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ORIGINAL PAPER

New synthesis of trimethylsilyl esters of phosphorus(III) acids

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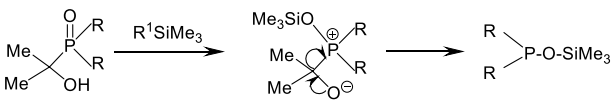
Abstract
A novel synthetically important reaction has been developed for the quick, convenient, and high-yield preparation of trimethylsilyl esters of phosphorus(III) acids, synthetically valuable Michaelis–Arbuzov reaction precursors. Commercially and synthetically available initial compounds used are derivatives of propan-2-ol phosphorylated in the second position capable of long-term storage and available silylating reagents: hexamethyldisilazane, bis(trimethylsilyl)acetamide, and diethyl(trimethylsilyl)amine. Also, a stepwise reaction scheme of the starting compounds with silylating reagents has been proposed.

Graphic abstract



Keywords Phosphorus(III) trimethylsilylether · Phosphorus compounds · Reaction mechanisms · Retro reactions

Introduction

Synthetic methods developed in the field of organophosphorus chemistry are of great importance for modern organic, medical, and pharmaceutical chemistry [1–4]. However, the number of ways to create a phosphorus–carbon bond in organophosphorus chemistry itself remains limited [5, 6]. One of the key reactions in the synthesis of organophosphorus compounds is the Michaelis–Arbuzov reaction. The introduction of trimethylsilyl esters of phosphorus(III) acids into it was shown to lead to better yields of target products in comparison to alkyl analogs. Therefore, trimethylsilyl esters of acids of III-valent phosphorus are popular precursors of the Michaelis–Arbuzov reaction that are used for the synthesis of organophosphorus compounds of various structures [5–9]. However, the number of methods for producing trimethylsilyl esters of III-valent phosphorus acids remains limited by the interaction of hydrophosphoryl compounds with trimethylchlorosilane [10], which requires careful monitoring of the reaction conditions, or interaction with hexamethyldisilazane in harsh conditions [11, 12] or in the presence of catalysts [13]. Therefore, the search for new methods for the synthesis of trimethylsilyl ethers of III-valent phosphorus remains relevant.

Results and discussion

We showed previously [8, 9] that trimethylsilyl diphenylphosphinite (**1a**), a promising precursor of the Michaelis–Arbuzov reaction, could be prepared quickly and in high yield by the reaction of stable (1-hydroxy-1-methylethyl)-diphenylphosphine oxide (**2a**) with silylating reagents: hexamethyldisilazane (HMDS), diethyl(trimethylsilyl)amine (DETS), and bis(trimethylsilyl)acetamide (BSA) in 1,4-dioxane at 100–110 °C. Therefore, it is of practical importance to study the possibility of using this reaction for preparing also synthetically valuable trimethylsilyl phosphinites and trimethylsilyl phosphites by the example of ethyl trimethylsilyl phenylphosphonite (**1b**) and diethyl trimethylsilyl phosphite (**1c**) starting from phosphinate and phosphonate analogs of

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phosphine oxide **2a**: ethyl (1-hydroxy-1-methylethyl)phenylphosphinate (**2b**) and diethyl (1-hydroxy-1-methylethyl)-phosphonate (**2c**).

Initial compounds **2a**–**2c** were prepared in high yields using modified method [14] (Abramov reaction) by the reaction of hydrophosphoryl compounds **3a**–**3c** with acetone in the presence of KF on Al_2O_3 support (KF/Al_2O_3) as a catalyst [15] (Scheme 1).

Since it can be expected that the two reaction products—phosphonite **1b** and phosphite **1c**—are more volatile than phosphinite **1a**, all attempts to synthesize compounds **1a**–**1c**, unlike the initial reaction [8, 9], were performed in the absence of 1,4-dioxane to minimize the loss of the products upon isolation. For this purpose, and since the silylating reagents HMDS, DETS, and BSA were also solvents (in this case the ratio of **2a–c** to them was 1:2), they were being introduced into the reaction at 100–110 °C for 30 min (Scheme 2).

