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Novel Mild Synthesis of Phosphorus Acid Anhydrides

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Novel Mild Synthesis of Phosphorus Acid Anhydrides

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Our recent work on ligand exchange in chlorophosphonium salts and chlorophosphoranes as well as on the reactions of phosphorus pseudohalogens with $\mathsf{P}^{\mathsf{III}}$ compounds resulted in the novel methods for the synthesis of various phosphorus and carboxylic acids anhydrides.

We have found that: i) Ph_3PCl_2 is an excellent reagent for the transformation of organic acids into anhydrides 1 and 2.

and diselenopyrophosphates 4.

$$[R_2P(0)X]_2 = \frac{Ph_3P}{-100^{\circ}C} = [R_2P(X)]_2O + Ph_3PO$$

iii) Reactions of compounds 3 or thiono(selenono)phosphates 5 with dialkyltrimethylsilyl phosphites are two alternative approaches to sym. monothio- and monoselenopyrophosphates 6

$$3 - \frac{R_3^{i}POSiMe_3}{-100^{\circ}C} - R_2P(0)XP(0)R_2^{i} + R_2P(X)OSiMe_3$$

$$2 R_2P(X)OSiMe_3 + SO_2Cl_2 + R_2POSiMe_3 - \frac{-75^{\circ}C}{} - \frac{6}{} + SO_2 + Me_3SiCl_3$$

$$X=S_2Se$$

All the reactions presented here are completed in few minutes even in very low temperatures giving the anhydrides of high purity in almost quantitative yields. Low temperature n.m.r. investigations showed that the phosphonium salts such as $Ph_3\dot{P}-0-P(0)<$; $Ph_3\dot{P}-0-P(S)<$ and $>\dot{P}-X-P(0)<$ are intermediates in the reactions studied.