber tubing open to atmosphere. Surge vessels, $S_1$ and $S_2$, with a total volume of about 40 litres were provided in the line to minimise the fluctuations in the pressure. A fine needle valve, $V_1$, was used for fine control of the pressure which was read on the barometer at every 20 minute intervals during each run. To keep the pressure drop minimum between the still and the open mercury manometer, $M$, the distance between them was kept to the minimum. A magnifying lens was used in taking the readings of the thermometers, manometer and barometer.

The data for the systems, acetic acid - ethylbenzene and acetic acid - p-xylene were taken at the prevailing atmospheric pressure of 725 mm of Hg. The variation of pressure inside the apparatus was mainly due to the fluctuations in atmospheric pressure, which were very small. Though the pressure variations were different in each reading, the average variation never exceeded three mm of Hg during the period of experimental runs for these two systems. The pressure drop inside the apparatus was considered negligible and the errors introduced thereby were within the accuracy of the experimental measurements.

3.3 Operating Procedure

About 100 ml of liquid mixture consisting of approximately known volumes of each of the pure components were added to the still. The liquid level submerged the lower thermometer pocket. For each run, such mixtures were prepared by progressively
increasing the amount of one of the components while decreasing the other. This procedure was repeated for all the binary mixtures.

After adding the liquid mixture to the still, the condenser was turned on and the load on the heater of the boiler was gradually increased to heat the liquid to boiling at approximately 0.5°C per minute. As the liquid temperature attained a constant value, the load on the heater on the vapour tube was increased until the temperature read by the vapour thermometer was about 0.5°C more than that read by the liquid thermometer. At this stage the blower was switched on and the pressure was adjusted to 760 mm of Hg with the help of the pinch cock. Finer control was obtained with the help of the needle valve. At the start of the condensate overflow the vapouriser heater was switched on and its load increased gradually till the entrainment in the vapouriser outlet tube disappeared. As the bubbling of vapours through the liquid started in the boiling chamber, the load on the boiler heater was reduced gradually to stabilise the rate of bubbling in both the boiler and condensate receiver. The adjustment of the load of the heaters on vapouriser and boiling tube was essential for uniform and fluctuation-free working of the still. This condition could be achieved with a little experience.

Though steady state could be attained in about 30 minutes in this still, three hours time was allowed after the start of stable recirculation to ensure perfect equilibrium between the two phases. During this period the temperature and pressure readings were taken at intervals of 20 minutes.
At the end of three hours small quantities of the liquid over the sampling stopcocks were drained off and the equilibrium samples were collected in sampling tubes which were kept in cold water to minimise evaporation losses during collection. The tubes were immediately stoppered. The heaters and the blowers were turned off and the contents of the still were drained off.