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Anastasy Kolodiazhna: Synthesis, Properties and stereochemistry of 2-Halo-1,2λ5-oxaphosphetanes

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Abstract

Results of research into four-membered 2-halo-1,2λ5-oxaphosphetane phosphorus(V)-heterocycles are presented. The preparation of 2-halo-1,2λ5-oxaphosphetanes by reaction of P-haloylides with carbonyl compounds is described. The mechanism of asynchronous [2+2]-cycloaddition of ylides to aldehydes was proposed on the base of low-temperature NMR investigations. 2-Halo-1,2λ5-oxaphosphetanes were isolated as individual compounds and their structures were confirmed by 1H-, 13C-, 19F- and 31P-NMR spectra. These compounds are convenient reagents for preparing of various organic and organophosphorus compounds hardly available by other methods. Chemical and physical properties of the 2-halo-1,2λ5-oxaphosphetanes are reviewed. The 2-chloro-1,2λ5-oxaphosphetanes, rearrange with formation of 2-chloroalkyl-phosphonates or convert into trans-phosphorylated alkenes depending on the substituents at the α-carbon atom. Prospective synthetic applications of 2-halo-1,2λ5-oxaphosphetanes are analyzed. The 2-halo-1,2λ5-oxaphosphetanes may be easily converted to various alkenylphosphonates: allyl- or vinylphosphonates, phosphorus ketenes, thioketenes, ketenimines.

Keywords: 2-halo-1,2λ5-oxaphosphetanes, allylphosphonates, vinylphosphonates, phosphorus ketenes

Background

One of the most interesting and intriguing classes of organophosphorus compounds are the 1,2-oxaphosphetanes—four-membered heterocycles containing pentacoordinated phosphorus [1,2,3,4,5,6,7]. Since 1,2-oxaphosphetanes are well-known intermediates in the Wittig reaction, a number of efforts have been made for their structural characterization both in solution and the solid state [8,9,10,11,12,13].

In 1967, Birum and Matthews had already reported the structural characterization (NMR and X-ray study) of the first isolated 1,2-oxaphosphetane 1. Compound 1 was prepared in 76% yield by allowing hexaphenylcarbodiphosphorane to react with hexafluoroacetone in dry diglyme [14] (Scheme 1).

Vedejs [3,4] succeeded in detecting of 1,2-oxaphosphetanes 2–4 by low temperature NMR spectroscopy during typical Wittig reactions and observed that these intermediates readily decompose upon warming to room temperature into alkenes and phosphine oxides (Scheme 2).

Schmutzler and co-workers reported several stabilized bis(trifluoromethylated) oxaphosphetanes 5, 6 (Scheme 3) [15] which were characterized by NMR, MS spectra and X-ray analysis. At room temperature Berry-pseudorotation was fast on the NMR time scale, impeding one from distinguishing apical and equatorial P-CF3 groups. Decreasing the temperature to −60 °C in toluene-d8 allowed resolving the signals for all CF3 groups of molecule 7 [16].

Kojima [17] reported the interesting anti-apicophilic spirophosphorane 8 bearing an oxaphosphetane ring. The structure of compounds 8 was confirmed by X-ray diffraction. Crystallization from hexane gave the pure anti-apicophilic derivative. Stereomutation of compound 8 was observed in the presence of acids and slowed down when DBU was present, suggesting that the isomerization into 9 is rather the result of a P–O bond breaking-recombination process. Evidently, this conversion represents an example of a thermodynamically

stable oxaphosphetanes, in which pseudorotation is faster than alkene formation (Scheme 4).

Most of the previously reported stable oxaphosphetane structures contain fluorine-bearing or bicyclic phosphole-type ligands either at the phosphorus position or at the 4 position in the oxaphosphetane ring 10, 11 [15,16,17,18,19,20]. Recently, Streubel and coworkers have prepared the first 1,2-oxaphosphetane complexes 12 formally similar to traditional oxaphosphetanes, using low-temperature ring expansion of epoxides with a Li/Cl phosphinidenoid complex [18] (Scheme 5).

Gilheany studied oxaphosphetane intermediates in the Wittig reaction by variable-temperature NMR spectroscopy [9,10,11]. Compound 13 was obtained by low-temperature acid quenching of the Wittig reaction of ylide with benzaldehyde, a suitable representative aromatic aldehyde (see Scheme 6). The major diastereomer was the syn-13 on the basis that the unquenched Wittig reaction gives the (Z)-alkene as the major product. In this manner, the syn/anti ratio of 13 was 89:11.

