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An Improved Method of Determining Solubility

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AN IMPROVED METHOD OF DETERMINING
SOLUBILITY.

W. S. HENDRIXSON.

In the continuation of work on acid sodium and acid potassium phthalates as standards in acidimetry and alkalimetry¹, it seemed desirable to study among other properties, the solubility of these salts.

It seems to be generally recognized that none of the methods for determining solubility is wholly satisfactory. Among the major difficulties are these: The somewhat complicated machinery necessary to agitate the liquid in the thermostat, and the solvent, and the difficulty of securing a specimen of the clear solution for analysis, without change of temperature. Since this laboratory, like most others, is supplied with a practically un-

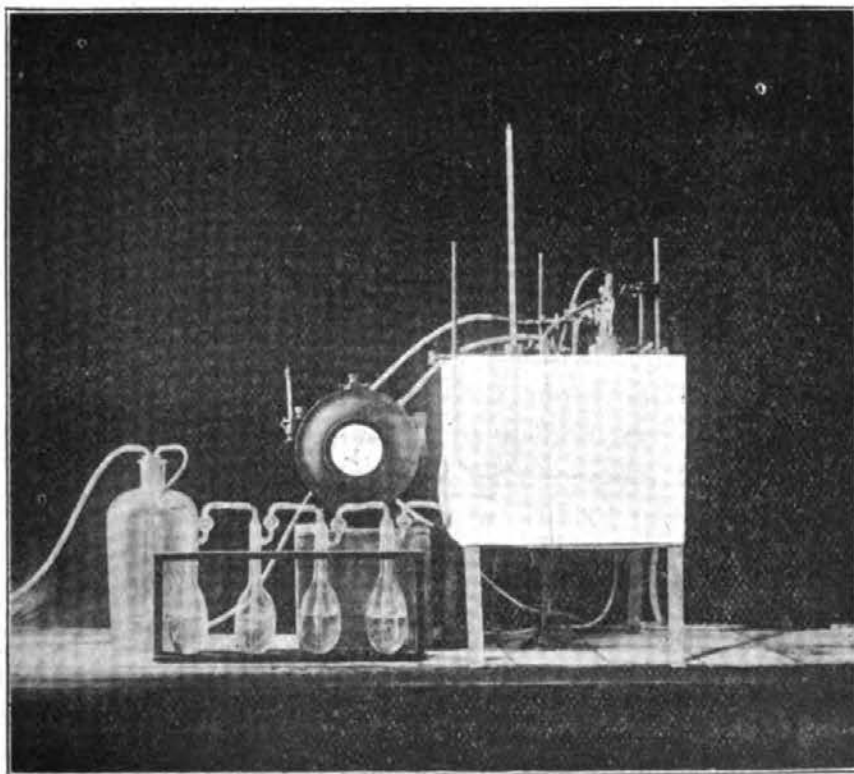


FIG. 1.

limited amount of compressed air, the attempt has been made to use it for the agitation of solvent and bath. To avoid the second difficulty a special form of pipet has been used, which could be wholly submerged during the time of agitation so as to be at the

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¹Proc. Iowa Acad. Sci., Vol. XXII, p. 217.

same temperature as the solution. The results have been so satisfactory that a statement describing the method apart from the main subject of investigation seems to be justified.

The arrangement of the whole apparatus is shown in figure 1. About ten gallons of water are contained in a tank of sheet iron covered with layers of asbestos, felt and canvas. The regulator is the ordinary form of Ostwald, designed for heating with gas. The vessel for the solution is a large test-tube and it contains a pipet holding about 20cc. as shown in figure 2, A. A large rubber stopper was cut through to one of the holes, the shank was inserted into the hole and the cut was closed with rubber cement. A second pipet for higher temperatures where the solu-

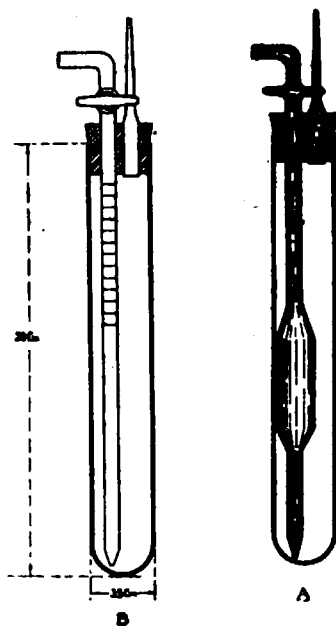


FIG. 2.

tion became more concentrated, is shown in figure 2, B. The meter shown in figure 1 is a test gas meter reading to .001 cubic foot.

