Each tube contained 7 g. of pure sulfur mixed separately with 0.3 g. and 1.0 g. of CoF$_3$; 0.3 g. and 1.0 g. of HgF; 0.5 g. and 1.0 g. of AgF. At the end of the experiments, all tubes were under fairly high pressure indicating formation of the gases had taken place. Only after prolonged heating (about forty-eight hours at 180–440°) was a slight reduction of the viscosity noted.

These experiments lead to the opinion that the observed failure of the sulfur fluorides to greatly affect the viscosity of sulfur is just one more instance of the abnormal behavior of fluorine and its compounds when viewed in comparison with the other halogens.

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The Melting Point of Mustard Gas

BY W. A. FELSING, C. A. HUNTING AND S. D. FELL

Recently du Vigneaud and Stevens[1] reported a study of the preparation and purification of mustard gas (*bis*-(β-chloroethyl) sulfide) and its action on yeast. They purified the mustard by three recrystallizations from absolute ethanol and once from petroleum ether; the product had a melting point of 14.5°. The authors also cite references to other reliable melting point determinations found in relatively recent literature (*i. e.*, 14.4° and 14.5°).

In Chemical Laboratory Report No. 369, Edgewood Arsenal, Edgewood, Md., dated November 29, 1918, the authors of this note reported the purification and the melting point of mustard gas. Crude Levinstein mustard gas, melting at 8–9°, was distilled at 10 mm. pressure, yielding a distillate melting at 13.6°. This material was thrice distilled at pressures below 10 mm. and subjected to partial freezing. It was collected on a Buchner filter and the adhering liquid was removed rapidly by suction. The collected crystals were melted and again the resulting liquid was partially re-frozen and the crystals collected as before. In all, the crystals were partially frozen, drained and remelted seven times. The final product weighed about 750 g. (original volume of distilled mustard gas was about 1 gallon).

The melting point of the purified mustard gas was determined by the usual procedure of taking temperature readings every thirty seconds until every trace of the crystals had disappeared. The melting point was determined graphically from a time–temperature plot. The melting point apparatus and the thermometer were kindly loaned by the Physical Chemistry Division of Johns Hopkins University. The short-range thermometer, whose ice-point was carefully checked and which was calibrated by the Physikalische Technische Reichsanstalt (P.R.T. No. 26260) was calibrated in 0.1° and temperatures could be estimated to 0.02° by the aid of a magnifier. Two series of determinations yielded a melting point of 14.45° and one of 14.44°.

Both of these values agree markedly well with the values given and cited by du Vigneaud and Stevens.

This note was suggested as a historical record by Chemical Corps Technical Command, Army Chemical Center, Md.

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[1] du Vigneaud and Stevens, THIS JOURNAL, **69**, 1808 (1947).

The Preparation of *p*-Dimethylaminobenzoic Anhydride

BY WILLIAM S. FONES

In connection with other work under way in this Laboratory it was necessary to prepare *p*-dimethylaminobenzoic anhydride. By heating *p*-dimethylaminobenzoic acid in acetic anhydride V. Meyer and Askenasy[1] isolated a substance (m. p. 109°) that analyzed correctly for an addition complex consisting of one molecule of *p*-dimethylaminobenzoic anhydride and one of acetic anhydride. This work was repeated by Van Der Haar,[2] who reported the complex (m. p. 109°) lost acetic anhydride when heated above its melting point to yield a substance sintering at less than 200° and melting at 218°. He assumed the latter material to be the free anhydride.

This assumption is shown to be in error by the present work. The anhydride was prepared by the action of phosphorus pentoxide on *p*-dimethylaminobenzoic acid in boiling xylene (method A) or by adding phosphorus oxychloride to a solution of *p*-dimethylaminobenzoic acid and triethylamine in chloroform (method B). The product from either reaction upon recrystallization from benzene had a m. p. of 157–159° (cor.); a mixed melting point determination showed no depression. By method A the anhydride was obtained in a yield of 30% based on acid used with a 36% recovery of unreacted acid whereas a 50% yield was obtained by method B but no starting material was recovered.

The pure compound gave the expected analysis and was further characterized by conversion to the known methyl *p*-dimethylaminobenzoate,[3] and *p*-dimethylaminobenzamide[4] through the action of methanol and ammonia, respectively.

Experimental

***p*-Dimethylaminobenzoic Anhydride (Method A).**—To a stirred suspension of 9.1 g. of *p*-dimethylaminobenzoic acid in 450 ml. of refluxing *m*-xylene there was added 8 g. of phosphoric anhydride. Stirring and refluxing were continued for seven hours with an additional 8-g. portion of phosphoric anhydride being added at the end of the first,

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[1] V. Meyer and P. Askenasy, *Ber.*, **26**, 1365 (1893).

[2] A. W. Van Der Haar, *Rec. trav. chim.*, **47**, 324 (1928).

[3] John Johnston, *Proc. Roy. Soc.* (*London*), **A78**, 82 (1906).

[4] German Patent 77,329 (1892); *Frdl.*, **4**, 173 (1899).