NOTES

A New Method of Preparing Phosphorus Pentafluoride.—It has been found that when a mixture of calcium fluoride and phosphorus pentoxide is heated, a gas is evolved, composed largely of phosphorus pentafluoride. The details of the method are as follows. A mixture of 25 g. of phosphorus pentoxide with approximately 55 g. of pure calcium fluoride (previously dried by heating in an iron crucible over a flame) was heated in a 2cm. iron pipe about 30 cm. long, capped at one end and connected to a 1cm. pipe at the other. Glass cannot be used. In order to reduce to a minimum the formation of hydrogen fluoride, the apparatus and materials had to be free from moisture. It was found necessary to bake out the apparatus, to use the best phosphorus pentoxide, to ignite the fluoride, to transfer it while still hot to the hot apparatus, and to measure out the materials rapidly (about 50 cc. of each) instead of weighing. The ingredients were mixed by shaking. The rate of gas evolution depended upon the temperature, and was quite rapid when a large flame was used. The gas fumed in moist air.

The vapor density of the resulting phosphorus pentafluoride was determined in the following way. The gas was allowed to pass under slight pressure for about 45 minutes through a tared bulb provided with two stopcocks. These were then closed, and the bulb was weighed, but not until one of the stopcocks was opened for a few seconds to liberate gas, of which some always escaped. The best value obtained gave a molecular weight of 119, instead of 126 required for PF_5. If the main impurity was hydrogen fluoride, and if it was in the monomolecular form at the low concentration prevailing, the gas had an approximate composition of 94% of phosphorus pentafluoride and 6% of hydrogen fluoride. The absorption of a small amount of water by the phosphorus pentoxide accounts for the presence of hydrogen fluoride.

The probable reaction is $5CaF_2 + 6P_2O_5 \longrightarrow 2PF_5 + 5Ca(PO_3)_2$. Although the gas is not pure, this method is superior to those previously described, since the necessary materials are easily available and the rate of gas evolution can be controlled.

Potassium fluoride is not a satisfactory substitute for the calcium salt since it is more difficult to obtain dry; moreover, it reacts too rapidly with the pentoxide.

We were unable to obtain any *iso*-amyl fluoride on passing phosphorus pentafluoride for one and one-half hours into boiling, anhydrous *iso*-amyl alcohol in a copper vessel under a copper reflux condenser.

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RECEIVED NOVEMBER 9, 1926
PUBLISHED MAY 10, 1927