Under these conditions, phosphine oxide **2a** reacts, as expected, with HMDS, DETS, and BSA to produce phosphinite **1a**. The yield of **1a** increases from 79% [8] to 82% (HMDS), from 80% [8] to 89% (DETS) and from 71% [8] to 87% (BSA) when no solvent is used (Scheme 2).

A more complex situation is observed for the reaction of phosphinate **2b** and phosphonate **2c** with the silylating reagents. Expected ethyl trimethylsilyl phenylphosphonite **1b** was obtained in 82% yield only when phosphinate **2b** reacted with DETS. The reaction of phosphinate **2b** with HMDS and BSA led only to ethyl phenylphosphonite **3b** in 82% and 74% yields, respectively (Scheme 2).

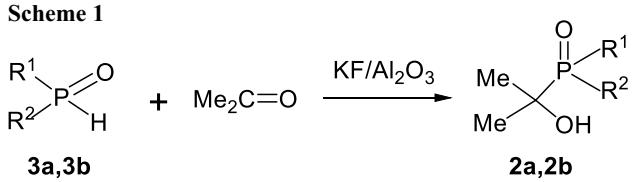
Phosphonate **2c** reacted with HMDS and DETS to give expected diethyl trimethylsilyl phosphite **1c** in 78% and 86% yields, respectively. The reaction of **2c** with BSA under the same conditions afforded a product of silylation at the hydroxy group, diethyl [1-(trimethylsiloxy)-1-methylethyl]-phosphonate (**4c**) in 62% yield (Scheme 2).

The observed reactions of **2a**–**2c** with silylating reagents provided a possibility to suggest a stage-to-stage scheme for the synthesis of compounds **1a**–**1c**:

(1) It is obvious for phosphinate **2b** that the thermal cleavage of phosphorus-carbon bond in the presence of HMDS and BSA (retro Abramov reaction) to form hydrophosphonite (H-phosphinate) **3b** proceeds faster than the silylation of **2b**. The resultant hydrophosphonite **3b** undergoes no further silylation under the reaction conditions. Considering this result as a general feature of all compounds **2a**–**2c**, we can draw a conclusion that hydrophosphoryl compounds **3a**–**3c** (Scheme 2) are not intermediate products in the reaction of compounds **2a**–**2c** with silylating reagents (Scheme 3, route 1). It is also known from the literature that the interaction of hydrophosphoryl compounds with silylating reagents takes place only under fairly harsh conditions or when interacting with hexamethyldisilazane, however, in rather harsh conditions (when heated to 110° C for 2 h in an inert atmosphere [11, 12] or in the presence of catalysts [13]).

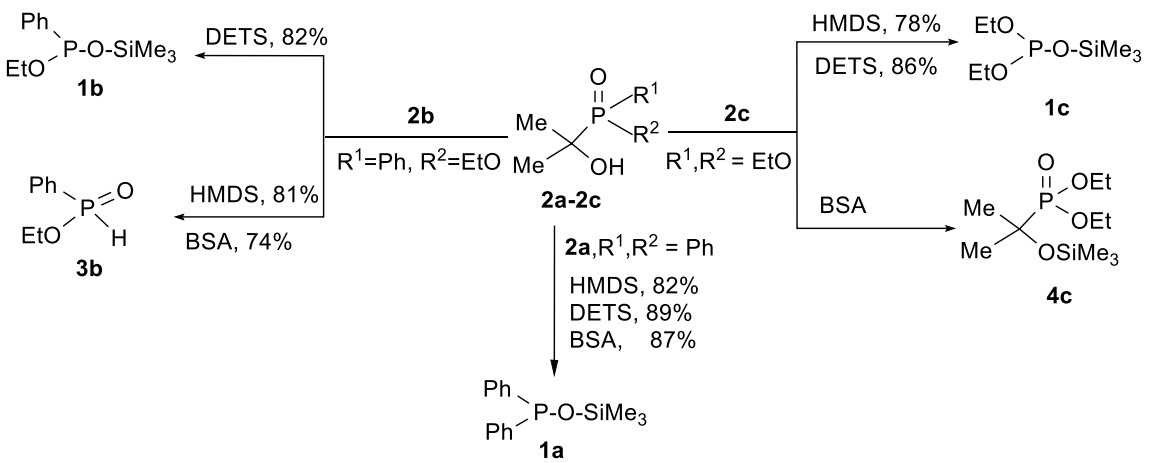
(2) The reaction of phosphonate **2c** with BSA leads to the silylation of the hydroxy group to give diethyl [(1-trimethylsiloxy)-1-methyl]phosphonate **4c**, prob-