Keglevich reported detection of enantiomers of P-stereogenic pentacoordinated phosphorus compounds [20]. Detailed 31P-NMR investigations of oxaphosphetes in optically active solvents have clearly shown that the most electronegative substituents (e.g., oxygen) prefers the apical position in a trigonal bipyramidal structure and that the pentacoordinated phosphorus atom is in a dynamic condition due to pseudorotation. Berger and coworkers found that 2-furyl groups on the phosphorus atom increase the thermal stabilities of oxaphosphetanes and succeeded in isolation and determination of the X-ray structure of tris(2-furyl) substituted oxaphosphetane, the stability of which is attributed to the electron-withdrawing properties of the 2-furyl group [21].

Among the stable oxaphosphetanes [1,2,3,4 ,5,6,7,8,9,10,11,12,13,14,15,16,17,18,19,20,21,22] , 2-halo-1,2λ5-oxaphosphetanes 14, which possess relatively high stability and diverse reactivity, attract particular interest [23,24,25,26,27,28,29,30,31,32, 33,34,35,36,37,38] (Scheme 7). These oxaphosphetanes containing fluorine, chlorine or bromine atoms bonded to phosphorus are an interesting class of pentacoordinated phosphorus heterocycles possessing peculiar properties. The chemical properties of 2-halo-1,2λ5-oxaphosphetanes, first of all of P-chloro- and P-fluoroylides, due to the presence of a labile halogen atom on phosphorus, are very specific and differ from the properties of triphenylphosphonium ylides. For example, reactions and conversions of 2-halo-1,2λ5-oxaphosphetanes proceed with preservation of the P-C bond and leads to the formation of different organophosphorus compounds. In addition, this type of compounds exhibit also physico-chemical properties uncharacteristic for traditional P,P,P-triorganosubstituted oxaphosphetanes. For the first time the 2-halo-1,2λ5-oxaphosphetanes were prepared in our laboratory almost twenty years ago and up to today we and some other authors are still studying their chemistry. In this article, we summarize the synthesis and properties of this type of organophosphorus compounds. The chemistry of 2-halo-1,2λ5-oxaphosphetanes was not previously analyzed, generalized or reviewed.

Results and Discussion

General Description

Since pentacoordinated phosphoranes formally have 10 electrons in the valence shell, they display a specific bonding model. Therefore pentacoordinated phosphoranes take a trigonal bipyramidal structure and there are two ligating sites, apical and equatorial sites. The apical bond consists of three-center four-electron bond using the p orbital of the central phosphorus atom, while the equatorial bond is a typical s bond using sp2 hybrid orbital of the phosphorus atom. This three-center four-electron bond forms three molecular orbitals.

It is generally known that pentacoordinated phosphoranes rapidly undergo intramolecular positional isomerization without bond cleavage. A very rapid non-dissociative intramolecular site exchange is usually explained by the Berry pseudorotation mechanism [19,22,23].

The 2-halo-1,2λ5-oxaphosphetanes (halogen = chlorine, bromine, fluorine) are the most stable representatives of this type of compounds. They can be purified by distillation under vacuum and stored in a refrigerator. At the same time they possess interesting chemical properties and participate in various chemical transformations [24,25,26,27,28].

The stability of 2-halo-oxaphosphetanes changes in the same sequence of substituents R4 at the endocyclic carbon atoms at position 4. The most stable are compounds containing strong electron-accepting groups at C(4), drawing off electron density from the oxygen atom as a result of which the three-centre apical bond O—P—Hal is strengthened. Chloro-oxaphosphetanes, having less electron-accepting substituents at C(4), are dissociated to a large extent and correspondingly are converted into vinylphosphine oxides at room temperature.

Synthesis of 2-Halo-1,2λ5-oxaphosphetane

Available methods for the synthesis of 2-halo-1,2λ5-oxaphosphetanes can be used for investigation of the reaction mechanism of phosphorus ylides with carbonyl compounds as well as for pre-

paring stable oxaphosphetanes that can be used as reactants for organic synthesis. The 2-halo-1,2λ5-oxaphosphetanes were prepared by reaction of P-fluoro-, chloro- or bromoylides with carbonyl compounds. P-Chloro- and P-bromoylides react with active ketones, containing a trifluoromethyl group, with the formation of stable [2+2]-cycloaddition products, 2-chloro- or 2-bromo-1,2λ5-oxaphosphetenes 14 were isolated in yields close to quantitative as crystalline substances or as liquids distillable in vacuum (Scheme 8, Table 1) [29,30,31,32,33,34,35,36,37,38,39,40,41,42]. The addition of P-halogen-ylides to ketones proceeded stereoselectively and led predominantly to the formation of one of the possible 2-halo-1,2λ5-oxaphosphetane diastereomers. 2-Halo-1,2λ5-oxaphosphetanes dissociate at the P-halogen bond in solution with the formation of cyclic phosphonium salts, as a result of which an equilibrium is established between the forms with five- and four-coordinate phosphorus atoms. Dissociation of 2-halo-1,2λ5-oxaphosphetanes is enhanced by reducing the electron-accepting properties of substituents and also by increasing priority solvent. The 2-halo-1,2λ5-oxaphosphetanes containing electron-accepting groups at C(4) are distinctly stabler than oxaphosphetanes with alkyl groups in this position [25,26,27].

