

The melting points of the semihydrate and anhydrous crystals were found to be  $29.32^{\circ}$  and  $42.35^{\circ}$ , respectively; and their rates of crystallization, respectively, 2.6 cm. and 33.3 cm. per minute at  $20^{\circ}$ .

WASHINGTON, D. C.

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### NOTES

**Preparation of Nitric Oxide from Sodium Nitrite.**—The usual method of preparing nitric oxide by the action of nitric acid on copper is rather tedious in operation and gives a product contaminated with nitrogen dioxide or nitrous oxide unless the concentration of the acid and the conditions are carefully controlled. The gas may be generated rapidly and in nearly pure condition by dropping concd. sulfuric acid into a flask or distilling bulb containing sodium nitrite covered with two or three times its weight of water. The nitrous acid liberated decomposes almost quantitatively according to the equation,  $3\text{HNO}_2 = \text{HNO}_3 + 2\text{NO} + \text{H}_2\text{O}$ .

A small amount of nitrogen dioxide which the gas contains may be removed by passing it through a wash bottle containing concd. sulfuric acid or by collecting it over water.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY

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**The Solubility of Urea in Water.**—While working with concentrated aqueous solutions of urea at various temperatures, it was found that the solubility of urea as recorded in Seidell's "Solubilities of Inorganic and Organic Compounds,"<sup>1</sup> is considerably too low. Upon examining the original reference,<sup>2</sup> it was found that the results were expressed as "per cent. gram molecules, that is, to the number of gram molecules of solute in 100 gram molecules of solution." In recalculating these results for Seidell's book, this expression had been misconstrued, the data being calculated as moles of solute in 100 moles of solvent, instead of solution.<sup>3</sup> We have recalculated Speyers' data and have also determined the solubility of urea in water at various temperatures.

Our method consisted in heating about 300–400 cc. of urea solution in a water-bath in the presence of solid urea<sup>4</sup> to a temperature a few degrees above that at which the solubility was to be determined. The solution

<sup>1</sup> W. Seidell, "Solubilities of Inorganic and Organic Compounds," D. Van Nostrand Co., New York, N. Y., 2nd ed., 1919, p. 737.

<sup>2</sup> Speyers, *Am. J. Sci.*, [IV] 14, 293 (1902).

<sup>3</sup> Speyers determined the solubilities of a number of carbon compounds in various solvents; we find that the same error has been made in recalculating the solubility data for Seidell's book.

<sup>4</sup> Synthetic urea purified by two recrystallizations from water was used.

was gradually cooled to the desired temperature and, after being maintained at this point for about ten minutes, a sample (25-35 g.) was taken by immersing a hot weighing bottle suspended from a copper wire. Samples were taken at approximately 10° intervals, between 0° and 70°,

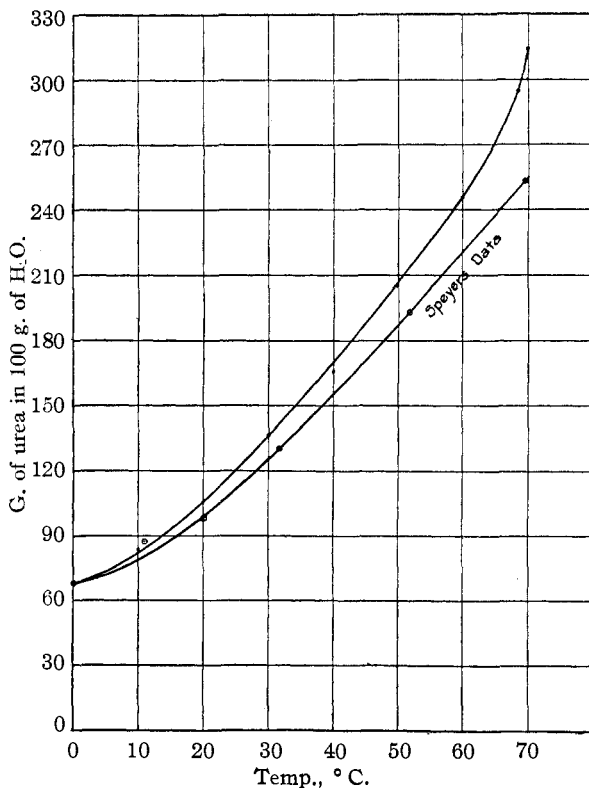


Fig. 1.

weighed, and the urea was determined by the urease method.<sup>5</sup> The solubilities at 0° and 10° agree very well with Speyers' values, but increasing differences occur with rise in temperature.

TABLE I  
SOLUBILITY OF UREA IN WATER

		Speyers' data									
1.	Temp., °C.	0.0	11.0	19.8	31.7	51.4	69.5				
2.	G. of urea in 100 g. of H <sub>2</sub> O	67.4	87.5	97.5	131.0	193.0	253.0				
		Data obtained in this study									
1.		0.0	10.0	20.0	30.0	39.7	50.0	50.6	60.0	68.5	70.0
2.		67.0	84.0	104.7	136.0	165.4	205.0	206.4	246.0	295.0	314.6

<sup>5</sup> Fox and Geldard, *Ind. Eng. Chem.*, **15**, 743 (1923).

