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Electrophilic and Nucleophilic Addition Reactions of α,β-Unsaturated Diphenylphosphoryl Compounds

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Abstract—Procedures were developed for the synthesis of diphenyl(prop-1-en-1-yl)phosphine oxide and cyclohex-1-en-1-yl(diphenyl)phosphine oxide by alkaline hydrolysis of triphenyl(prop-1-en-1-yl)phosphonium bromide and cyclohex-1-en-1-yl(triphenyl)phosphonim bromide, respectively. The bromination of diphenyl(vinyl)phosphine oxide, diphenyl(prop-1-en-1-yl)phosphine oxide, and cyclohex-1-en-1-yl(diphenyl)phosphine oxide with excess bromine gave the corresponding 1,2-dibromo derivatives. Dehydrobromination of 1,2-dibromoethyl(diphenyl)phosphine oxide and 1,2-dibromopropyl(diphenyl)phosphine oxide with sodium hydroxide afforded 1-bromoethenyl(diphenyl)phosphine oxide and 1-bromoprop-1-en-1-yl(diphenyl)phosphine oxide. Addition of bromine and methanol to 1-bromoethenyl(diphenyl)phosphine oxide and 1-bromoprop-1-en-1-yl(diphenyl)phosphine oxide was studied.

Keywords: bromination, dehydrobromination, nucleophilic and electrophilic addition, diphenylphosphoryl compounds, phosphine oxides, radical halogenation.

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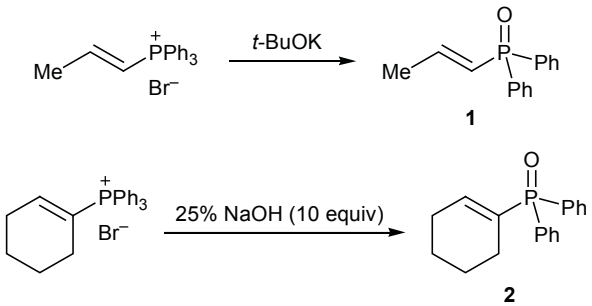
Tertiary phosphine oxides constitute one of the most important classes of phosphorus(V) compounds. They are widely used as complexing agents, extractants for metal salts, monomers, etc. The presence in a phosphine oxide molecule of functional groups capable of coordinating metal ions increases the efficiency and selectivity of complexation [1]. In view of the above stated, in this work we studied reactions of α,β-unsaturated diphenylphosphine oxides with electrophilic and nucleophilic reagents with the goal of obtaining functionally substituted diphenylphosphoryl compounds.

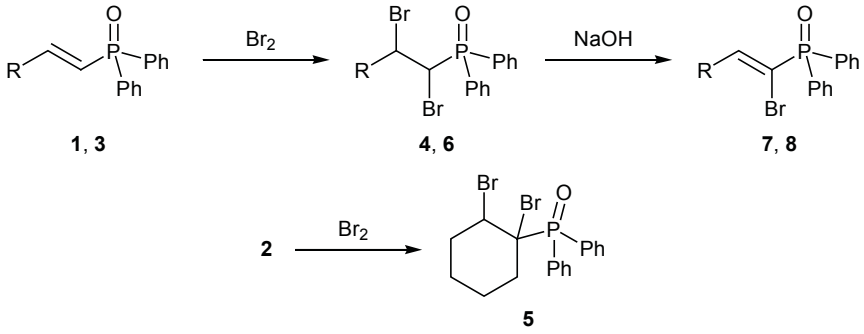
The substrates were diphenyl(prop-1-en-1-yl)phosphine oxide (**1**), cyclohex-1-en-1-yl(diphenyl)phosphine oxide (**2**), and previously reported diphenyl(vinyl)phosphine oxide (**3**) [2]. Unlike the known method [3], we synthesized phosphine oxide **1** by reacting triphenyl(prop-1-en-1-yl)phosphonium bromide with potassium *tert*-butoxide in THF at –5°C; the yield of **1** was slightly higher than 60% (Scheme 1). Phosphine oxide **2** was prepared in a high yield by alkaline hydrolysis of cyclohex-1-en-1-yl(triphenyl)phosphonium bromide with 10 equiv of 25% aqueous sodium hydroxide. Cyclohex-1-en-1-yl(triphenyl)phosphonium bromide was obtained in turn by prototropic isomerization of cyclohex-2-en-1-yl(triphenyl)phosphonium bromide [4].

