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Synthesis and Reactivity of Hydrolysis Products of Cyclic Phosphites with Amino Substituents

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SYNTHESIS AND REACTIVITY OF HYDROLYSIS PRODUCTS OF CYCLIC PHOSPHITES WITH AMINO SUBSTITUENTS

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Abstract. Hydrolysis of several cyclic aminophosphites are discussed and compared with that of cyclic chloro/ phenoxy phosphites. An X-ray structure of a hydrolysis product, $(C_6H_1NH_3)^+O^-P(O)(H)(2,2'-OC_6H_4-C_6H_4OH)$ has been determined.

The cyclic phosphite (C₆H₁₁NH)P{2,2'-O₂(C₆H₄)₂}(1) undergoes hydrolysis in aqueous medium to afford the acyclic salt (C₆H₁₁NH₃)⁺O⁻P(O)(H)(2,2'-OC₆H₄-C₆H₄OH) (2) which is characterized by ¹H and ³¹P NMR, elemental analysis as well as a single crystal X-ray structural analysis. Compound 2 [m.p. 180°C; ³¹P NMR : 2.98 ppm] upon thermal treatment loses the cyclohexyl

amino group to lead to a phosphite with a P-H group $[^1J(P-H) = 732 \text{ Hz}]$. A similar feature is observed for the amine salt $(C_6H_1NH_3)^+O^-P(O)(H)(OCH_2CMe_2-CH_2OH)$; in this case, however, one of the products is identified as the cyclic phosphite $(H)(O)P(OCH_2CMe_2CH_2O)$ which is characterized by its Pudovik product with p-chlorobenzaldehyde. $[^1J_1]$

Although the phenoxy compound (PhO)P(OCH₂CMe₂CH₂O) hydrolyses to the <u>ring</u> (phosphorinane) <u>preserved</u> product H(O)P(OCH₂CMe₂CH₂O),² the oxinate (NC₉H₆O)P(OCH₂CMe₂-CH₂O) leads to the <u>ring opened</u> product (C₉H₆ONH⁺)O⁻P(O)(H)(OCH₂CMe₂-CH₂OH).

Molecular structure of 2; H atoms not shown. Selected distances (Å) and bond angles (°): P1-O1 1.482; P1-O2 1.471; P1-O3 1.589; P-H 1.33; O1...O4 2.71; O1-P1-O2 119.8; O1-P1-O3 110.6; O2-P1-O3 104.3.

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