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SHORT COMMUNICATIONS

Epoxidation of 4,5-dialkyl-2,3-dihydro-1*H*-phosphole 1-oxides

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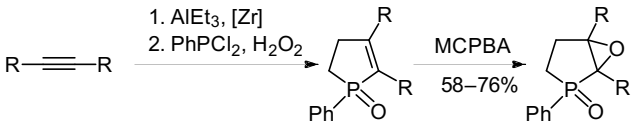


A method was developed for the synthesis of 1,5-dialkyl(cycloalkyl,phenyl)-2-phenyl-6-oxa-2-phosphabicyclo[3.1.0]hexane 2-oxides from alkynes, involving the epoxidation of unsaturated cyclic organophosphorus compounds with *m*-chloroperbenzoic acid. The organophosphorus compounds were obtained by a reaction sequence consisting of catalytic cycloalumination of symmetrical acetylenes with triethylaluminum in the presence of bis(cyclopentadienyl)zirconium(IV) dichloride catalyst leading to the formation of 2,3-disubstituted aluminacyclopent-2-enes and *in situ* reaction of the latter with dichlorophenylphosphine.

Keywords: aluminacyclopentenes, 2,3-dihydrophospholes, epoxyphospholanes, heterocyclic compounds, organoaluminum compounds, zirconocene dichloride, cycloalumination, epoxidation, metal complex catalysis.

The growing interest toward epoxidated phospholanes is motivated by their possible applications as synthetic intermediates for the preparation of nucleoside phosphosugar derivatives, such as azidothymidine (AZT),[1] ribavirin,[2] 4'-thiodideoxynucleosides,[3] and aristeromycin,[4] which have shown promising activity as HIV protease inhibitors, antitumor and antibacterial drugs. Besides that, phosphorus compounds in general tend to have pronounced biological activity. For example, the phosphorus compound bialophos is known as an antifungal agent,[5] while phosphonomycin, which combines phosphonate and epoxide groups in its molecular structure, is used as a broad spectrum antibiotic.[5,6] As recently demonstrated by Yamashita and coauthors,[7] some substituted phospholanes exhibit antitumor activity against leukemia cells.

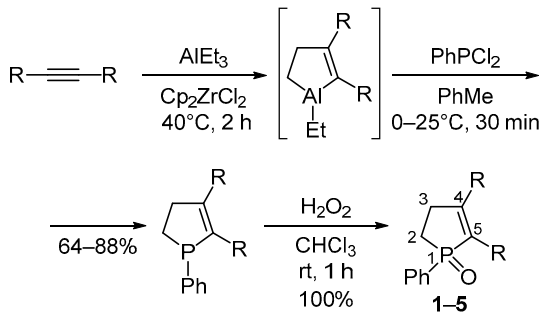
Taking into account the great medicinal potential of cyclic phosphorus compounds, the chemistry of phosphosugars and their analogs represents one of the most interesting and rapidly growing fields of medicinal chemistry, while modification of 2,3-dihydrophospholes provides an effective tool of synthetic exploration, enabling the preparation of practically significant cyclic organophosphorus compounds (OPC) with the required structures.

During our studies aimed at the development of preparative methods for the synthesis of cyclic OPC, we explored the epoxidation of our obtained compounds using the example of 4,5-disubstituted 2,3-dihydro-1*H*-phosphole 1-oxides in reactions with known epoxidating agents (peroxy acids and metal peroxides).

The starting 2,3-dihydro-1*H*-phosphole 1-oxides were obtained according to a published procedure.[8–10] The aluminacyclopent-2-enes obtained *in situ* from disubstituted symmetrical acetylenes and $AlEt_3$ reacted with $PhPCl_2$ in PhMe over 30 min, resulting in the replacement of Al with P atoms, leading to the formation of the respective 2,3-dihydrophospholes in 64–88% yields. The obtained 2,3-dihydrophospholes were then converted to 2,3-dihydro-1*H*-phosphole 1-oxides **1**–**5** in quantitative yields by treatment with H_2O_2 (Scheme 1).