Scheme 1

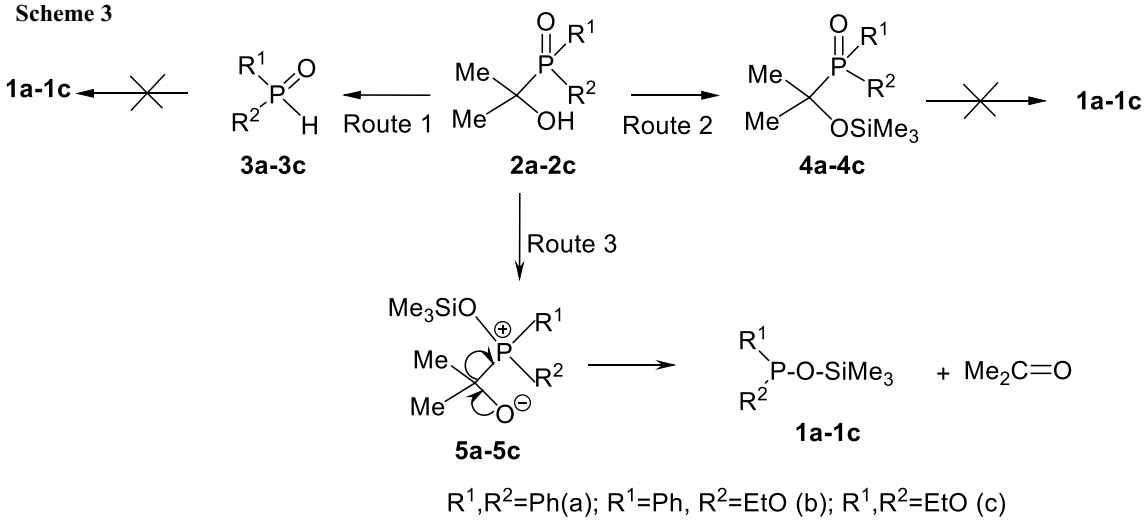


R^1,R^2=Ph(a); R^1=Ph, R^2=EtO (b); R^1,R^2=EtO (c)

Scheme 2



Scheme 3



ably due to reduced nucleophilicity of the phosphoryl group in **2c** comparing to compounds **2a** and **2b**. Resulting silyl ester **4c** remains unchanged under reaction conditions. This fact enables us to conclude that silyl esters **4a**–**4c**, like hydrophosphoryl compounds **3a**–**3c**, are not intermediate products in the synthesis of final compounds **1a**–**1c** from **2a**–**2c** (Scheme 3). To confirm this assumption, we synthesized phosphonate **4c** and its analogs, diphenyl[1-(trimethylsilyloxy)-1-methylethyl]phosphine oxide **4a** and ethyl phenyl[1-(trimethylsiloxy)-1-methylethyl]phosphinate **4b**, by the reaction of silyl esters **1a**–**1c** with acetone in the presence of CuBr (Scheme 4).

Silyl esters **4a** and **4b**, like **4c**, were found to be unchanged under reaction conditions (in the presence of HMDS or BSA) (Scheme 3, route 2). This indicates that, as we supposed earlier [8], the stage-to-stage scheme for the synthesis of final silyl esters **1a**–**1c** begins with silylation of the phosphoryl group of initial compounds **2a**–**2c** to produce zwitterions **5a**–**5c** whose subsequent rearrangement affords final compounds **1a**–**1c** (Scheme 3, route 3).