We previously showed that triphenyl- and tributylphosphonium bromides containing an allyl type substituent on the phosphorus atom are readily brominated with molecular bromine at the β,γ-double bond at –5 to 0°C to give the corresponding 2,3-dibromo derivatives in high yields [5, 6]. On the other hand, it was found that isomeric phosphonium salts with an α,β-unsaturated bond do not add bromine under similar conditions. The observed difference in the behaviors of allyl- and prop-1-en-1-ylphosphonium salts toward electrophiles (such as bromine) is likely to be related to π–*d*

Scheme 1.



Scheme 2.



1, **4**, **8**, R = Me; **3**, **6**, **7**, R = H.

conjugation between the α,β-unsaturated bond and *d* orbital on the phosphorus atom, which induces a partial positive charge on the β-carbon atom and thus hampers electrophilic attack.

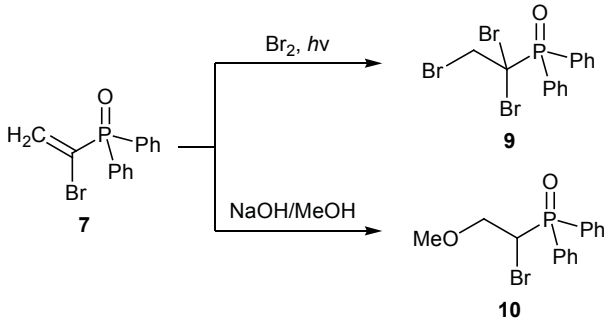
In this work we showed that, unlike α,β-unsaturated phosphonium salts, analogous diphenylphosphoryl compounds, in particular phosphine oxides **1**–**3**, readily react with excess bromine in chloroform at room temperature to give 1,2-dibromopropyl(diphenyl)phosphine oxide (**4**), 1,2-dibromocyclohexyl(diphenyl)-phosphine oxide (**5**), and 1,2-dibromoethyl(diphenyl)-phosphine oxide (**6**) in 87.8, 60.3, and 74% yield, respectively (Scheme 2). When the bromination of **2** was carried out in the presence of a catalytic amount of copper(II) bromide, the yield of **5** increased to 86%. It should be noted that radical bromination of **3** was reported in [7].

Most probably, the different properties of α,β-unsaturated phosphine oxides and phosphonium salts are determined by considerably higher electronegativity of phosphonium group compared to phosphoryl, so that the sp^2-carbon atom in phosphonium salts is deactivated toward electrophilic attack.

Dehydrobromination of dibromoalkylphosphine oxides **4** and **6** with 2 equiv of sodium hydroxide afforded 1-bromoethenyl(diphenyl)phosphine oxide (**7**) and 1-bromoprop-1-en-1-yl(diphenyl)phosphine oxide (**8**), respectively, in high yields (Scheme 2). Unlike compounds **4** and **6**, phosphine oxide **5** failed to undergo dehydrobromination with a number of bases such as triethylamine, sodium carbonate, or sodium hydroxide at room or elevated temperature.

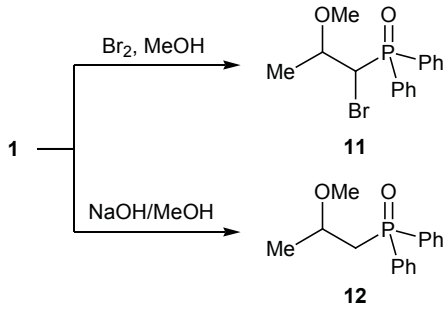
1-Bromo(chloro)vinylphosphine oxides obtained from diphenyl(vinyl)phosphine oxide [7] were reported to readily add nucleophiles [8]. However, the reaction of 1-bromoethenyl(diphenyl)phosphine oxide (**7**) with molecular bromine in chloroform at room temperature produced only 25% of bromination product **9**. When the bromination was carried out under radical initiation conditions, diphenyl(1,1,2-tribromoethyl)phosphine oxide (**9**) was formed in quantitative yield (Scheme 3). Phosphine oxide **7** also reacted with methanol in the presence of sodium hydroxide to give 1-bromo-2-methoxyethyl(diphenyl)phosphine oxide (**10**) in quantitative yield.

Scheme 3.



We also studied reactions of phosphine oxide **1** with bromine in methanol and with methanol in the presence of a base. The reaction of **1** with bromine in methanol afforded conjugate addition product, 1-bromo-2-methoxypropyl(diphenyl)phosphine oxide (**11**), and 2-methoxypropyl(diphenyl)phosphine oxide (**12**) was obtained from **1** and methanol in the presence of sodium hydroxide (Scheme 4).

