

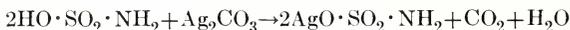
The Preparation and Properties of Ethyl Amidosulfonate

W. W. BINKLEY with ED. F. DEGERING, Purdue University

Ephraim and Lasocky (1) reported the preparation of methyl amidosulfonate from methyl iodide and silver amidosulfonate in 1911. No further investigation of this compound or its homologues has since been reported. The present study was undertaken with the hope of obtaining ethyl amidosulfonate and then studying its properties.

Preparation of Amidosulfonic Acid.—Amidosulfonic acid may be obtained by saturating an aqueous solution of hydroxylamine hydrochloride, or better hydroxylamine sulfate, with sulfur dioxide. A more economic course, however, was pursued. Sodium amidosulfonate was prepared by the reduction of sodium nitrite with sulfur dioxide in the presence of sodium carbonate. The acid was then liberated from the sodium salt with sulfuric acid (2). Well-formed crystals of this colorless acid were easily obtained. The acid has a sharp sour taste. The yields were 50 to 60% (Table I a).

Preparation of Silver Amidosulfonate.—Although Divers and Haga (2) did not disclose their method for preparing silver amidosulfonate, they recommended obtaining it from amidosulfonic acid. It was obtained readily from amidosulfonic acid and silver carbonate, according to the following equation:



Excellent yields of colorless, striated prisms of silver amidosulfonate were obtained (Table I, b).

Preparation of Ethyl Amidosulfonate.—Finely divided silver amidosulfonate (10 g.) was placed in a large test tube equipped with a mechanical stirrer. A solution of 4 ml. of ethyl iodide dissolved in 25 ml. of ethyl ether was poured into the test tube, and the stirring begun. The reaction started at once as evidenced by the formation of yellow silver iodide. The agitation was continued for 24 hours and the ether removed by decantation. This residue was extracted with two 25 ml. portions of hot absolute ethyl alcohol. The alcohol was removed by distillation, and a light yellow solid with a sweet odor remained. The last traces of ethyl alcohol, color, and odor were removed from the residue by washing with a few milliliters of ethyl ether on a porous plate. The presence of carbon, hydrogen, sulfur, and nitrogen was established in the usual manner and sulfur was determined quantitatively (Table I, c). Ethyl amidosulfonate is a crystalline solid which decomposes without evidencing a definite melting point. It begins to sweat between 140° and 150°C. and to decompose with foaming and charring at 209°C. The yields were 2 to 3 g.

TABLE I.—Analytical Results

	Theoretical	Obtained
a. Molecular weight of amidosulfonic acid by neutral equivalent	97 g.	96.7 g. \pm 0.2
b. Silver in silver amidosulfonate.	52.7%	52.9% \pm 0.1
c. Sulfur in ethyl amidosulfonate.	25.6%	25.1% \pm 0.4

Summary.—Ethyl amidosulfonate may be prepared as a crystalline solid from ethyl iodide and silver amidosulfonate.

Bibliography

1. F. Ephraim and E. Lasocky, 1911. *Über Nitro-sulfamid und Sulfohydrazid.* Ber. 44, 395.
2. E. Divers and T. Haga, 1896. The preparation and properties of amidosulfonic acid and its salts. *Jour. Chem. Soc.* 69, 1634.