The earliest studies used Na_2O_2 as epoxidating agent.[11,12] However, the reactions of 4,5-disubstituted 2,3-dihydro-1*H*-phosphole 1-oxides **1**–**5** with an excess of Na_2O_2 in ethanol at various temperatures from 30 to 70°C did not produce the expected epoxides even after 6 h.

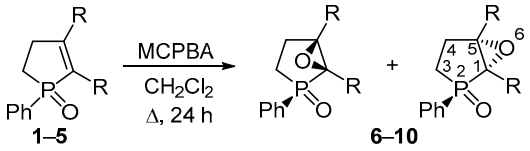
Scheme 1



1 R = Et, **2** R = *n*-Pr, **3** R = *n*-Bu, **4** R = Ph, **5** R + R = $(CH_2)_{10}$

The application of a more advanced epoxidating agent – MCPBA,[13] which is widely used in alkene epoxidation reactions, was more successful. 4,5-Disubstituted 2,3-dihydro-1*H*-phosphole 1-oxides **1**–**5** were found to undergo epoxidation reaction with a triple excess of MCPBA upon refluxing in CH_2Cl_2 (Scheme 2).

Scheme 2



6 R = Et (69%), **7** R = *n*-Pr (74%), **8** R = *n*-Bu (76%)
9 R = Ph (70%), **10** R+R = $(CH_2)_{10}$ (58%)

The by-products were removed by extracting the organic layer with aqueous $NaHCO_3$ solution. The products were isolated as light-yellow oily liquids.

The new 1,5-dialkyl(cycloalkyl,phenyl)-2-phenyl-6-oxa-2-phosphabicyclo[3.1.0]hexane 2-oxides were structurally characterized by ^1H, ^{13}C, and ^{31}P NMR spectroscopy. Thus, ^{13}C NMR spectra of compounds **6**–**10** contained characteristic signals in the range of 64.9–74.0 ppm instead of the signals of sp^2-hybridized carbon atoms, providing evidence that the epoxidation reaction had indeed occurred (Fig. 1). Besides that, the chemical shift of phosphorus atom in ^{31}P NMR spectra of the target products was shifted upfield by 8–12 ppm relative to the ^{31}P NMR signal of the starting 2,3-dihydrophosphole 1-oxides (62–64 ppm). The experimentally determined chemical shift values, as well as the heteronuclear coupling constants (J_{PC}) for the C-1 carbon atoms (96.6–102.7 Hz) were in agreement with the published spectral data for the structurally related 2-methyl(phenyl)-6-oxa-2-phosphabicyclo[3.1.0]hexane 2-oxides.[14–16]

The epoxidation reaction of 2,3-dihydro-1*H*-phosphole 1-oxides **1**–**5** was found to be nonstereoselective, since the entire series of the synthesized compounds existed as diastereomers in 1:2 ratio. It should be noted that the isomers of compounds **9** and **10** had different ^{31}P NMR chemical shifts (Fig. 2), while 2-phenyl-6-oxa-2-phosphabicyclo[3.1.0]hexane 2-oxide 1,5-dialkyl derivatives **6**–**8** had ^{31}P NMR signals with similar chemical shifts that merged into one signal, which was broadened due to the interaction with protons bonded to the ring carbon atoms. In order to correctly determine the chemical shift values in ^{31}P NMR spectra of 2-phenyl-6-oxa-2-phosphabicyclo[3.1.0]-hexane 2-oxide 1,5-dialkyl derivatives **6**–**8**, the spectra were acquired with suppression of the proton signals.

Thus, we have demonstrated that 4,5-disubstituted 2,3-dihydro-1*H*-phosphole 1-oxides in reactions with an excess of *m*-chloroperbenzoic acid produced the respective epoxides in 58–76% yields. The obtained compounds may be of considerable practical interest.

