Notes

Improved Method of Sealing the Capillary Tubes in the Rast Modification of the Barger Method of Molecular Weight Determination

By Joseph R. Spies

In the determination of molecular weights by the Rast modification of the Barger method it has been found that by operating in the manner described below the comparatively large air space at one end of the capillary tube and also the necessity of twice drawing out one end of this capillary can be avoided. The method has the further advantage that the liquid is fixed in both ends of the capillary without heating the capillary close to the enclosed solutions. This minimizes the danger of volatilizing some solvent or possibly decomposing some of the solute.

Assume that the standard solution and the test solution have been introduced into the capillary with the prescribed 3–4 mm. air space separating them, and that the tube has been melted shut on the right and drawn out 2 cm. to hairbreadth on the left as Rast describes.[1] Now instead of breaking off the left end at the end of the hair, it is allowed to remain, leaving a funnel-shaped reservoir. The tube then appears as illustrated in Fig. 1. The right end is now broken off and the tube tilted so that the liquid flows back through the hair to a position "a." The tube is then drawn out on the right to hairbreadth for 2 cm., broken at the end of this hair and tilted so that the liquid runs into and fills it, whereupon it is sealed by carefully touching it in a micro flame. Some of the liquid should remain in the funnel on the left so that the hair on the left is still filled. This funnel is then broken off and the end of the hair passed rapidly through a micro flame to expel a small portion of the solution before sealing as on the right.

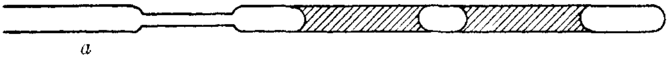

Fig. 1.

A 5 × 17 cm. glass plate with a line etched through the center parallel to the short side supplants the 1–2 × 17 cm. plate used by Rast. This allows eight to ten tubes to be placed on one plate. The etched line takes the place of the hair covered with Canada balsam. When it stands in water, balsam gradually becomes opaque and must be replaced daily.

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[1] K. Rast, *Ber.*, **54**, 1979–1987 (1921).

The Dielectric Constant of Solid Hydrogen Sulfide

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Preliminary measurements have been made on the variation of the dielectric constant of solid hydrogen sulfide with temperature. Heat capacity measurements made in this Laboratory by Professor W. F. Giauque and Dr. R. W. Blue[1] have shown the existence of three crystalline modifications of hydrogen sulfide. It was therefore of interest to learn whether any rotational motion of the electric moments of the molecules in the crystal was taken up at the transitions.

A heterodyne beat method similar to that described by Zahn[2] with the oscillators operating on a wave length of 649 meters was used for the measurement of the dielectric constant. The test condenser (cap. 150 $\mu\mu$f) consisted of three concentric cylinders of gold rigidly separated by four glass rods cutting diametrically through them. The condenser was sealed in a glass cylinder and the temperature controlled by an apparatus similar to that used by Giauque and Wiebe,[3] the gold condenser replacing their calorimeter. A copper–constantan thermocouple was used for the temperature measurement.

The value of the dielectric constant below the lower transition at 103.5° K. is about 2.9 and above the lower transition for the second modification about 9, a discontinuous change taking place at the temperature of the transition. At the upper transition at about 126.3°K., there is apparently no marked change of the dielectric constant taking place. The value of the constant for the liquid at the melting point 187.6°K. is about 9.4. The accuracy of the measurements in the solid state is probably no better than 10% due to the effect of the large cracks encountered in warming the hydrogen sulfide.

From the results given above it appears that there is little or no rotation of the electric moment of the hydrogen sulfide molecule below the lower transition. At this transition a change in crystalline form must take place permitting rotation of the electric moments and from the large change in the dielectric constant it can be concluded that the molecules go from a state of little or no rotation of the electric moment to one of almost complete rotational freedom of the electric moment. This is further verified by the facts that at the upper transition there is no great difference between the dielectric constants of the two crystalline modifications of hydrogen sulfide concerned in the transition and that the value of the dielectric constant for the liquid at the melting point is of the same magnitude as the values for the crystalline modifications above the lower transition.

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(1) Not yet published.

(2) Zahn, *Phys. Rev.*, **24**, 400 (1924).

(3) Giauque and Wiebe, THIS JOURNAL, **50**, 161 (1928).