The tetrahydrofuran layer was separated and evaporated *in vacuo* to an oil. This oil was fractionally distilled, dissolved in abs. ether and dried over sodium hydroxide pellets. The pure hydrazine was obtained by fractionally distilling this ether solution.

N-Nitrosomethylaniline was reduced to 1-methyl-1-phenylhydrazine[5] in 77% yield which is an oil boiling at 106–109° (13 mm.).

N-Nitrosopiperidine was reduced to N,N-pentamethylenehydrazine[6] in 75% yield. It is an oil boiling at 146–148° (730 mm.).

N-Nitrosodicyclohexylamine was reduced to 1,1-dicyclohexylhydrazine in 48% yield. It is a white crystalline solid boiling at 95–98° (4 mm.) which sublimes above 162°. The hydrochloride forms white flaky crystals from ether, m.p. 238–240° (cor.). The methiodide forms small white plates from abs. ether, m.p. 250–252° (cor.). *Anal.* Calcd. for hydrochloride $C_{12}H_{25}N_2Cl$: Cl, 15.23. Found: Cl, 14.94. *Anal.* Calcd. for methiodide $C_{13}H_{27}N_2I$: I, 37.51. Found: I, 37.06.

Skita and Rolfes[7] attempted to prepare 1,1-dicyclohexylhydrazine by reducing N-nitrosodicyclohexylamine with zinc and hydrochloric acid and obtained dicyclohexylamine. When they used zinc in absolute alcohol with acetic acid no hydrazine could be isolated.

The 1,1-dicyclohexylhydrazine has a nauseating and irritating odor beyond that produced by high molecular weight amines. This compound in lethal doses in white mice produces convulsions characteristic of central nervous system stimulation. The LD_{50} dose in mice by the intraperitoneal route is 77 mg./kg.

---

[5] W. W. Hartman and L. V. Roll, "Organic Syntheses," Coll. Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1943, p. 418.

[6] Knorr, *Ann.*, **221**, 299 (1883).

[7] Skita and Rolfes, *Ber.*, **53**, 1251 (1920).

DEPARTMENT OF PHARMACOLOGY
COLLEGE OF MEDICINE
STATE UNIVERSITY OF IOWA
IOWA CITY, IOWA

Preparation of Telluric Acid

BY H. J. HORNER AND GUY WILLIAM LEONARD, JR.

RECEIVED MARCH 26, 1952

Several methods for the preparation of telluric acid have been reported in the literature and are listed by Gilbertson.[1] These methods either give an impure product or require large excesses of oxidizing agent and special preparation of the tellurium. The preparation as developed by Mathers and co-workers[2] is the most widely used. However, purification of the crude telluric acid thus obtained required repeated recrystallizations from concentrated nitric acid solutions.

Hydrogen peroxide is the ideal oxidizing agent, but when it is used alone or in the presence of an acid, a very large excess of peroxide is needed. In basic solutions the oxidation of tellurium dioxide by a slight excess of hydrogen peroxide proceeds nicely to completion. By using ammonia solutions, the by-products are readily separated from the telluric acid.

Experimental

The commercial grade tellurium dioxide (approximately 76% pure) was found to contain impurities which vigorously catalyzed the decomposition of hydrogen peroxide. The tellurium dioxide was purified by dissolving it in 5 *N* sodium hydroxide. After filtering, the tellurium dioxide was reprecipitated by adding 10 *N* nitric acid to the filtrate until the solution was acid to phenolphthalein. The supernatant liquid was then decanted and the precipitate washed five times with distilled water. The purified material was dried for 24 hours at 110°. This method produced a compound of about 98% purity. Subsequent repetition of the above procedure made no appreciable change in the purity, because of coprecipitation of sodium salts.

Preliminary determinations revealed no apparent reaction between pure tellurium dioxide and 30% hydrogen peroxide. However, in the presence of dilute ammonia solution some oxidation occurred with the formation of a gum which prevented further reaction. Nevertheless the reactions occurring in concentrated ammonium hydroxide produced a white crystalline precipitate of an ammonium tellurate which did not interfere with the oxidation of the tellurium dioxide.

Procedure.—A ten-gram sample of tellurium dioxide, purified and oven-dried, was placed in a 250-ml. erlenmeyer flask fitted with a rubber stopper. To the flask was added a mixed solution of 10 ml. of 30% hydrogen peroxide and 75 ml. of concentrated ammonium hydroxide. After the pressure generated by the initial reaction had subsided the flask was stoppered and set aside. During this period, the flask was frequently shaken. After 24 hours, the mixture was heated on a steam-bath until the vapors were free of ammonia, and the volume of solution had been reduced to 45 ml. Next, 10 ml. of concentrated nitric acid was added to the above solution. During the addition of the nitric acid, the ammonium tellurate dissolved. After 24 hours the supernatant liquid was removed from the crystals of telluric acid which had formed, and the liquid was further concentrated to 20 ml. Another 10 ml. of concentrated nitric acid was added and after an additional 24 hours the crops of crystals were combined. Further purification was accomplished by one recrystallization from distilled water. This method produces telluric acid of 99.99% purity and in yields of 75–80%. The telluric acid was analyzed by the method of Gooch and Howland.[3] The flame test and Nessler reagent showed that the telluric acid was not contaminated with either sodium or ammonium ions.

---

[1] L. I. Gilbertson, THIS JOURNAL, **55**, 1460 (1933).

[2] F. C. Mathers, C. M. Rice, H. Broderick and R. Torney, *Inorganic Syntheses*, **3**, 145 (1950).

[3] F. A. Gooch and J. Howland, *Am. J. Sci.*, [3] **48**, 375 (1894).

DEPARTMENT OF CHEMISTRY
KANSAS STATE COLLEGE
MANHATTAN, KANSAS

The Effect of Fluorine on Praseodymium Trifluoride

BY THEODORE P. PERROS AND CHARLES R. NAESER

RECEIVED MARCH 17, 1952

Since it has been shown that cerium tetrafluoride could be prepared quantitatively from cerous fluoride with fluorine at 500°,[1] it was thought to be of interest to determine what effect elemental fluorine would have on praseodymium trifluoride at this and other temperatures. Klemm[2] fluorinated praseodymium trichloride and obtained a mixture of the chloride and fluoride.

Experimental

Fluorine was prepared by the apparatus described by Von H. von Wartenberg.[1] A graphite anode was used. The rate of production of fluorine was about 35 ml. per minute.

A combustion boat was made from a fluorite cupel since most other substances which might be used for a boat are reacted upon by fluorine at temperatures above 500°.

A nickel tube $^1/_8$" in thickness, $^7/_8$" inside diameter and 14" in length was used to house the combustion boat. The reaction tube was heated to the desired temperatures in an electric combustion furnace. The joints between the apparatus and the reaction tube were sealed with plaster of Paris. Before each run the system was dried by air.

Praseodymium trifluoride was prepared by the addition of hydrofluoric acid to a solution of the trichloride which had been warmed to 80°. The precipitate was filtered and

---

[1] Von H. von Wartenberg, *Z. anorg. allgem. Chem.*, **244**, 337 (1940).

[2] Klemm and Henkel, *ibid.*, **220**, 180 (1934).