[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, GEORGIA SCHOOL OF TECHNOLOGY]

Some Physical Properties of Morpholine–Water Solutions[1]

BY HAROLD B. FRIEDMAN, ALFRED BARNARD, WILLIAM B. DOE AND CARL L. FOX

Commercial morpholine[2] to which about 3% of water was added was distilled and the fraction boiling below 126°, containing the chief impurities, namely, water and ethyl butanol, was discarded. The remainder was collected and refluxed over clean metallic sodium for four hours and again distilled, the distillate having the corrected boiling point of 127.9° at 739 mm. Repeated determinations of the refractive index at 20° gave 1.4540 as compared with the most recent value of 1.4545 found by Dermer and Dermer.[3]

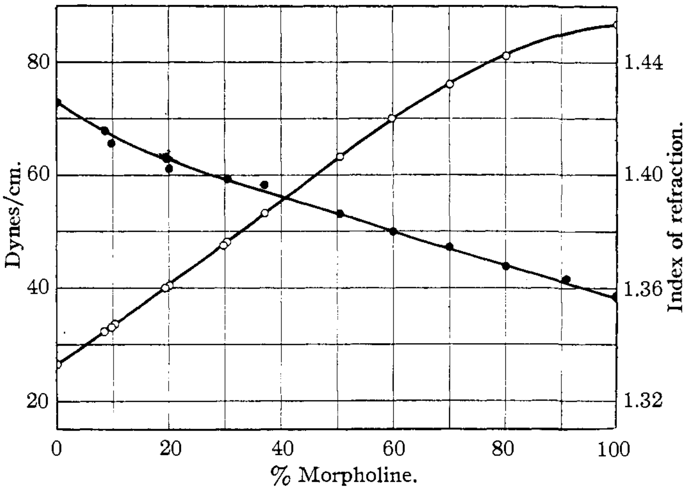


Fig. 1.—Morpholine–water at 20°: ○, index of refraction; ●, surface tension.

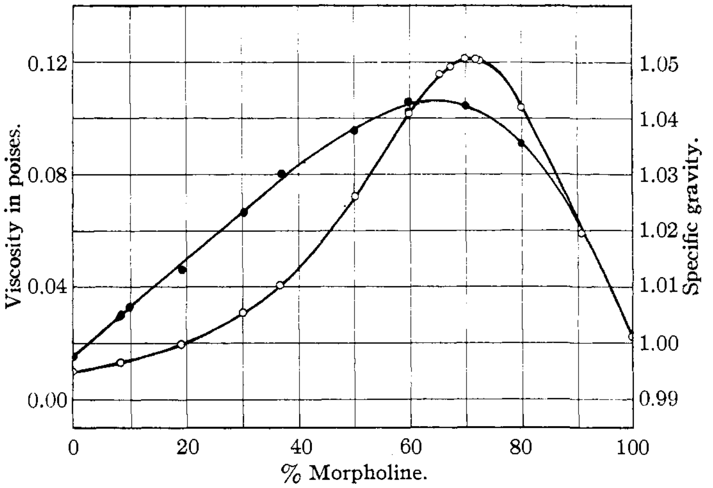
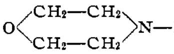


Fig. 2.—Morpholine–water at 20°: ○, viscosity; ●, specific gravity.

Physical properties of aqueous solutions of this material were determined as follows: the specific gravity using a specific gravity bottle with an enclosed thermometer; the indices of refraction with an Abbe refractometer; the surface tensions by the capillary rise method; and the viscosities by means of an Ostwald viscometer calibrated

---

[1] We are indebted for the material used in this research to the Carbide and Carbon Chemicals Corp., 30 E. 42d Street, New York, N. Y., in coöperation with the Mellon Institute. To both oft he organizations we express our thanks.

[2] The structure of morpholine,  $O\left\langle\begin{matrix}CH_2{-}CH_2\\CH_2{-}CH_2\end{matrix}\right\rangle N{-}H$, indicates that it is tetrahydro-1,4,2-oxazine.

[3] Dermer and Dermer, THIS JOURNAL, 59, 1148 (1937).

with water whose viscosity was taken as 0.01005 poise. The measurements were checked repeatedly and the data given in the table represent the average of several observations.

