

to be applicable in H₂O₂ decomposition. This feature will be discussed fully in a later communication.

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End-Point Cryoscopy

MANY methods in use for the cryoscopy of solutions suffer from the difficulty of equating an observed freezing point with a definite concentration. The procedure developed by us has avoided this difficulty by determining the temperature of a solution at which the last trace of frozen solvent melts. Thus the freezing point is measured at a concentration which has been established by direct weighing. This is particularly useful for organic solutions where accurate analysis (as for equilibrium cryoscopy) is often impractical.

Substance	Molecular weight	Slope of concentration v. melting point	Slope × molecular weight
Naphthalene	128	225	288
Benzoic acid	122	227	277
Nitrobenzene	123	225	277
Benzil	210	135	273
Methyl salicylate	152	187	284

The constancy of the last column indicates the precision attainable.

About 5 ml. of solution is shaken in a small glass bulb after cooling sufficiently to produce a small amount of frozen solvent. The temperature, measured by a thermistor inside the bulb, is plotted against time; this is most conveniently achieved with a recording amplifier. The temperature remains steady for a while, then increases regularly and slowly while any frozen solvent remains, but the slope changes sharply as the last trace of solid melts. The intersection of the two linear portions is a measure of the

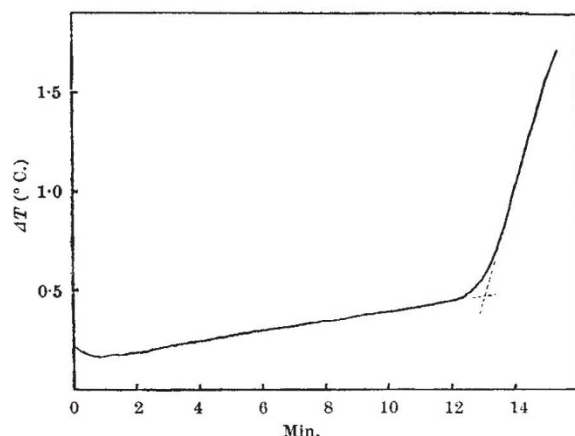


Fig. 1. Temperature-time curve for naphthalene in dioxane

required melting point. A typical curve is shown in Fig. 1. Each determination requires about 10 min.

The results given in Table 1 were obtained for dioxane solutions, 4-5 measurements being made for each substance.

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Adsorption of Long-Chain Aliphatic Amines on Glass Vessels

WORK involving the use of aliphatic amines in aqueous solutions is usually carried out in glass apparatus. We have found that in the case of *n*-dodecylamine a significant decrease in the concentration occurs at pH values greater than 7 due to adsorption of the amine at the glass-water interface. A larger decrease occurs if a plastic, such as polyethylene, is in contact with the solution.

Fig. 1 presents data obtained using 250 ml. of *n*-dodecylamine solution in a spherical glass vessel. Concentration loss is plotted against the final solution concentration after contact with the vessel for 15 min. Results obtained using a polythene beaker and an oxide-coated nickel crucible are given for comparison, the volume of amine solution used in the latter case being 50 ml.

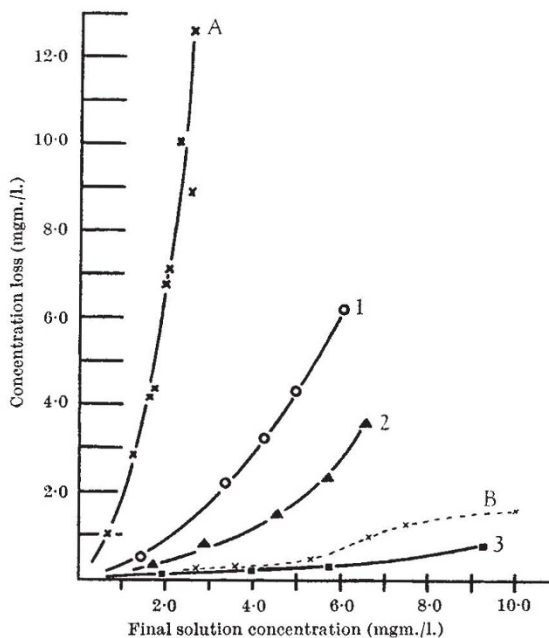


Fig. 1. Loss of *n*-dodecylamine from aqueous solutions of differing final concentrations in: (1) glass at pH 9.5; (2) glass at pH 8.5; (3) glass at pH 7.0; (A) polythene at pH 9.5; (B) oxide-coated nickel at pH 9.5. (Total initial amine concentration = concentration loss + final solution concentration)

If the surface of the glass was smooth, the area in contact with the solution was 160 cm.². Assuming close packing of the amine molecules and an area of 20.5 Å.² per molecule, the quantity of amine required to give a complete monolayer was 2.4 × 10⁻⁵ gm. The loss to the vessel was very much larger than this, so multi-layers must have been formed. This is in agreement with results obtained for the adsorption of