The reaction of P-bromomethylides with fluorinated acetophenone afforded in high yield oxaphosphetanes 15, which were isolated as crystalline compounds (Scheme 9 and Table 1, entries 10–13). The compounds 15 exist in solution as cyclic phosphonium salt.

At the same time the 2-chloro-1,2λ5-oxaphosphetanes 16 exist as mixture of P(IV) and P(V)-forms. These compounds can be distilled under vacuum and dissolved in non-polar solvents (benzene) (Scheme 10 and Table 1, entries 1–9). Reaction of P-Fluoroylids with aldehydes and ketones proceeds in ether or pentane at −40−−20 °C and leads to the formation of stable 2-fluoro-1,2λ5-oxaphosphetanes 17.

The compounds 17 are liquids distilling in vacuo, the structure of which was proved by means of mass and NMR spectra. The 31P-NMR spectra of 2-fluoro-oxaphosphetanes 17 present doublets with 800 Hz 1JPF constants in the high magnetic field of a NMR spectrum at −37−−8 ppm. This corresponds to a pentacoordinate state of compounds 17 [24,26,27,28,29,30,31]. Tetracoordinated forms of 2-fluoro-oxaphosphetanes 17 were not registered by 19F- and 31P-NMR spectroscopy (Scheme 11).

C-Silyl-P-chloroylides 18 react with carbonyl compounds to afford 2-chloro-1,2λ5-oxaphosphetanes 19. The oxaphosphetanes 19 bearing an electronegative CF3 substituent at C-4 are relatively stable and can be isolated and analyzed by NMR (Scheme 12). The NMR spectra of these compounds reveal signals at 0.2 ppm, singlet (Me3Si), at 2 ppm, doublet, 3JPH 18.0 Hz (C3H), and at 4.5 ppm (C4H). 13C-NMR signals of C-3 and C-4 carbons were found at 30 and 90 ppm, correspondingly. The 31P-NMR signals of 2-chloro-1,2λ5-oxaphosphetanes 19 at δP +48 ppm (R1 = i-PrO) and at δP = 60 ppm (R1 = Et2N) correspond to tetracoordinate phosphorus included in four-membered phosphetane cycle (Table 1) [25,32].

Sotiropulos and Bertrand [33] reported the addition of phosphacumulene ylides 20 bearing the diazo group to isocyanates, leading to the formation of products 22. It was proposed that initial nucleophilic attack of the ylide carbon atom on the carbonyl carbon gives a oxaphosphetane 21, which depending on the relationship of the oxygen atom (or NR group) to nitrogen or phosphorus rearranges into products of 1,4- or 1,5-cyclisation 22 (Scheme 13). Benzaldehyde gives the 2-chlorooxaphosphetane 23 with the ylide 20 which readily eliminates hydrogen chloride and a nitrogen molecule being converted into an acetylene phosphonate 24 (Scheme 14).

A number of 2-fluoro-1,2λ5-oxaphosphetanes 25 were prepared by reaction of P-fluoroylides with aldehydes and ketones (Scheme 15, Table 2). The 2-fluoro-1,2λ5-oxaphosphetanes 25 are stable compounds, which can be isolated and purified by distillation under vacuum or by crystallization from non-polar solvents. These compounds are much distinguished from unstable adducts of carbonyl compounds with triphenylphosphonium ylides. Cycloadducts of P-fluoroylides with carbonyl compounds, 2-fluoro-1,2λ5-oxaphosphetanes, are also much more stable than 2-chloro- or 2-bromo-1,2λ5-oxaphosphetanes [25,26,27,28,29,30,31].

The stability of 2-fluoro-oxaphosphetanes is explained by the high electronegativity of the fluorine atom, compared to the electronegativities of chlorine and bromine. The P-F bond in 2-fluoro-1,2λ5-oxaphosphetanes is very strong, and, therefore, these compounds do not dissociate with formation of cyclic phosphonium salts, what is observed, for example, with 2-chloro-1,2λ5-oxaphosphetanes. Various stable 2-fluoro-1,2λ5-oxaphosphetanes were synthesized, isolated as pure specimens, and characterized (Table 2). Typical representatives of such compounds 26–29 are shown in Scheme 16, Scheme 17 and Scheme 18 [26,29].

The 31P-NMR spectra of compounds 25 show a doublet at −6 to −45 ppm with 760–850 Hz PF coupling constants appropriate for axial fluorine atoms.

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