Experimental

^1H, ^{13}C, and ^{31}P NMR spectra were acquired on a Bruker Avance-400 instrument (400, 100, and 162 MHz, respectively) in $CDCl_3$. The two-dimensional homonuclear

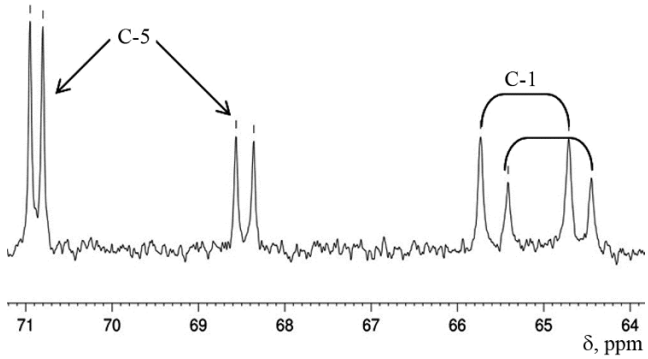


Figure 1. Fragment of ^{13}C NMR spectrum of epoxidated phospholane **10**.

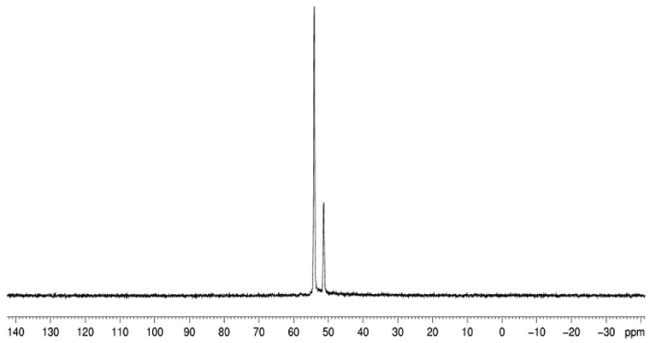


Figure 2. ^{31}P NMR spectrum of epoxidated phospholane **10**.

(COSY) and heteronuclear (^1H–^{13}C HSQC, ^1H–^{13}C HMBC) NMR spectra were acquired according to the standard procedures from Bruker. Mass spectrum of compound **8** was recorded on a Bruker Autoflex-III MALDI TOF/TOF instrument for samples on 2,5-dihydroxybenzoic acid and α-cyano-4-hydroxycinnamic acid matrices in reflective mode, with recording of positive ions. Mass spectra (GC-MS) of the other compounds were recorded on a Shimadzu GC-2010 instrument equipped with a GCMS-QP2010 Ultra mass selective detector and a Supelco 5ms capillary column (60 m × 0.25 mm × 0.25 μm); the carrier gas was helium; injector temperature 260°C, interface temperature 260°C, ion source temperature 200°C. Elemental analysis was performed on a Carlo Erba 1106 elemental analyzer. Chromatographic analysis was performed on a Shimadzu GC-9A gas chromatograph, using a 2000 × 2 mm column, the stationary phase consisted of silicone SE-30 (5%) on Chromaton N-AW-HMDS carrier (0.125–0.160 mm), the carrier gas was helium (30 ml/min), temperature program from 50 to 300°C at the rate of 8°C/min. Column chromatography was performed with Acros silica gel (0.060–0.200 mm). The reactions with organometallic compounds were accomplished under dry argon flow. The solvents were dried and distilled immediately prior to the use. Commercially available Cp_2ZrCl_2, phosphines (Acros), and 92% $AlEt_3$ (from Redkinsk Experimental Factory) were used.

Preparation of 4,5-disubstituted 2,3-dihydro-1*H*-phosphole 1-oxides 1–5 (General method). A round-bottom flask was charged sequentially with Cp_2ZrCl_2 (0.15 g, 0.5 mmol), alkyne (10 mmol), and $AlEt_3$ (1.37 ml,

10 mmol) with stirring at 0°C under dry argon atmosphere. The temperature was raised to 40°C, and the mixture was stirred for 4 h, then diluted with PhMe (20 ml), the mixture was cooled to –5°C, treated by dropwise addition of $PhPCl_2$ (1.36 ml, 10 mmol), and stirred at room temperature for an additional 30 min. The reaction mixture was treated with saturated aqueous NH_4Cl solution; the reaction products were extracted with Et_2O, dried over anhydrous $MgSO_4$, and the solvent was removed by evaporation. The residue was taken up in $CHCl_3$ (10 ml), vigorously stirred, and treated by slow addition of 30% H_2O_2 (0.08 ml, 10 mmol), then stirred for a further 1 h. The reaction mixture was then washed with water (3×10 ml), the organic layer was dried over anhydrous $MgSO_4$, and the solvent was removed by evaporation. The products were separated by vacuum distillation. The spectral characteristics of compounds **1**–**5** were in agreement with the literature data.[8–10]