TABLE I

At 20°

Morpholine, %	Index of refraction	Viscosity in poises	Specific gravity	Surface tension, dynes/cm.
8.558	1.3447	0.01327	1.0057	67.80
9.740	1.3460	.....	1.0069	65.45
10.690	1.3472	.....	....	...
19.394	1.3598	.01963	1.0137	62.62
19.966	1.3608	.....	....	60.80
29.659	1.3750	.....	....	...
30.405	1.3763	.03082	1.0236	59.15
37.004	1.3863	.04064	1.0305	58.00
50.449	1.4066	.07227	1.0383	52.85
59.927	1.4201	.10223	1.0432	49.65
65.479	....	.11613	....	...
67.646	....	.11865	....	...
69.925	1.4322	.12168	1.0425	47.05
72.040	....	.12128	....	...
72.633	....	.12093	....	...
80.136	1.4428	.10433	1.0357	43.62
91	1.4502	.05954	....	41.60
100.000	1.4540[a]	.02282	1.0007[a]	38.72

[a] From "Beilstein," 4th ed., Vol. XXVII, p. 5.

We also confirmed Knorr's[4] observation of the evolution of heat when morpholine is mixed with water, finding the maximum effect to occur at about 70% morpholine, which corresponds approximately to the maximum viscosity. In the hope of finding whether a compound was formed in these solutions we attempted to measure their freezing points, but found this to be impossible except in the more dilute ones because of the slowness of adjustment of the freezing point equilibrium in solutions of such high viscosity. Seventy per cent. morpholine solution corresponds exactly to the composition of the possible dihydrate, *i. e.*, $C_4H_9ON \cdot 2H_2O$.

Summary

The specific gravities, indices of refraction, viscosities, and surface tensions of morpholine–water mixtures have been determined at 20°

[4] Knorr, *Ann.*, **301**, 3 (1898).

ATLANTA, GA. RECEIVED APRIL 20, 1940

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, THE CITY COLLEGE OF THE COLLEGE OF THE CITY OF NEW YORK]

The Solubility of Lead Iodate in Ammonium Acetate Solution

BY SYLVAN M. EDMONDS AND NATHAN BIRNBAUM

The fact that lead sulfate dissolves in ammonium acetate, of classic application in analytical chemistry, has been attributed to the formation of undissociated lead acetate[1] or of an acetoplumbite complex ion.[2] Sanved[3] in reviewing the more general investigations of the increased solubility of lead acetate and other lead salts in alkali acetate solution came to the conclusion that the ion formed is probably $PbC_2H_3O_2^+$, a result of the primary dissociation of lead acetate. Unfortunately, the experimental data have all been obtained for high and varying salt concentration, that is, under conditions where any application of the mass action law in terms of ionic concentrations would, at the very most, be qualitative in nature. In this investigation, the solubility of lead iodate has been determined at constant ionic strength, in the presence of varying acetate ion concentrations. From the data it should be possible to decide upon the reaction involved and to calculate the corresponding equilibrium constant.

Experimental

Materials.—Lead iodate was prepared and aged as described by La Mer and Goldman.[4] A sample dried at 110° was analyzed for iodate by solution in excess potassium iodide and hydrochloric acid followed by titration of the liberated iodine with sodium thiosulfate solution. The latter was standardized against potassium iodate. The percentage of lead iodate thus calculated was 99.91%. The water solubility was determined after shaking at 25 ± 0.01° until constant values were obtained. Equilibrium usually required from three days to a week. The dissolved iodate was determined upon filtered aliquots by addition of potassium iodide and hydrochloric acid and titration of the liberated iodine with thiosulfate. The water solubility thus found was 3.58×10^{-5} mole per liter (±0.3%). La Mer and Goldman reported the value 3.61×10^{-5} mole per liter. La Mer and Goldman stated that they could not reproduce the solubility of lead iodate to 0.1%.

---

(1) Noyes and Whitcomb, THIS JOURNAL, **27**, 747 (1905); Fox, *J. Chem. Soc.*, **95**, 878 (1909).

(2) White, *Am. Chem. J.*, **31**, 4 (1904); **35**, 218 (1906); Blomberg, *Chem. Weekblad*, **11**, 1030 (1914); *Z. Elektrochem.*, **21**, 438 (1915); Conrad, Diss., Göttingen, 1903; Labendzinski, Diss., Breslau, 1904.

(3) Sanved, *J. Chem. Soc.*, 2967 (1927).

(4) THIS JOURNAL, **52**, 2791 (1930).