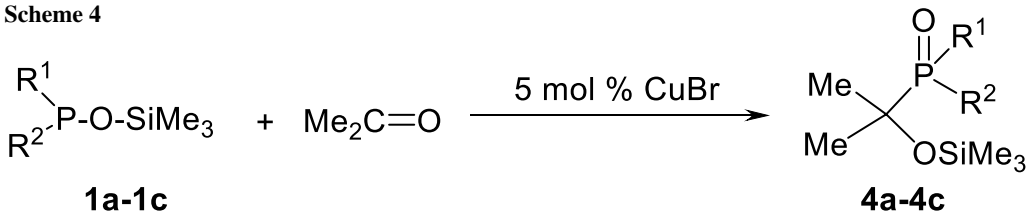
Conclusion

Thus, we have described a novel reaction (Scheme 3, route 3) which at the same time is a new, fast and convenient method for the synthesis of silyl esters of phosphorus(III) acids **1a**–**1c**, reactive precursors of the Michaelis–Arbuzov reaction. Additional advantage of the proposed method is the availability of initial compounds **2a**–**2c** (Scheme 1). This method allows isolation of compounds **2a**–**2c** from the reaction mixture as analytically pure products. All initial compounds **2a**–**2c** can be stored without special precautions for a long time without degradation.

Experimental

^{1}H, ^{31}P, and ^{13}C NMR spectra ($CDCl_3$, 25 °C) were obtained on a Bruker Avance 400 spectrometer operating at 400.13, 161.98, and 100.05 MHz, respectively. $CDCl_3$ was distilled over P_2O_5 and stored over K_2CO_3 at 0 °C in the dark. All syntheses were conducted under an inert atmosphere. Catalyst KF/Al_2O_3 was prepared by the procedure described in the literature [15]. Elemental analysis for C, H, and N

Scheme 4



was performed on a Carlo Erba 1106 automated analyzer. Elemental analysis for P and Si was carried out by spectrophotometry on a Cary 100 Scan instrument.

Diethyl (1-hydroxy-1-methylethyl)phosphonate (2c) Catalyst KF/Al_2O_3 (1 g) was added to a mixture of 5 g of diethyl phosphite (**4c**, 4.65 cm^3, 36.5 mmol) and 5.3 g of acetone (6.76 cm^3, 91.2 mmol) under stirring. An exothermal reaction began after 3–5 min. The reaction mixture temperature was maintained at 20–25 °C using external cooling. After heat evolution ceased, the mixture was stirred at 20 °C for three units. The catalyst was separated by filtration, washed with acetone (2 × 5 cm^3), the solvent was removed from the filtrate at 40 °C and 14 Torr to give 6.6 g (92%) of phosphonate **2c**. B.p.: 133–135 °C at 14 Torr (Ref. [16] 132–134 °C at 14 Torr); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 4.61 (s, 1H, OH), 4.07 (ddd, $^3J_{H,P}$ = 7.08 Hz, $^2J_{H,H}$ = 10.16 Hz, $^3J_{H,H}$ = 7.08 Hz, 4H, 2O-C<u>H</u>$_2$-CH$_3$), 1.38 (d, $^3J_{H,P}$ = 15.56 Hz, 6H, 2CH$_3$), 1.33 (t, $^3J_{H,H}$ = 7.08 Hz, 6H, 2O-CH$_2$-C<u>H</u>$_3$) ppm; ^{31}P{^{1}H} NMR (161.98 MHz, $CDCl_3$): δ = 27.65 s ppm.