Scheme 4.



EXPERIMENTAL

The ^1H and ^{31}P NMR spectra were recorded at 303 K on a Varian Mercury-300 spectrometer (USA) at 300.08 and 121.47 MHz, respectively, using DMSO-d_6–CCl_4 (1 : 3) as solvent; the chemical shifts were measured relative to tetramethylsilane (^1H) or 85% H_3PO_4 (^{31}P). Dimethyl sulfoxide-d_6 (isotope purity 99.9%) was purchased from the Isotope Laboratory of the University of Cambridge.

Diphenyl(prop-1-en-1-yl)phosphine oxide (1). Potassium *tert*-butoxide, 3.5 g (31 mmol), was added with stirring under argon to a suspension of 5.96 g (15 mmol) of triphenyl(prop-1-en-1-yl)phosphonium bromide in 30 mL of anhydrous THF, cooled to –5°C. After 3 h, the mixture was adjusted to room temperature, the organic phase was separated by decanting, and the residue was washed with anhydrous THF. The combined organic extracts were evaporated under reduced pressure, and the residue was recrystallized from cyclohexane. Yield 2.35 g (64.7%), white crystals, mp 105–107°C. ^1H NMR spectrum, δ, ppm: 2.01 d.t (3H, Me, J = 6.6, 1.9 Hz), 6.38 d.d.q (1H, CH=CH, J = 24.3, 16.8, 1.7 Hz), 6.63 d.d.q (1H, CH=CH, J = 19.6, 16.8, 6.6 Hz), 7.39–7.54 m (6H, H_{arom}), 7.56–7.71 m (4H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 19.72 d (J_{PC} = 18.4 Hz), 123.9 d (J_{PC} = 102.2 Hz), 127.77 d (J_{PC} = 11.9 Hz), 130.45 d (J_{PC} = 9.7 Hz), 130.7 d (J_{PC} = 2.6 Hz), 131.09 d (J_{PC} = 2.5 Hz), 131.25 d (J_{PC} = 9.6 Hz), 133.75 d (J_{PC} = 102.5 Hz), 145.87 d (J_{PC} = 2.5 Hz). ^{31}P NMR spectrum: δ_P 20.23 ppm. Found, %: C 74.79; H 5.83; P 12.55. $C_{15}H_{15}OP$. Calculated, %: C 74.38; H 6.20; P 12.81.

Cyclohex-1-en-1-yl(diphenyl)phosphine oxide (2). A mixture of 1 g (2.4 mmol) of cyclohex-1-en-1-yl(triphenyl)phosphonium bromide and 3.8 g (24 mmol) of 25% aqueous sodium hydroxide was heated for 30 min on a boiling water bath. The mixture was cooled to room temperature and extracted with benzene. The extract was washed with water and dried over $MgSO_4$, the solvent was distilled off under reduced pressure, and the residue was washed with anhydrous diethyl ether and dried under reduced pressure. Yield 0.58 g (85.7%), white crystals, mp 173–175°C. ^1H NMR spectrum, δ, ppm: 1.60–1.75 m (4H, 4-H, 5-H), 2.02–2.28 m (4H, 3-H, 6-H), 6.29 br.d (1H, 2-H, J_{PH} = 20.1 Hz), 7.40–7.68 m (10H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 20.98, 21.53 d (J_{PC} = 8.3 Hz), 23.9 d (J_{PC} = 9.3 Hz), 25.65 d (J_{PC} = 14.5 Hz), 127.68 d (J_{PC} = 2.5 Hz), 127.82 d (J_{PC} = 4.5 Hz), 127.92 d (J_{PC} = 5.7 Hz), 130.85 d (J_{PC} = 3.2 Hz), 131.07 d (J_{PC} = 2.5 Hz), 131.15 d (J_{PC} = 5.1 Hz), 131.3 d (J_{PC} = 3.2 Hz), 141.34 d (J_{PC} = 8.2 Hz). ^{31}P NMR spectrum: δ_P 27.79 ppm. Found, %: C 76.23; H 7.01; P 10.78. $C_{18}H_{19}OP$. Calculated, %: C 76.59; H 6.74; P 10.99.

