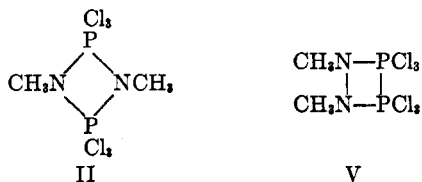






volatile than the dimer, again ruling out higher polymers.

The proton magnetic resonance spectrum of II was obtained in phosphorus oxychloride at 105° to differentiate between the two possible isomeric structures, II and V.<sup>5</sup> The spectrum was a simple



1:2:1 triplet which would result from the coupling of each proton with two symmetrically disposed phosphorus atoms as in II. The proton magnetic resonance spectrum of V would consist of a doubled doublet—i.e., four lines of equal intensity. This confirms structure II. Chapman *et al.*<sup>5</sup> also proposed structure II on the basis of infrared and Raman spectra and other data.

The unusual stability of this compound, as indicated by its thermal stability and ascribed by Chapman<sup>5</sup> to the delocalization of the ring electrons, is being investigated further.

Michaelis<sup>6</sup> proposed an alternating phosphorus-nitrogen four-membered ring for his phosphinimine dimer (C<sub>6</sub>H<sub>5</sub>N—POCl)<sub>2</sub>, and it is likely that the *N*-phenyltrichlorophosphinimine reported by Gilpin<sup>7</sup> was also a dimer containing the same four-membered phosphorus-nitrogen ring.

It seems reasonable that compound II, its homologs, and analogs have previously been formed in other reactions of phosphorus pentachloride and *N*-alkyl- or *N*-arylamides of various organic acids but have gone undetected, either because they occurred only in small amounts, or because the imidoyl chlorides were used without purification.

#### EXPERIMENTAL

**Reaction of *N*-methyltrifluoroacetamide with phosphorus pentachloride.** One hundred and forty grams (1.1 moles) of *N*-methyltrifluoroacetamide and 230 g. (1.1 moles) of phosphorus pentachloride were combined in a 500-ml. round bottomed flask fitted with a Vigreux column and a distillation head leading to a Dry Ice-acetone trap. The reaction vessel was heated to melt the amide and gas evolution commenced immediately. Heating was continued to maintain a brisk evolution of gas. Gas evolution ceased when all the phosphorus pentachloride had dissolved. Heating was continued until the distillate temperature reached 60°.

***N*-Methyltrifluoroacetimidoyl chloride (I).** The condensate in the trap was distilled to give 49 g. of low boiling material and 70 g. (44% yield) of *N*-methyltrifluoroacetimidoyl chloride, b.p. 47–48°/705 mm. The infrared absorption spectrum showed a very strong sharp band at 5.90 μ attributed to the >C=N<sup>-</sup>, and the typical trifluoromethyl group bands in the 8 to 9 μ region.

(5) The NMR spectrum of II was recorded and interpreted by Donald W. Moore of this laboratory.

(6) A. Michaelis, *Ann.*, 407, 290 (1915).

(7) J. E. Gilpin, *Am. Chem. J.*, 29, 352 (1897).

*Anal.* Calcd. for C<sub>3</sub>H<sub>5</sub>ClF<sub>3</sub>N: C, 24.76; H, 2.07; Cl, 24.37 F, 39.17. Found: C, 24.68; H, 2.28; Cl, 24.62; F, 39.20.

**Trifluoroacetyl chloride.** The 49 g. of low boiling material, which was recondensed in a Dry Ice-acetone trap, contained an appreciable amount of solid at -78°. An infrared spectrum at 15 mm. pressure of the gas, formed by warming the condensate, was identical with the spectrum of trifluoroacetyl chloride at the same pressure. At 440 mm. pressure the infrared spectrum of the gas from the condensate, gave, in addition to the trifluoroacetyl chloride spectrum, bands at 3.38 μ and 3.54 μ, due to the presence of hydrogen chloride,<sup>8</sup> and a small peak at 5.90 μ, due to the presence of a small amount of *N*-methyltrifluoroacetimidoyl chloride.

Trifluoroacetyl chloride and hydrogen chloride do not form a solid complex at -78°, but *N*-methyltrifluoroacetimidoyl chloride, a liquid at -78°, combines with hydrogen chloride to give a solid which accounts for the solid in the trap at -78°. The solid was warmed slowly from -78° to -20° with no sign of melting or dissociation. Above -20° shrinkage began to occur and at -10° the solid liquified and gas evolution occurred.

**1,3-Dimethyl-2,2,2,4,4,4-hexachloro-1,3,2,4-diazadiphosphetidine (II).** The residue in the initial reaction vessel was heated on the steam bath under reduced pressure to remove the phosphorus oxychloride, leaving 87 g. (52% yield) of 1,3-dimethyl-2,2,2,4,4,4-hexachloro-1,3,2,4-diazadiphosphetidine, m.p. 170–176°. Sublimation at 140°/0.1 mm., gave II, m.p. 174–179°. When the melt was further heated in the capillary melting point tube gas evolution occurred at 250°.

*Anal.* Calcd. for CH<sub>2</sub>Cl<sub>2</sub>NP: C, 7.22; H, 1.82; Cl, 63.93; N, 8.42; P, 18.61. Found: C, 7.63; H, 2.41; Cl, 63.16; N, 8.42; P, 18.46.

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(8) R. H. Pierson, A. N. Fletcher, and E. St. Clair Gantz, *Anal. Chem.*, 28, 1239 (1956).

### Synthesis of Phenyl-1,2-ethenediol Cyclic Carbonate<sup>1</sup>

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Previous reports describe the synthesis and polymerization of 1,2-ethenediol cyclic carbonate (I, vinylene carbonate).<sup>2</sup> Compared to other 1,2-disubstituted ethylenes, the ring structure of I appeared to enhance the reactivity in vinyl poly-

(1) Presented in part before the Division of Organic Chemistry at the 140th National Meeting, American Chemical Society, Chicago, Ill., September 1961.

(2) (a) M. S. Newman and R. W. Addor, *J. Am. Chem. Soc.*, 75, 1263 (1953); 77, 3789 (1955). (b) K. Hayashi and G. Smets, *J. Polymer Sci.*, 27, 275 (1958). (c) J. M. Judge and C. C. Price, *J. Polymer Sci.*, 40, 439 (1959). (d) C. G. Overberger, H. Bilech, and R. G. Nickerson, *J. Polymer Sci.*, 27, 381 (1958). (e) W. K. Johnson and T. L. Patton *J. Org. Chem.*, 25, 1042 (1960).