Preparation of 1,5-disubstituted 2-phenyl-6-oxa-2-phosphabicyclo[3.1.0]hexane 2-oxides 6–10 (General method). A solution of 4,5-dialkyl(phenyl,cyclododecyl)-2,3-dihydro-1*H*-phosphole 1-oxide **1**–**5** (8 mmol) in CH_2Cl_2 (16 ml) was treated by the addition of MCPBA (4.14 g, 24 mmol) and refluxed for 24 h. The reaction mixture was neutralized with aqueous $NaHCO_3$ solution (7 g in 35 ml of water) with vigorous stirring for 1 h. The aqueous layer was extracted with $CHCl_3$ and dried over anhydrous $MgSO_4$. The product-containing layer was evaporated at reduced pressure on a rotary evaporator and purified by method of column chromatography (eluent hexane–EtOAc–MeOH, 5:3:1). All epoxides were obtained as 1:2 mixtures of diastereomers (a single asterisk (*) in ^{1}H spectra denote the signal of a single isomer, while two asterisks (**) denote the combined signals of both isomers).

1,5-Diethyl-2-phenyl-6-oxa-2-phosphabicyclo[3.1.0]-hexane 2-oxide (6). Yield 1.38 g (69%), yellow oil. IR spectrum, ν, cm^{-1}: 3058, 2960, 2932, 2602, 2486, 1715, 1466, 1437, 1270, 1168, 1150, 1134, 1070, 839, 745, 706, 505, 484. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.65–1.08 (12H, m, $CH_2C\underline{H}_3$**); 1.51–1.79 (10H, m, $C\underline{H}_2CH_3$ and 3-CH_2**); 1.94–2.21 (4H, m, 3,4-CH_2**); 2.24–2.46 (2H, m, 4-CH_2*); 7.28–7.97 (10H, m, H Ph**). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 13.4, 13.5, 13.7 and 13.8 (CH_3); 21.1 (*J* = 70.7) and 23.0 (*J* = 69.5, C-3); 25.4 (*J* = 12.2) and 26.0 (*J* = 12.2, C-4); 29.8 and 30.4 ($\underline{C}H_2CH_3$); 65.2 (*J* = 96.6) and 65.6 (*J* = 101.6, C-1); 68.6 (*J* = 20.1) and 70.9 (*J* = 15.1, C-5); 128.5 (*J* = 11.6) and 129.0 (*J* = 11.3, C-3,5 Ph); 129.9 (*J* = 10.0, C-2,6 Ph); 129.9 (*J* = 90.0, C-1 Ph); 131.3 (*J* = 9.8, C-2,6 Ph); 132.2 (*J* = 2.4) and 132.3 (*J* = 2.6, C-4 Ph). ^{31}P NMR spectrum, δ, ppm: 53.2; 53.5. Mass spectrum, *m*/*z* (I_{rel}, %): 250 $[M]^+$ (100). Found, %: C 67.15; H 7.66. $C_{14}H_{19}O_2P$. Calculated, %: C 67.19; H 7.65.