1,2-Dibromopropyl(diphenyl)phosphine oxide (4). Bromine, 1 g (6.8 mmol), was added dropwise with stirring at room temperature to a solution of 0.82 g (3.4 mmol) of phosphine oxide **1** in 12 mL of anhydrous chloroform. The mixture was stirred for 3 h and diluted with 20 mL of chloroform, excess bromine was neutralized with a saturated solution of sodium sulfite, and the organic phase was separated, washed in succession with saturated solutions of sodium hydrogen carbonate and sodium chloride, and dried over $MgSO_4$. The solvent was distilled off under reduced pressure, and the residue was washed with anhydrous diethyl ether, and dried under reduced pressure. Yield 1.2 g (87.8%), yellow crystals, mp 135–136°C. ^1H NMR spectrum, δ, ppm: 1.84 d (3H, Me, J = 6.6 Hz), 4.42 q.d.d (1H, C**H**BrMe, J = 4.7, 2.2, 6.6 Hz), 5.54 d.d (1H, PCHBr, J = 2.7, 2.2 Hz), 7.41–7.62 m (6H, Ph), 7.93–8.06 m (4H, Ph). ^{31}P NMR spectrum: δ_P 26.2 ppm. Found, %: C 44.45; H 4.11; Br 39.57; P 8.06. $C_{15}H_{15}Br_2OP$. Calculated, %: C 44.78; H 3.73; Br 39.80; P 7.71.

1,2-Dibromocyclohexyl(diphenyl)phosphine oxide (5). *a*. The reaction of 1.68 g (6 mmol) of phosphine oxide **2** with 1.9 g (12 mmol) of bromine in 20 mL of anhydrous chloroform was carried out as described above for the synthesis of **4**. Yield 1.6 g (60.3%), a mixture of two diastereoisomers with mp 174–178°C. ^1H NMR spectrum, δ, ppm: 1.53–2.89 m (8H, CH_2), 4.37–4.44 m and 4.46–4.54 m (1H, CHBr), 7.38–7.65 m (6H, Ph), 7.83–7.97 m (2H, Ph), 8.14–8.26 m (2H, Ph). ^{31}P NMR spectrum, δ_P, ppm: 30.12, 30.76. Found, %: C 49.15; H 4.58; Br 35.77; P 7.36. $C_{18}H_{19}Br_2OP$. Calculated, %: C 48.87; H 4.29; Br 36.19; P 7.01.

b. The reaction was carried out in a similar way with 0.4 g (1.4 mmol) of **2**, 0.5 g (2.8 mmol) of bromine, and 0.16 g (0.7 mmol) of $CuBr_2$ in 12 mL of anhydrous chloroform. Yield 0.53 g (85.7%), a single diastereoisomer with mp 175–177°C (from benzene). ^1H NMR spectrum, δ, ppm: 1.53–2.89 m (8H, CH_2), 4.47–4.49 m (1H, CHBr), 7.39–7.66 m (6H, Ph), 7.87–7.96 m (2H, Ph), 8.16–8.27 m (2H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 19.06, 19.9 d (J_{PC} = 7.5 Hz), 27.96, 31.5 d (J_{PC} = 5.0 Hz), 51.6, 68.3 d (J_{PC} = 69.4 Hz), 127.27 d (J_{PC} = 11.9 Hz), 127.76 d (J_{PC} = 11.6 Hz), 129.2 d (J_{PC} = 99.3 Hz), 130.98 d (J_{PC} = 2.7 Hz),

131.46 d (J_{PC} = 2.7 Hz), 131.75 d (J_{PC} = 103.4 Hz), 132.13 d (J_{PC} = 7.9 Hz), 132.24 d (J_{PC} = 7.9 Hz). ^{31}P NMR spectrum: δ_P 30.76 ppm. Found, %: C 49.17; H 4.71; Br 35.87; P 7.43. $C_{18}H_{19}Br_2OP$. Calculated, %: C 48.87; H 4.29; Br 36.19; P 7.01.

