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XLVI. *On the Mutual Solubilities of Diethylamine and Water.*

By ROBERT TABOR LATTEY, B.A.*

PAIRS of liquids which exhibit partial miscibility are usually divided into three classes:—(1) Those which are completely miscible above a certain temperature and only partially miscible at lower temperatures; (2) Those which are only partially miscible at all observed temperatures; and (3) Those which are completely miscible below a certain temperature above which they become partially miscible.

The only cases of the third class which have been at all fully studied are: sym-trimethylpyridine and water, triethylamine and water (Rothmund, *Zeit. Phys. Chem.* xxvi. p. 433, 1898), diethylamine and water (Guthrie, *Phil. Mag.* xviii. p. 500, 1884) †.

The case of nicotine and water recently studied by Hudson (*Zeit. Phys. Chem.* xlvii. p. 113, 1904) forms an interesting combination of cases (1) and (3) since these liquids are completely miscible below 64° or above 205°, but between these temperatures their mutual solubilities are only partial; the solubility-temperature curve representing these phenomena is a closed elliptical figure.

This work was originally undertaken with the idea of extending Guthrie's curve showing the relation between temperature and the miscibility of diethylamine and water. It was soon found that either Guthrie's results were incorrect or that some unexpected influence was raising the temperature of separation. The results obtained at first were not only in disagreement with Guthrie's, but were not in concordance with one another. At length the behaviour of one of the tubes (which happened to contain 58·6 per cent. of the amine) furnished a clue to the difficulty; the contents of this tube separated into two layers at 140°·2, after being cooled to about 130° and mixed they separated at 138°; a second and third repetition of the observation gave 137° and 135° respectively. Obviously some change was taking place in the contents of the tube: this might either be due to an action of water on diethylamine at high temperature or to the solution of glass. Experiment showed that about 1 mgrm. of solid had been taken up by the liquid. Experiments were therefore begun in tubes of Jena glass; the results so obtained were concordant, and measurements made on individual tubes did not differ from one another more than observations of a melting-point usually differ.

* Communicated by the Author.

† The case of dimethylamine and water, given by Prof. van't Hoff in his "Lectures on Theoretical and Physical Chemistry," is a misprint. (Private communication.)

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The results obtained were as follows :—

Per cent. amine.	Temperature of separation.	Per cent. amine.	Temperature of separation.
21.73	154.5	39.63	143.5
22.65	151.7	45.78	144.15
25.04	147.5	48.89	145
25.06	147	51.71	146.8
28.60	144.4	54.24	148.5
30.93	144	54.78	150.3
34.03	143.2	58.59	152.3
38.18	143.5	58.99	156

The “critical” temperature so found is 143.5 and the “critical” concentration 37.4 per cent. of amine ; whereas Guthrie’s results lead to 121° and 19 per cent. respectively. That this divergence is attributable to the glass seems not

unreasonable when we read Guthrie’s statement that “the 62.35-per-cent. solution caused the surface of the glass (soft

German) to peel off in visible scales when heated to 150°–160°."

An experiment with Müller's new "Resistance" glass showed that even the slightly greater solubility of this glass as compared with Jena glass caused a lowering of the temperature of over 2°.

It seems therefore probable that the present results are slightly vitiated by the solution of glass, but the extreme insolubility of Jena glass makes it likely that the inaccuracy due to its solution is but slight.

It is interesting to note that the form of the curve is more that of the curve for trimethylpyridine (Rothmund, *Zeit. Phys. Chem.* xxvi. p. 462, 1898) than of that for triethylamine (*ibid.* p. 461).

An attempt was made to determine the densities of saturated solutions of the amine and water at various temperatures by heating a solution containing 40·5 per cent. of amine in a graduated tube and observing the volumes of the layers at various temperatures; the weights of the layers being calculated from the percentage composition of the saturated solutions as read off from the solubility curve. The attempt proved abortive since the values obtained are so largely affected by small errors in the data used; it was, however, obvious that there is (1) practically no sudden change in total volume on separation, and (2) very little difference in the densities of the two saturated solutions which are in equilibrium at temperatures between 143°·5 and 153°.

Experimental Details.

Suitable quantities of water and diethylamine were weighed out from "weighing pipettes" into a small stoppered bottle. The mixture so made was transferred as soon as possible to a tube sealed at one end and drawn to a capillary in the middle; by changes of pressure the liquid was got into the lower part of the tube, which was then sealed off at the capillary.

A tube so prepared was heated in a paraffin bath so arranged that the filament of an incandescent electric lamp could be viewed through the tube. The appearance of a fog was very easily seen in this way, and consequently the temperature at which the mixture under observation separated into two layers could be ascertained with a fair degree of accuracy. The tube was supported by a simple mechanical device whereby the contents could be remixed between observations without removing the tube from the bath.

Oxford, May 1905.