In order to check the results obtained, determinations were repeated at a few of the higher temperatures, using Speyers' apparatus with slight modifications. The results obtained with this apparatus agreed closely with the first determinations. A comparison of Speyers' data and those obtained in this study is shown in Fig. 1 and Table I.

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**Electrode Vessel for Liquids Heavier and Lighter than the Liquid Junction Potential Eliminator.**—In the electrode vessel to be described the hydrogen-ion concentration of solutions having a density equal to or greater or less than that of the bridge solution can be measured. Sharp and reproducible liquid junctions or flowing junctions can also be conveniently and rapidly made between the electrode solution and the bridge solution by including the feature embodied in the apparatus of F. A. Elliott<sup>1</sup> and Elliott and Acree.<sup>2</sup>

The ground-glass joints and the intricate stopcocks of the Elliott-Acree apparatus, which can be made and repaired only by well equipped and highly skilled glass blowers, are not used, thus making the new apparatus less expensive and more durable. Furthermore, solutions containing suspended matter, such as quinhydrone in quinhydrone electrode work or soil suspensions which would settle down into the bridge liquid in the Elliott or Elliott-Acree apparatus, may be tested without developing this difficulty.

### Electrode Vessel

The tube *d* for holding the electrode solution is connected by a loop *a*, *b*, *c* to the bridge tube *e*, which contains the bridge solution, in such a way that the liquid junction can be made at either of two points in the loop, depending upon the densities of the two solutions making contact.

At *a* and *c* on the loop are connected horizontal branch tubes, with stopcocks for making liquid junctions at these points. At *b* is another branch tube, with a stopcock, through which air may be removed from the loop when the latter is being filled. For liquids denser than the bridge solution the junction contact is made at *a* by filling the empty loop *a*, *b*, *c* to the stopcock *h* with bridge solution and allowing the denser solution from *d* to rise from *h* to *a* by drawing off solution through Stopcock *a*. For liquids

<sup>1</sup> "Elliott Ion-O-Meter," The Will Corporation, Rochester, N. Y., Bulletin 100, 1921.

<sup>2</sup> "Hydrogen-ion Determinations," *Pyroelectric Bi-monthly Bulletin*, 14, September, 1920, pp. 3-8 (Pyroelectric Instrument Company, Trenton, N. J.). Slagle and Acree, *Abstracts of Bacteriology*, 5, 5 (1921).

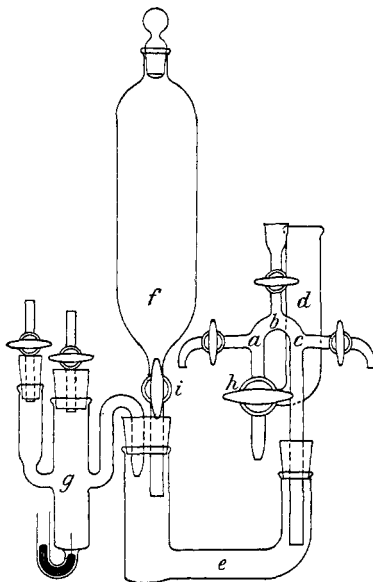
less dense than the bridge solution the junction is made at *c* by filling the empty loop *a, b, c* with the solution from *d* and drawing off solution through the stopcock *c* until sharp contact is made. A three-way, T-bore stopcock *h* is placed between the loop and the tube *d*, which is joined as closely as possible to the stopcock *h*, to minimize the distance between the liquid junction at *a* and the position for the platinum electrode, particularly when working with solutions of low conductivity, such as concentrated sugar solutions. The tube *d* connects in the horizontal position to Stopcock *h*, so that any suspended matter which may settle from the electrode solution does not interfere with the stopcock and is easily washed out through the bottom arm of the stopcock *h*.

To empty the loop for cleaning and changing solutions, the stopcock *h* is opened to bring all three branches together. Stopcocks *a, b* and *c* are opened and the solution is drained.

The electrode vessel is best made of Pyrex glass.

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**A Sensitive Glass Manometer for Gases which Attack Mercury.**<sup>1</sup>—Baume and Robert<sup>2</sup> constructed a manometer for use with gases that attack mercury, consisting essentially of a flat glass diaphragm exposed to the corrosive gas, whose motion was communicated to a superincumbent column of mercury. This liquid is not suitable for the thin diaphragms required for sensitive manometers, and we have therefore substituted for it a light paraffin oil.

The construction of such a manometer, using this liquid, is evident from Fig. 1. *M* is a thin, disk-shaped, glass membrane 5 to 6 cm. in diam-

<sup>1</sup> Translated from the German and abbreviated by A. L. Dixon.