2-Phenyl-1,5-dipropyl-6-oxa-2-phosphabicyclo-[3.1.0]hexane 2-oxide (7). Yield 1.64 g (74%), yellow oil. IR spectrum, ν, cm^{-1}: 3062, 2961, 2932, 2873, 2605, 2482, 1771, 1712, 1574, 1465, 1437, 1246, 1177, 1152, 1132, 1109, 750, 730, 705, 502, 482. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.67 (3H, t, ^3J = 7.2, $CH_2C\underline{H}_3$*); 0.81 (3H, t, ^3J = 7.2, $CH_2C\underline{H}_3$*); 0.90–1.00 (6H, m, $CH_2C\underline{H}_3$**); 1.04–1.22 (8H, m, $C\underline{H}_2CH_3$**); 1.47–1.82 (10H, m, $C\underline{H}_2CH_2CH_3$ and 3-CH_2**); 1.93–2.18 (4H, m, 3,4-CH_2**); 2.23–2.46 (2H, m, 4-CH_2*); 7.24–8.01 (10H, m, H Ph**). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 14.0, 14.2, 14.3 and 14.4 (CH_3); 18.5, 18.8, 19.1 and 19.3 ($\underline{C}H_2CH_3$); 21.2 (*J* = 70.7) and 23.1 (*J* = 69.5, C-3); 26.5 and 26.7 ($\underline{C}H_2CH_2CH_3$); 28.0 (*J* = 12.2) and 28.5 (*J* = 12.2, C-4); 32.8 ($\underline{C}H_2CH_2CH_3$); 65.1 (*J* = 97.0) and 65.5 (*J* = 101.3, C-1); 68.3 (*J* = 19.8) and 70.8 (*J* = 14.8, C-5); 128.5 (*J* = 11.7) and 128.9 (*J* = 11.2, C-3,5 Ph); 130.0 (*J* = 9.4, C-2,6 Ph); 131.1 (*J* = 92.5, C-1 Ph); 131.3 (*J* = 9.2, C-2,6 Ph); 132.2 (*J* = 2.4) and 132.3 (*J* = 2.7, C-4 Ph). ^{31}P NMR spectrum, δ, ppm: 53.1; 53.3. Mass spectrum, *m*/*z* (I_{rel}, %): 278 $[M]^+$ (100). Found, %: C 69.08; H 8.37. $C_{16}H_{23}O_2P$. Calculated, %: C 69.05; H 8.33.

1,5-Dibutyl-2-phenyl-6-oxa-2-phosphabicyclo[3.1.0]-hexane 2-oxide (8). Yield 1.86 g (76%), yellow oil. IR spectrum, ν, cm^{-1}: 3056, 2960, 2932, 2872, 2606, 2482, 1708, 1466, 1437, 1264, 1173, 1151, 1110, 838, 813, 740, 704, 507, 484. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.59 (3H, t, ^3J = 6.8, $CH_2C\underline{H}_3$*); 0.72 (3H, t, ^3J = 7.2, $CH_2C\underline{H}_3$*); 0.78–0.92 (6H, m, $CH_2C\underline{H}_3$**); 1.00–1.37 (8H, m, $C\underline{H}_2CH_3$**); 1.40–1.55 (8H, m, $C\underline{H}_2CH_2CH_3$**); 1.57–1.75 (8H, m, $C\underline{H}_2(CH_2)_2CH_3$**); 1.80–2.18 (6H, m, 3,4-CH_2**); 2.27–2.47 (2H, m, 4-CH_2*); 7.35–7.96 (10H, m, H Ph**). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 13.2, 13.3, 13.5 and 13.6 (CH_3); 20.6 (*J* = 70.9) and 22.5 (*J* = 69.4, C-3); 22.4, 22.5 and 22.6 ($\underline{C}H_2CH_3$); 25.1 (*J* = 12.2) and 25.7 (*J* = 11.9, C-4); 26.3, 26.9, 27.3 and 27.5 ($\underline{C}H_2CH_2CH_3$); 30.1 and 30.7 ($\underline{C}H_2(CH_2)_2CH_3$); 64.9 (*J* = 97.2) and 65.2 (*J* = 102.7, C-1); 68.5 (*J* = 20.8) and 70.9 (*J* = 14.8, C-5); 128.3 (*J* = 11.8) and 128.8 (*J* = 11.3, C-3,5 Ph); 131.1 (*J* = 9.3) and 132.1 (*J* = 11.5, C-2,6 Ph); 131.6 (*J* = 90.0, C-1 Ph); 132.2 and 133.9 (C-4 Ph). ^{31}P NMR spectrum, δ, ppm: 52.9; 53.2. Mass spectrum, *m*/*z* (I_{rel}, %): 307.18 $[M+H]^+$ (100). Found, %: C 70.58; H 8.92. $C_{18}H_{27}O_2P$. Calculated, %: C 70.56; H 8.88.