1,2-Dibromoethyl(diphenyl)phosphine oxide (6) was synthesized in a similar way from 0.3 g (1.3 mmol) of **3** and 0.42 g (2.6 mmol) of bromine in 12 mL of anhydrous chloroform. Yield 0.37 g (74%), mp 150–151°C. ^{1}H NMR spectrum, δ, ppm: 3.64 d.d.d (1H, CH_2Br, J = 11.6, 10.8, 4.8 Hz), 3.91 d.d.d (1H, CH_2Br, J = 11.6, 5.9, 3.0 Hz), 5.43 d.d.d (1H, CHBr, J = 10.8, 3.0, 2.1 Hz), 7.38–7.60 m (6H, Ph), 7.90–8.01 m (4H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 32.24 d (J_{PC} = 5.0 Hz), 46.59 d (J_{PC} = 61.1 Hz), 127.84 d (J_{PC} = 12.4 Hz), 128.29 d (J_{PC} = 11.8 Hz), 130.8 d (J_{PC} = 9.4 Hz), 130.96 d (J_{PC} = 9.4 Hz), 131.55 d (J_{PC} = 2.8 Hz), 131.71 d (J_{PC} = 2.7 Hz), 133.37 d (J_{PC} = 8.8 Hz). ^{31}P NMR spectrum: δ_P 27.74 ppm. Found, %: C 42.98; H 3.16; Br 41.59; P 7.71. $C_{14}H_{13}Br_2OP$. Calculated, %: C 43.29; H 3.35; Br 41.24; P 7.99.

1-Bromoethenyl(diphenyl)phosphine oxide (7). A mixture of 0.25 g (0.64 mmol) of phosphine oxide **6** and 0.052 g (1.3 mmol) of sodium hydroxide in 12 mL of anhydrous benzene was stirred for 3 h at room temperature. The mixture was filtered, and the precipitate was washed with anhydrous benzene (2×10 mL). The filtrate was combined with the washings and dried over $MgSO_4$, the solvent was distilled off under reduced pressure, and the residue was washed with diethyl ether and dried under reduced pressure. Yield 0.14 g (70.2%), white crystals, mp 70–74°C. ^{1}H NMR spectrum, δ, ppm: 6.65 d.d (1H, $=CH_2$, J = 29.7, 2.1 Hz), 6.77 d.d (1H, $=CH_2$, J = 12.4, 2.1 Hz), 7.50–7.65 m (6H, Ph), 7.71–7.79 m (4H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 125.5 d (J_{PC} = 93.3 Hz), 128.0 d (J_{PC} = 12.4 Hz), 129.2 d (J_{PC} = 108.6 Hz), 131.5 d (J_{PC} = 9.8 Hz), 131.6, 131.9 d (J_{PC} = 2.8 Hz), 134.5 d (J_{PC} = 10.5 Hz). ^{31}P NMR spectrum: δ_P 24.74 ppm. Found, %: C 54.48; H 4.26; Br 25.87; P 10.42. $C_{14}H_{12}BrOP$. Calculated, %: C 54.72; H 3.91; Br 26.06; P 10.09.

1-Bromoprop-1-en-1-yl(diphenyl)phosphine oxide (8). A mixture of 0.31 g (0.77 mmol) of phosphine oxide **4** and 0.06 g (1.5 mmol) of sodium hydroxide in 15 mL of anhydrous THF was refluxed for 3 h. The mixture was then treated as described above for the synthesis of **7**. Yield 0.22 g (89%), a hygroscopic mixture of *E* and *Z* isomers at a ratio of 2 : 1. ^{1}H NMR spectrum, δ, ppm: 2.02 d.d (3H, Me, *E*, J = 6.6, 2.5 Hz), 2.16–2.21 m (3H, Me, *Z*), 7.05–7.17 m (1H, =C**H**Me, *E*), 7.19–7.31 m (1H, =C**H**Me, *Z*), 7.4–7.64 m (6H, Ph), 7.71–7.86 m (4H, Ph). ^{31}P NMR spectrum, δ_P, ppm: 24.49, 27.24 (intensity ratio 2 : 1). Found, %: C 56.38; H 4.75; Br 25.17; P 9.94. $C_{15}H_{14}BrOP$. Calculated, %: C 56.07; H 4.36; Br 24.92; P 9.66.

Diphenyl(1,1,2-tribromoethyl)phosphine oxide (9). Bromine, 0.35 g (2.2 mmol), was added dropwise with vigorous stirring to a solution of 0.54 g (1.8 mmol) of phosphine oxide **7** in 15 mL of anhydrous chloroform under irradiation at a power of 500 W. The mixture was refluxed for 6 h, the solvent was removed, and the residue was washed with anhydrous diethyl ether and dried under reduced pressure. Yield 0.76 g (90.4%), hygroscopic material. ^{1}H NMR spectrum, δ, ppm: 4.29 d (2H, CH_2Br, J = 4.3 Hz), 7.49–7.7 m (6H, Ph), 8.14–8.26 m (4H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 43.20 d (J_{PC} = 8.7 Hz), 62.33 d (J_{PC} = 53.6 Hz), 127.25, 127.95 d (J_{PC} = 104.5 Hz), 128.03 d (J_{PC} = 12.2 Hz), 128.64, 132.43 d (J_{PC} = 2.9 Hz), 132.86 d (J_{PC} = 8.6 Hz). ^{31}P NMR spectrum: δ_P 29.37 ppm. Found, %: C 36.31; H 2.86; Br 51.13; P 6.33. $C_{14}H_{12}Br_3OP$. Calculated, %: C 35.97; H 2.57; Br 51.39; P 6.64.