Ethyl (1-hydroxy-1-methylethyl)phenylphosphinate (2b) Ethyl (1-hydroxy-1-methylethyl)phenylphosphinate (**2b**) was obtained in a similar manner. Yield 94%; m.p.: 94–96 °C (Ref. [17] 94–96 °C); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 7.81–7.88 (m, 2H, *o*–H, Ph), 7.55–7.52 (m, 1H, *p*–H, Ph), 7.47–7.42 (m, 2H, *m*-H, Ph), 4.16 (ddq, $^3J_{H,P}$ = 7.08 Hz, $^2J_{H,H}$ = 10.20 Hz, $^3J_{H,H}$ = 7.08 Hz, 1H, O-C<u>H</u>$_2$-CH$_3$), 3.97 (ddq, $^3J_{H,P}$ = 7.08 Hz, $^2J_{H,H}$ = 10.16 Hz, $^3J_{H,H}$ = 7.08 Hz, 1H, O-C<u>H</u>$_2$-CH$_3$), 3.51 (s, 1H, OH), 1.39 (d, $^3J_{H,P}$ = 14.20 Hz, 3H, CH$_3$), 1.35 (d, $^3J_{H,P}$ = 14.16 Hz, 3H, CH$_3$), 1.33 (t, $^3J_{H,H}$ = 7.08 Hz, 3H, O-CH$_2$-C<u>H</u>$_3$) ppm; ^{31}P{^{1}H} NMR (161.98 MHz, $CDCl_3$): δ = 43.45 s ppm.

(1-Hydroxy-1-methylethyl)diphenylphosphine oxide (2a) (1-Hydroxy-1-methylethyl)diphenylphosphine oxide (**2a**) was conducted in a similar manner, 92% yield of **2a** as colorless needles. M.p.: 136–138 °C (Ref. [18] 137–139 °C); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 8.02–7.96 (m, 4H, o–H, Ph), 7.51–7.47 (m, 2H, m-H, Ph), 7.44–7.40 (m, 4H, p–H, Ph), 3.61 (bs, 1H, OH), 1.40 (d, $^3J_{P,H}$ = 13.56 Hz, 6H, 2CH$_3$) ppm; ^{31}P{^{1}H} NMR (161.98 MHz, $CDCl_3$): δ = 34.59 s ppm.

Reaction of (1-hydroxy-1-methylethyl)diphenylphosphine oxide (2a) with HMDS HMDS (6.13 g, 38.2 mmol) was added to 5.0 g of phosphine oxide **2a** (19.2 mmol) and the mixture was heated at 110 °C. Phosphine oxide **2a** completely dissolved after 10 min. The mixture was heated for additional 20 min, cooled to 25 °C, excess HMDS was removed at 14 Torr. The residue was distilled twice under reduced pressure to give 4.31 g (82%) of trimethylsilyl diphenylphosphinite (**1a**).

The reaction of phosphine oxide **2a** with DETS (87% yield of phosphinite **1a**) and BSA (89% yield of phosphinite **1a**) was performed in a similar manner. All obtained samples of trimethylsilyl diphenylphosphinite **1a** were combined. Colorless needles; m.p.: 24–26 °C (Ref. [8, 9] 24–26 °C); b.p.: 110–112 °C at 1 Torr (Ref. [8, 9] 110–112 °C at 1 Torr); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 7.56–7.51 (m, 4H, *o*–H, Ph), 7.38–7.30 (m, 6H, *m,p*–H, Ph), 0.27 (s, 9H, Si(CH$_3$)$_3$) ppm; ^{31}P{^{1}H} NMR (161.98 MHz, $CDCl_3$): δ = 94.90 s ppm [8].

The following reactions were performed in similar manner:

Reaction of ethyl (1-hydroxy-1-methylethyl)phenylphosphinate (2b) with DETS The yield of phosphonite **1b** is 82%; b.p.: 85–87 °C at 1 Torr (Ref. [19] 53 °C at 0.05 Torr); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 7.59–7.56 (m, 2H, *o*–H, Ph), 7.43–7.38 (m, 3H, *m,p*–H, Ph), 4.83 (ddq, $^3J_{H,P}$ = 7.32 Hz, $^2J_{H,H}$ = 10.16 Hz, $^3J_{H,H}$ = 7.00 Hz, 1H, O-C<u>H</u>$_2$-CH$_3$), 3.97 (ddq, $^3J_{H,P}$ = 7.32 Hz, $^2J_{H,H}$ = 10.16 Hz, $^3J_{H,H}$ = 7.00 Hz, 1H, O-C<u>H</u>$_2$-CH$_3$), 1.21 (t, $^3J_{H,H}$ = 7.00 Hz, 3H, O-CH$_2$-C<u>H</u>$_3$), 0.28 (s, 9H, Si(CH$_3$)$_3$) ppm; ^{31}P{^{1}H} NMR (161.98 MHz, $CDCl_3$): δ = 145.32 s ppm [19].

