

Booth and Pierce.⁹ While the earlier workers simply mixed alcoholic solutions of beryllium nitrate and benzoylacetone, the present method involves neutralization of the benzoylacetone in the presence of beryllium ion. The reaction proceeds with the dilution of the alcoholic solution by a considerable volume of water, thus facilitating crystallization of the water-insoluble product.

Five grams of benzoylacetone (0.032 mole) in 20 ml. of 95% ethanol is added to a solution of 3.3 g. of beryllium(II) nitrate 4-hydrate (0.016 mole) in 35 ml. of 95% ethanol. Thirty milliliters of 1 *N* aqueous sodium hydroxide (0.030 mole) is added dropwise to the solution while it is stirred rapidly. As the sodium hydroxide is added, crystals begin to form. After addition is complete, the solution is allowed to stand 30 minutes and then filtered. The product is washed with small amounts of alcohol and water and then dried at 110°. After recrystallization from benzene and again drying at 110°, the melting point is 210°. Booth and Pierce reported 211°. *Anal.* Calcd. for $[\text{Be}(\text{C}_{10}\text{H}_9\text{O}_2)_2]$: C, 72.45; H, 5.47. Found: C, 72.67; H, 5.58.

Partial Resolution with *d*-Quartz.—Partial resolution of bis-(benzoylacetono)-beryllium was attained by shaking 25 ml. of a benzene solution of the complex with powdered quartz for 15 minutes. The quartz was removed by filtration and the rotation read at the sodium D line using a Schmidt and Haensch Polarimeter (No. 9143). The readings given represent from six to ten settings of the instrument and are compared with a benzene blank.

Concn., %	Quartz, g.	α
0.2	1	+0.011 \pm 0.003
2.0	2	+0.026 \pm 0.002
2.0	2	+0.019 \pm 0.004

The racemization of the complex is slow enough to allow adequate verification of the observed rotation.

Time, hr.	Initial	1.5	5	9
α	+0.019	+0.020	+0.013	0.000

(9) H. S. Booth and D. G. Pierce, *J. Phys. Chem.*, **37**, 59 (1933).

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The Preparation and Properties of Tin(II) Chlorofluoride

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In connection with investigations concerning methods of preparing tin(II) fluoride¹ for use as an anticariogenic agent,² crystallin tin(II) chlorofluoride was found to be formed when hydrofluoric acid was added to a concentrated aqueous solution of tin(II) chloride.

Subsequent studies showed tin(II) chlorofluoride to be very effective in reducing the acid solubility of powdered human dental enamel as well as the incidence of experimental dental caries in rats.³ Moreover the results of a clinical study involving the topical application of tin(II) chlorofluoride solutions to the erupted permanent teeth of school children⁴ indicate that this compound is even more effective in reducing the incidence of new dental caries than tin(II) fluoride when solutions of the two compounds are administered at the same fluoride level.

(1) Wm. H. Nebergall, Joseph C. Muhler and Harry G. Day, *This Journal*, **74**, 1604 (1952).

(2) C. L. Howell, C. Gish, R. Smiley and J. C. Muhler, *J. Amer. Dent. Assoc.*, in press.

(3) J. C. Muhler and H. G. Day, *J. Dent. Res.*, in press.

(4) C. L. Howell and J. C. Muhler, *Science*, **120**, 316 (1954).

The work reported herewith will make it possible for those who may wish to study the anticariogenic activity of tin(II) chlorofluoride to prepare samples of this compound for their own use.

Experimental

The Preparation of Tin(II) Chlorofluoride from Tin(II) Chloride and Hydrofluoric Acid.—Tin(II) chloride dihydrate, 112.8 g. (0.5 mole), was weighed into a 200-ml. polyethylene beaker and 15 g. of oxygen-free, boiling water was added to dissolve the salt. With an atmosphere of oxygen-free nitrogen maintained above the solution, it was heated to 55° on a steam-bath, and 22.5 g. (0.55 mole) of 49.6% hydrofluoric acid was slowly added to the solution. Crystals of the product formed when the mixture was allowed to cool to room temperature. The supernatant liquid was decanted and the crystals were washed three times with 95% ethanol; the alcohol was decanted after each washing. The product was dried in an evacuated desiccator over a 1:1 mixture of anhydrous calcium chloride and potassium hydroxide. The yield of the product was 21.8 g. (25%).

Anal. Calcd. for SnClF: Sn, 68.53; F, 10.97. Found: Sn, 68.7, 68.3, 68.5; F, 10.7, 10.7, 10.8.

Properties of Sn(II) Chlorofluoride.—Tin(II) chlorofluoride crystallizes as colorless prisms which melt at 185–190°. The compound dissolves in water (55% at 25°) to give solutions which are essentially clear initially; within an hour a white precipitate is formed due to hydrolysis of the salt. A 4% solution (the concentration used in topical application⁴) was found to have a pH of 2.0 immediately after preparation and also after standing for two hours.

X-Ray Powder Diffraction Data for Tin(II) Chlorofluoride.—In the following table are listed the interplanar spacings ("d") and the relative line intensities found to be characteristic of SnClF.

5.09 VS	2.68 M	2.27 M	1.61 M	1.39 W
3.89 VS	2.63 VW	2.19 M	1.58 M	1.34 W
3.35 VS	2.53 W	2.02 S	1.48 W	1.25 VW
3.07 M	2.44 W	1.90 M	1.45 W	1.24 VW
2.95 S (diffuse)	2.34 M	1.76 S	1.43 W	1.23 W

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The Orthobaric Density of Pure Nitric Acid¹

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In the course of an investigation of the thermal decomposition of pure nitric acid, it became necessary to know accurately the orthobaric density of the pure acid between 0 and 32°. The "International Critical Tables"² tabulate values of the density of pure nitric acid between 5 and 30° based on a compilation of data published previous to 1927. More recent measurements, however, do not agree well with these data. Consequently, it was decided to measure the liquid density of pure nitric acid at a series of temperatures in this temperature range.

Materials.—Pure nitric acid was prepared by the reaction of 100% sulfuric acid on anhydrous potassium nitrate at 0° and under high vacuum. The nitric acid formed was collected in a container kept at the temperature of liquid nitrogen. The total acidity of the samples obtained in this manner was found, by volumetric analysis, to vary between 99.95 and 100.05%. No trace of NO₂ or of SO₂

(1) This paper is based upon work performed under contract Number AF 33(038)-10381 with W.A.D.C., Wright-Patterson Air Force Base, Ohio.

(2) "International Critical Tables," Vol. III, McGraw-Hill Book Co., New York, N. Y., 1928, p. 59.