At first the thought was to stir both the bath and the solvent with the same stream of air, but this introduced undesirable complications and two streams were used. Ordinary air from the reservoir was passed into the bath at the bottom and near the middle at the rate of about three cubic feet per hour, which was found ample as shown by two standard thermometers whose bulbs were placed in different parts of the bath for comparison.

The air for the solution was first passed into a large bottle having a layer of strong potash solution, then through four bottles of the same sort of solution of concentration 1 to 2. It then passed through the meter and through four gas washing bottles with water which were wholly immersed in the bath. The purpose of these last bottles was to saturate the air with water vapor at the temperature of the bath. The use of air for stirring in such cases is not new², but so far as could be ascertained there has been no attempt to compensate for the unavoidable loss of solvent by evaporation. The air then passed through the pipet and into the solution. The bottles, tube for the solution, the pipet and all connections, save only the tip of the capillary exit tube from the solution vessel, were wholly immersed.

Before undertaking actual determinations of solubility the degree of compensation for evaporation was tested by many experiments extending over periods from one to five hours, at temperatures from 25° to 65°, the rate of the air current being about 1.5 cubic foot per hour. This rate is sufficient for the agitation of the solution. The tube containing the water and pipet was weighed at the beginning and end of each experiment. In some experiments there was a small loss, in others a slight gain in weight, the difference never amounting to more than .035 gram per cubic foot of air. Since it was shown that the gain or loss was closely proportional to the volume of air, and, therefore, to the time, it is evident that it could not exert any appreciable influence on the results in such work. A few blank tests have been made at 80°. At this temperature there was an apparent loss of about 0.2 gram of solvent per cubic foot of air. This loss is probably due to secondary causes that it may not be possible to eliminate at such a high aqueous tension. Even if confirmed by later experiment it seems hardly probable that it could cause a degree of supersaturation that could materially affect the results. However, there are other considerations that make it seem probable that this method is best suited for moderate temperatures, and that at or near the boiling point of the solvent some one of the special methods for such temperatures may more conveniently be used.

In carrying out an actual determination the substance under investigation was dissolved in about 60cc. of water in sufficient

²Pawlewski, *Berichte V. Deutsch Chem. Gessel.*, p. 1040.

amount to give an abundant crystallization on cooling to the desired temperature of the experiment. The calibrated pipet was so inserted as to leave its tip above the surface of the solution. The whole was then placed in the bath and when solution and bath were near the same temperature the air was started through the pipet which was then lowered into the solution. At the end of a two-hour period the pipet was raised slightly, its stop-cock was turned and the hose conducting the air was removed. In a few moments the solution became perfectly clear, and the pipet was filled with it to the stop-cock by suction. The pipet was removed, washed, dried and weighed. The solution was run into the titration flask, and the pipet was washed into the same with warm water. The whole was then titrated. From the weights and known volume of the pipet, the concentration per gram and per cc. and also the density of the solution could be readily calculated.

No filtering device for the pipet was necessary, nor any drip cap, since the solution could not run out till the stop-cock was opened. With other substances which settle slowly or not at all it would be necessary to use a filtering device of gauze or other material as in other methods. In such case it would probably be better to pass the air into the solution through an extra tube and not through the pipet.

After pipetting out at the end of the first experiment the test-tube and remaining solution were left in the bath. After emptying the pipet and drying it, it was reinserted and a second experiment was carried out extending over an additional period of two hours, or four hours in all. The duplicate experiments give almost perfectly concordant results. These and other data on the acid phthalates are reserved for another communication.

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