<sup>2</sup> Baume and Robert, *Compt. rend.*, **168**, 1199 (1919). See also, for similar manometers, Ladenburg, *Verh. Deutsch. physik. Ges.*, **3**, 20 (1906). Bodenstein, *Z. physik. Chem.*, **69**, 26 (1909). Scheffer and Treub, *ibid.*, **81**, 308 (1913). Johnson, *ibid.*, **61**, 457 (1908). Particularly, Daniels and Johnston, *THIS JOURNAL*, **43**, 53 (1921).

eter, which serves as the diaphragm. Its chief requirement is that it shall not bulge too suddenly when subjected to pressure. The membrane is

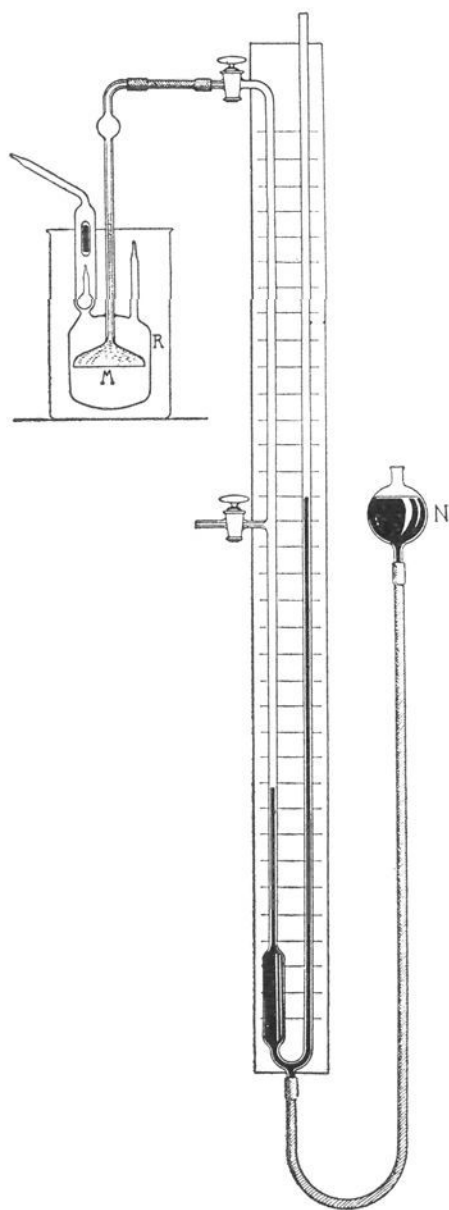


Fig. 1.

When this connection is made and the tube *e* sealed off, the apparatus is ready for the determination. At this time the barometer is read.

By means of a magnet the piece of iron is made to break the bulb *K*. Immediately, the pressure begins to rise and is kept balanced by raising the leveling bulb *N*. The pressure is accurately determined by the difference in level in the manometer *Q*. When *K* contains a very volatile liquid, the introduction of glass wool at *x* slows down the rate of the increase in pressure.

sealed to the end of the capillary *k* by the considerably thicker glass *a*. The space *p* inside of the membrane and the capillary are filled with the paraffin oil, which may be colored by an oil-soluble dye. The membrane is surrounded by a thick-walled jacket *R*. A small, thin-walled bulb *K* in which is sealed the sample, and a small piece of iron likewise sealed in glass, are introduced into *b*. The tube at *e* is then constricted and a current of dry air is conducted through the apparatus, passing out through the tube *f*. When the manometer is dry the tube *f* is sealed off and the apparatus is placed in a thermostat at the temperature at which the vapor-pressure determination is to be made. Only the open end of *e* projects from the thermostat. When the entire vessel has attained a constant temperature the position of the meniscus in *k* is marked. At *h* the apparatus is connected by heavy rubber tubing to a mercury manometer *Q*, as shown.

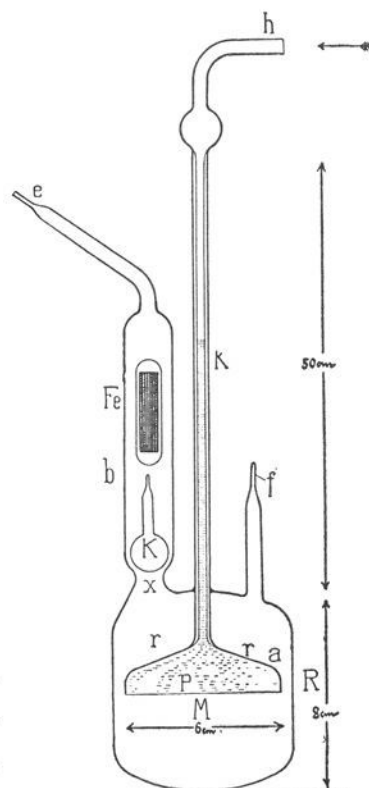


Fig. 2.—Compensation pressure.

rate of the increase in pressure.

