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# 1,3-DIMETHYL-1,3,2-DIAZAPHOSPHOLANES

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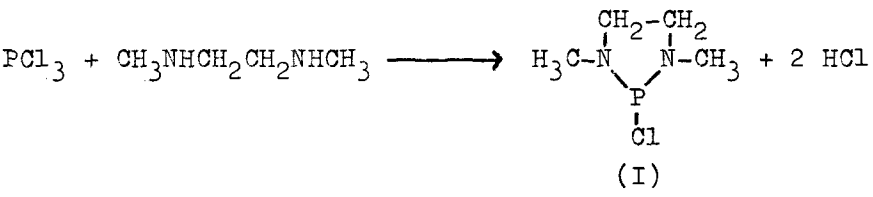
In the course of studies on the reactions of dimeric phenyliminophosphorictrichloride with ammonia, primary and secondary amines (1, 2) the reaction with N-N'-dimethyl-ethylenediamine was included. The formation of 1,3-dimethyl-2-chloro-2-phenylimino-1,3,2-diazaphospholane might be expected, but actually a mixture of unidentified polymers was obtained.

1,3-dimethyl-2-chloro-1,3,2-diazaphospholane (I) was, however, obtained according to the reaction

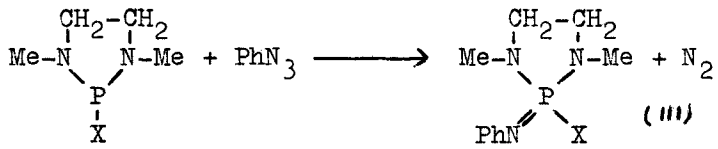


in 72 % yield, Bp. 39-40° at $10^{-1}$ torr, $n_D^{20} = 1,5300$.

In an analogous manner the corresponding oxo-compound was obtained, when phosphorus oxytrichloride was used in the place of phosphorus trichloride. The yield of 1,3-dimethyl-2-oxo-2-chloro-1,3,2-diazaphospholane is 67 %, its f.p. 213-215°.

The chlorophospholane (I) was found to react with dimethylamine to give 1,3-dimethyl-2-dimethylamino-1,3,2-diazaphospholane (II) in 89 % yield. B.p. 58-59°, $n_D^{20} = 1,4869$.

(I) or (II) can be made to react in ether with phenyl azide to give the corresponding phenylimino compounds:



(III)

(IIIa) X = Cl, yield 67 %, f.p. 34-36°

(IIIb) X = $Me_2N$, yield 93 %, f.p. 30-31°

The imino compounds are characterized by an intense IR-band at 1220 $cm^{-1}$ (IIIa) and 1190 $cm^{-1}$ (IIIb) respectively, due to the P=N valence frequency. The "oxidation" of phosphorus is also evident from the results of an $^{31}P$ NMR investigation. The absorptions of the P nuclei are found for (I) and (II) at $-172{,}8.10^{-6}$ and $-116{,}5.10^{-6}$ respectively, while for (IIIb) a signal is found at $-7{,}6.10^{-6}$ (all in relation to 85 % $H_3PO_4$ as external standard).

References:

(1) V.Gutmann, Ch.Kemenater and K.Utvary, Monatsh. 96, 836 (1965)

(2) V.Gutmann, Ch.Kemenater and K.Utvary, to be published