1,2,5-Triphenyl-6-oxa-2-phosphabicyclo[3.1.0]hexane 2-oxide (9). Yield 1.60 g (58%), yellow oil. IR spectrum, ν, cm^{-1}: 3056, 2960, 2927, 2853, 1768, 1713, 1648, 1603, 1567, 1394, 1265, 1200, 1160, 1113, 1072, 1026, 904, 745, 698, 544, 527, 481. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.13–2.38 (4H, m, 3-CH_2**); 2.58–2.96 (2H, m, 4-CH_2*); 3.01–3.16 (2H, m, 4-CH_2*); 6.87–8.06 (30H, m, H Ph**). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 21.2 (*J* = 68.9) and 23.0 (*J* = 69.2, C-3); 28.6 and 29.4 (C-4); 67.8 (*J* = 100.4) and 69.4 (*J* = 96.8, C-1); 70.2 (*J* = 21.1) and 74.0 (*J* = 15.0, C-5); 126.3 and 127.1 (C-4 Ph); 127.5 (C-3,5 Ph); 127.8 (*J* = 11.2, C-3,5 PPh); 128.2 (*J* = 2.4, C-4 PPh); 129.6 (*J* = 90.0, C-1 PPh); 131.0 (*J* = 9.4, C-2,6 PPh); 132.2 (C-1 Ph); 134.4 (*J* = 67.8, C-1 Ph). ^{31}P NMR spectrum, δ, ppm: 50.9; 51.9. Mass spectrum, *m*/*z* (I_{rel}, %): 346 $[M]^+$ (100). Found, %: C 76.34; H 5.55. $C_{22}H_{19}O_2P$. Calculated, %: C 76.29; H 5.53.

1-Phenyldodecahydro-3a,13-epoxycyclododeca[*b*]-phosphole 1-oxide (10). Yield 1.86 g (70%), yellow oil. IR spectrum, ν, cm^{-1}: 3056, 2929, 2860, 1764, 1469, 1438, 1258, 1195, 1183, 1111, 820, 748, 724, 541, 516, 452. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.84–1.76 (45H, m,

4,5,6,7,8,9,10,11,12,13-CH_2 and 2,3-CH_2**); 1.99–2.55 (3H, m, 3-CH_2**); 7.22–7.74 (10H, m, H Ph**). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 21.7 (*J* = 15.5), 21.9, 22.0, 22.1 and 22.9 (C-4,7,8); 23.0 (*J* = 68.6) and 23.1 (*J* = 53.3, C-2); 23.3, 23.5 (*J* = 5.6), 25.1, 25.4, 25.7, 25.9, 26.1 (*J* = 2.0), 26.2, 26.4, 26.8 and 27.0 (C-5,6,9,10,11,12,13); 27.4 (*J* = 17.2, C-3); 65.4 (*J* = 99.1) and 65.9 (*J* = 95.5, C-13a); 69.5 (*J* = 21.5) and 71.7 (*J* = 15.6, C-3a); 128.4 (*J* = 11.6) and 128.7 (*J* = 11.3, C-3,5 Ph); 129.9 (*J* = 9.6, C-2,6 Ph); 130.0 (*J* = 92.3, C-1 Ph); 131.3 (*J* = 9.1, C-2,6 Ph); 132.1 (*J* = 2.4) and 132.2 (*J* = 2.6, C-4 Ph). ^{31}P NMR spectrum, δ, ppm: 51.3; 54.1. Mass spectrum, *m*/*z* (I_{rel}, %): 332 $[M]^+$ (100). Found, %: C 72.39; H 8.84. $C_{20}H_{29}O_2P$. Calculated, %: C 72.26; H 8.79.

Supplementary information file containing ^{1}H, ^{13}C, and ^{31}P spectra of compounds **7**–**10** is available at the journal website at http://link.springer.com/journal/10593.

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