Mol. wt. 134.3, 136.1, mean, 135.2. Last fraction. Subs., 0.0383, 0.0506; vol., 302.0; pressure, mm., 17.7, 22.9; *t*, 28.1, 28.5; mol. wt. 134.6, 137.6, mean, 136.2. Calcd. for $(C_2H_5)_3SiF$: mol. wt., 134.2.

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A Hydrate of 1,3,5-Xylenoxyacetic Acid

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In a study of phenolic fractions obtained from coal tar, we have found it desirable to prepare derivatives of certain pure phenols for purposes of comparison. One of the derivatives which we prepared was the oxyacetic acid formed by interaction of symmetrical xylenol and chloroacetic acid in the presence of sodium hydroxide. We were especially interested in this compound because Schneider and Shohan,[1] working with a symmetrical xylenol prepared by synthesis, found that their specimen melted at 111° whereas Schütz and collaborators,[2] Steinkopf and Höpner,[3] and Brückner[4] state that its melting point is 85 or 86°. We find this discrepancy to be explained by the fact that the material reported melting at 86° is a monohydrate, the anhydrous substance melting at 111°.

The xylenol which we used was a coal tar product melting at 63.2°, obtained from the U. S. Bureau of Mines. The hydrate of 1,3,5-xylenoxyacetic acid, easily prepared by crystallizing the product from water, melts actually at 81.0°, but is unstable, losing its water of crystallization on standing for a few days over phosphorus pentoxide in a vacuum desiccator at room temperature. The product then melts at 111.2°. A mixture of equal parts of hydrate and anhydrous material melted indistinctly between 97 and 104°. The hydrate loses at least a part of its water when it is dissolved in warm benzene and is reprecipitated by addition of petroleum ether. Schneider and Shohan reported 86–92° as the melting range of their product before recrystallization; they then recrystallized from benzene and found the melting point to be 111°. It is probable that their first product was largely the hydrate and that it lost water when warmed with benzene.

We report the melting point of the hydrate as 81.0° on the basis of the following evidence. The crude reaction product, isolated from aqueous alkaline medium by precipitation with hydrochloric acid, melted at 81–83°. We recrystallized it from water, and allowed it to stand in a desiccator

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(1) Schneider and Shohan, *Bur. Mines, Repts. Investigations* No. **2969**, 1929, 10 pp.

(2) Schütz and co-workers, *Ber.*, **56**, 1971 (1923).

(3) Steinkopf and Höpner, *J. prakt. Chem.*, [2] **113**, 141 (1926).

(4) Brückner, *Erdöl und Teer*, **4**, 562, 580, 598 (1928); *C. A.*, **23**, 1246 (1929); *Z. Angew. Chem.*, **41**, 1043, 1062 (1928).

in an atmosphere saturated with water vapor at room temperature, until weight equilibrium was reached. It then melted sharply at 81.0° corr. when heated at the usual rate of 1–2° per minute. The value 85 or 86° probably corresponds to a partially dehydrated product; for, if the temperature of the bath be raised very slowly, about 1° in three minutes, the hydrated substance will begin to melt at 81°, but at the same time will gradually become dehydrated; with cautious heating it is possible to reach 111°, the melting point of the anhydrous material, before fusion is complete; but it is also possible with irregular heating to observe melting at almost any point intermediate between 81 and 111°.

The solubility of the anhydrous substance in boiling water is about 1 g. in 100 cc.; in benzene, somewhat more than 1 g. in 10 cc.

Another property of 1,3,5-xylenoxyacetic acid not mentioned in the literature is its volatility in a current of steam. We found that 100 g. of steam in the course of one hour carried over 0.0066 g. weighed as anhydride.

Analysis.—A determination of water of crystallization was made in a micro vacuum drying tube over phosphorus pentoxide at about 70°: weight of sample, 4.395 mg.: H_2O found, 0.395 mg. or 8.99%; calcd. for $C_{10}H_{12}O_3.H_2O$, 9.09%. Micro combustion analysis of the anhydrous material remaining after the water determination gave the following results: weight of sample, 4.000 mg.: CO_2, 9.81 mg.; H_2O, 2.44 mg. C found, 66.88%; calcd., 66.63%; H found, 6.82%; calcd., 6.72%. This microanalysis was made by Mr. F. C. Silbert of this Laboratory.

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Rotenone in a Species of Spatholobus

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Some time ago the Insecticide Division received a sample of roots[1] of a fish-poisoning plant from Burma, which the Economic Botanist of that country considered to be *Spatholobus roxburghii* Benth., a large, woody climber. In the course of tests of numerous plant extracts, Drake and Spies[2] found that an acetone extract of this root material possessed a high toxicity to goldfish. Recent tests by F. L. Campbell of the Bureau of Entomology also indicate that the extract is highly toxic to mosquito larvae.

While testing a number of plant extracts of high fish and insect toxicity for rotenone by the blue color test recently outlined,[3] the author found that the acetone extract of *S. roxburghii* gave a deep blue color, estimated to be

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(1) The material received consisted mainly of the bark of the root.

(2) Drake and Spies, *J. Econ. Entomol.*, **25**, 129 (1932).

(3) Jones and Smith, *Ind. Eng. Chem., Anal. Ed.*, **5**, 75 (1933).