Reaction of phosphinate 2b with HMDS and BSA The yield of ethyl phenylphosphonite **3b** is 81% (HMDS) and 74% (BSA). All obtained samples of ethyl phenylphosphinate **3b** were combined. B.p.: 96–98 °C at 1 Torr (Ref. [20] 94–99 °C at 1 Torr); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 7.71–7.65 (m, 2H, *o*–H, Ph), 7.51–7.47 (m, 1H, *m*-H, Ph), 7.43–7.38 (m, 2H, *p*–H, Ph), 7.49 (d, $^1J_{P,H}$ = 563 Hz, 1H, P–<u>H</u>), 4.12–3.97 (m, 2H, O-C<u>H</u>$_2$-CH$_3$,), 1.27 (t, 3H, O-CH$_2$-C<u>H</u>$_3$, $^3J_{H,H}$ = 7.06 Hz) ppm [21]; ^{31}P{^{1}H} NMR (161.98 MHz, $CDCl_3$): δ = 24.55 s ppm (Ref. [22] 23.5 ppm).

Reaction of diethyl (1-hydroxy-1-methylethyl)phosphonate (2c) with HMDS and DETS The yield of diethyl trimethylsilyl phosphite **3c** is 78% (HMDS) and 86% (DETS). B.p.: 62–65 °C at 14 Torr (Ref. [23] 61–63 °C at 14 Torr); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 3.88–3.75 (m, 4H, 2CH$_2$), 1.26 (t, $^3J_{H,H}$ = 7.04 Hz, 6H, 2C<u>H</u>$_3$-CH$_2$), 0.24 (s, 9H, Si(CH$_3$)$_3$) ppm; ^{31}P{^{1}H} NMR (161.98 MHz, $CDCl_3$): δ = 127.51 s ppm.

Reaction of diethyl (1-hydroxy-1-methylethyl)phosphonate (2c) with BSA The yield of diethyl [1-(trimethylsiloxy)-1-methylethyl]phosphonate (**4c**) is 62%. B.p.: 95–98 °C at 1 Torr (Ref. [24] 70–72 °C at 0.07 Torr); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 4.10 (dq, $^3J_{H,P}$ = 7.08 Hz, $^3J_{H,H}$ = 7.08 Hz, 4H, 2O-C<u>H</u>$_2$-CH$_3$), 1.42 (d, $^3J_{H,P}$ = 15.60 Hz, 6H, 2CH$_3$), 1.27 (t, $^3J_{H,H}$ = 7.10 Hz, 6H, 2O-CH$_2$-C<u>H</u>$_3$), 0.12 (s, 9H, Si(CH$_3$)$_3$) ppm; ^{31}P{^{1}H} NMR (161.98 MHz, $CDCl_3$): δ = 25.24 s ppm [20].

Synthesis of diethyl [1-(trimethylsiloxy)-1-methylethyl]-phosphonate (4c) Copper(I) bromide (0.13 g, 0.9 mmol, 5 mol %) was added with stirring to a solution of 3.2 g of diethyl trimethylsilyl phosphite (**1c**, 18.2 mmol) in 4.2 g of acetone (5.4 cm^3, 72.4 mmol). After 15–20 min, the temperature of the reaction mixture increased to 30–35 °C and 20–30 min later became 20 °C. The mixture was stirred for additional 3 h at 20 °C, the excess acetone was removed, 20 cm^3 of CH_2Cl_2 was added, the mixture was washed with 5% aqueous NH_3 (2×5 cm^3) and 5 cm^3 of water. The organic layer was separated and dried with Na_2SO_4. The drying agent was separated by filtration, washed with 2×5 cm^3 of CH_2Cl_2, the combined organic layer was concentrated at 14 Torr, and the residue was purified by distillation to give 2.98 g (68%) of phosphonate **4c**. B.p., ^{1}H NMR, and $^{31}P\{^1H\}$ NMR of compound **4c** synthesized by this method coincide with those of **4c**, synthesized by reaction compound **2c** with BSA.