1-Bromo-2-methoxyethyl(diphenyl)phosphine oxide (10). A mixture of 0.3 g (0.98 mmol) of phosphine oxide **7** and 0.04 g (0.98 mmol) of sodium hydroxide in 8 mL of methanol was stirred for 7 h at room temperature. The solvent was removed, the residue was extracted with chloroform, the extract was washed with water, dried over $MgSO_4$, and evaporated under reduced pressure, and the residue was washed with anhydrous diethyl ether and dried under reduced pressure. Yield 0.31 g (93.3%), hygroscopic material. ^{1}H NMR spectrum, δ, ppm: 3.25 s (3H, OMe), 3.67–3.81 m (2H, OCH_2), 5.19–5.25 m (1H, CHBr), 7.42–7.56 m (6H, Ph), 7.88–7.96 m (4H, Ph). ^{13}C NMR spectrum, δ_C, ppm: 44.00 d (J_{PC} = 67.4 Hz), 57.79, 71.2 d (J_{PC} = 4.3 Hz), 127.7 d (J_{PC} = 12.2 Hz), 127.99 d (J_{PC} = 11.7 Hz), 130.71 d (J_{PC} = 9.2 Hz), 130.98 d (J_{PC} = 9.2 Hz), 131.1 d (J_{PC} = 104.6 Hz), 131.19 d (J_{PC} = 2.3 Hz), 131.29 d (J_{PC} = 3.1 Hz). ^{31}P NMR spectrum: δ_P 26.85 ppm. Found, %: C 53.48; H 5.01; Br 23.18; P 8.86. $C_{15}H_{16}BrO_2P$. Calculated, %: C 53.09; H 4.72; Br 23.59; P 9.14.

2-Methoxypropyl(diphenyl)phosphine oxide (11) was synthesized in a similar way by reaction of 0.3 g (1.2 mmol) of phosphine oxide **1** with 0.05 g (1.2 mmol) of sodium hydroxide in 8 mL of methanol. The product was isolated by double reprecipitation. Yield 0.2 g (61%), hygroscopic material. ^{1}H NMR

spectrum, δ, ppm: 1.21 d.d (3H, Me, J = 6.4, 1.0 Hz), 2.3 d.d.d (1H, PCH_2, J = 15.2, 9.1, 5.0 Hz), 2.71 d.d.d (1H, PCH_2, J = 15.2, 12.2, 7.7 Hz), 3.11 s (3H, OMe), 3.52–3.69 m (1H, CH), 7.4–7.51 m (6H, Ph), 7.54–7.69 m (4H, Ph). ^{31}P NMR spectrum: δ_P 26.97 ppm. Found, %: C 70.34; H 6.59; P 11.68. $C_{16}H_{19}O_2P$. Calculated, %: C 70.07; H 6.93; P 11.31.

1-Bromo-2-methoxypropyl(diphenyl)phosphine oxide (12). Bromine, 0.4 g (2.4 mmol), was added to a solution of 0.3 g (1.2 mmol) of phosphine oxide **1** in 16 mL of methanol. The solvent was removed under reduced pressure, the residue was treated with chloroform and water, and the organic phase was separated and dried over $MgSO_4$. The solvent was distilled off under reduced pressure, and the residue was purified by double reprecipitation. Yield 0.23 g (54.3%), hygroscopic material. ^1H NMR spectrum, δ, ppm: 1.27 d (3H, Me, J = 7.1 Hz), 3.19 s (3H, OMe), 3.18–3.49 m (1H, C**H**OMe), 5.22–5.28 m (1H, CHBr), 7.41–7.57 m (6H, Ph), 7.90–8.03 m (4H, Ph). ^{31}P NMR spectrum: δ_P 25.59 ppm. Found, %: C 54.02; H 4.86; Br 23.01; P 8.45. $C_{16}H_{18}BrO_2P$. Calculated, %: C 54.39; H 5.09; Br 22.66; P 8.78.

CONFLICT OF INTEREST

The authors declare the absence of conflict of interest.

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