The following compounds were obtained in a similar manner:

Diphenyl[1-trimethylsiloxy)-1-methylethyl]phosphine oxide(4a) Yield 81%; m.p.: 89–91 °C (from heptane); ^{1}H NMR (400.13 MHz, $CDCl_3$): $\delta = 8.02–7.97$ (m, 4H, *o*–H, Ph), 7.44–7.34 (m, 6H, *m,p*–H, Ph), 1.42 (d, $^3J_{H,P} = 14.00$ Hz, 6H, $2CH_3$), 0.00 (s, 9H, $Si(CH_3)_3$) ppm; $^{31}P\{^1H\}$ NMR (161.98 MHz, $CDCl_3$): $\delta = 31.76$ s ppm; ^{13}C NMR (100.05 MHz, $CDCl_3$): $\delta = 132.58$ (d, $^2J_{C,P} = 7.3$ Hz, *o*-C, Ph), 131.49 (d, $^4J_{C,P} = 2.9$ Hz, *p*–C, Ph), 130.45 (d, $^1J_{C,P} = 91.9$ Hz, *ipso*-C, Ph), 127.99 (d, $^3J_{C,P} = 10.9$ Hz, *m*-C, Ph), 75.42 (d, $^1J_{C,P} = 97.0$ Hz, P-C^1), 25.10 (d, $^3J_{C,P} = 5.11$ Hz, C^1-$\underline{C}H_3$), 2.34 (s, $Si(CH_3)_3$) ppm.

Ethyl [1-(trimethylsilyloxy)-1-methylethyl]phenylphosphinate (4b). Yield 83%; b.p.: 167–170 °C at 1 Torr; ^{1}H NMR (400.13 MHz, $CDCl_3$): $\delta = 7.78–7.73$ (m, 2H, *o*–H, Ph), 7.49–7.45 (m, 1H, *p*–H, Ph), 7.40–7.36 (m, 2H, *m*-H, Ph), 4.01 (ddq, 1H, O-C$\underline{H}_2$-CH_3, $^3J_{H,P} = 7.00$ Hz, $^2J_{H,H} = 10.08$ Hz, $^3J_{H,H} = 7.02$ Hz), 3.97 (ddq, $^3J_{H,P} = 7.00$ Hz, $^2J_{H,H} = 10.08$ Hz, $^3J_{H,H} = 7.02$ Hz, 1H, O-C$\underline{H}_2$-CH_3), 1.48 (d, $^3J_{H,P} = 13.80$ Hz, 3H, CH_3), 1.31 (d, $^3J_{H,P} = 15.64$ Hz, 3H, CH_3), 1.28 (t, $^3J_{H,H} = 7.02$ Hz, 3H, O-CH_2-C$\underline{H}_3$), -0.02 (s, 9H, $Si(CH_3)_3$) ppm; $^{31}P\{^1H\}$ NMR (161.98 MHz, $CDCl_3$): $\delta = 42.31$ s ppm; ^{13}C NMR (100.05 MHz, $CDCl_3$): $\delta = 133{,}62$ (d, $^2J_{C,P} = 8.75$ Hz, *o*-C, Ph), 132.05 (d, $^4J_{C,P} = 2.92$ Hz, *p*–C, Ph), 128.01 (d, $^3J_{C,P} = 116.72$ Hz, *ipso*-C, Ph), 127.86 (d, $^3J_{C,P} = 11.68$ Hz, *m*-C, Ph), 73.78 (d, $^1J_{C,P} = 127.66$ Hz, P-**C**^{1}), 60.99 (d, $^3J_{C,P} = 7.29$ Hz, O-$\underline{C}H_2$-CH_3), 25.31 (d, $^3J_{C,P} = 7.29$ Hz, C^1-$\underline{C}H_3$), 24.64 (d, $^3J_{C,P} = 3.64$ Hz, C^1-$\underline{C}H_3$), 16.56 (d, $^3J_{C,P} = 5.83$ Hz, O-CH_2-$\underline{C}H_3$), 2.40 (s, $Si(CH_3)_3$) ppm.

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