TMSCl-Promoted Addition of Diethyl Phosphite to an Imine for the Synthesis of Bis[1-diethoxyphosphorylalkyl]amines

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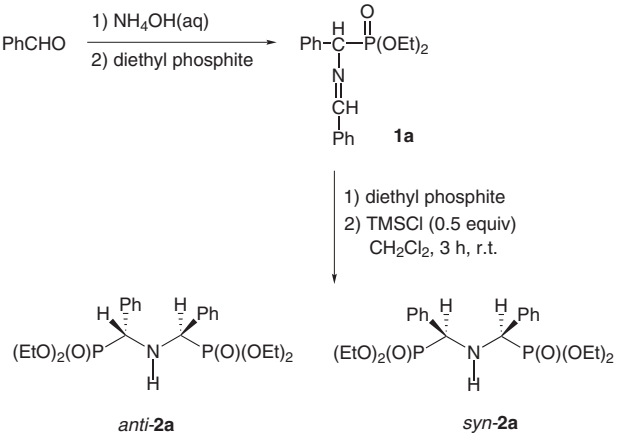
Abstract: A convenient preparative approach for the synthesis of bis[1-diethoxyphosphorylalkyl]amines has been developed. Treatment of aromatic aldehydes with ammonia and reaction with diethyl phosphite gives diethyl aryl[(arylmethylidene)amino]methylphosphonates, which can be easily reacted with diethyl phosphite in the presence of chlorotrimethylsilane to give bis[1-diethoxyphosphorylalkyl]amines. This method is easy, rapid, and good-yielding for the synthesis of bis[1-diethoxyphosphorylalkyl]amines from simple starting materials.

Key words: TMSCl, diethyl phosphite, imines, bis[1-diethoxyphosphorylalkyl]amines, addition reaction

Organophosphorus compounds have found a wide range of applications in the areas of industrial, agricultural, and medicinal chemistry owing to their biological and physical properties as well as their use as synthetic intermediates.[1] α-Functionalized phosphonic acids are valuable intermediates for the preparation of medicinal compounds and synthetic intermediates.[2–4] Among the α-functional phosphonic acids, 1-aminophosphonic acids are an important class of compounds that exhibit a variety of interesting and useful properties. 1-Aminophosphonic acids are important substitutes for the corresponding α-amino acids in biological systems.[5] Indeed a number of potent antibiotics,[6] enzyme inhibitors,[7] and pharmacological agents[8] are 1-aminophosphonic acids or peptide analogues. Aminophosphonic acids are also found as constituents of natural products.[9] In contrast to the widely studied 1-aminophosphonic acids derivatives,[10–13] relatively few papers have reported on the chemistry of bis[1-diethoxyphosphorylalkyl]amines as an important class of 1-aminophosphonic acid derivatives. Bis[1-dialkoxyphosphorylalkyl]amines have been used as chelating agents for polyvalent, particularly for alkaline earth metal ions.[14]

Many effective methods for the preparation of 1-aminophosphonic acids have been developed, but, to the best of our knowledge, few synthetic routes to bis[1-diethoxyphosphorylalkyl]amines have been reported. These methods involve prolonged heating of primary amines with chloromethylphosphonic acids in alkaline solution[15]and a Mannich-type reaction of an amine, formaldehyde, and phosphorous acid is also reported.[16] Recently, a new method for the preparation of bis[1-diethoxyphosphorylalkyl] amines in the reaction of 1-iminocarboxylate salts with dialkyl chlorophosphites has been reported.[17] However, these methods have drawbacks, including harsh reaction conditions, long reaction times, and side reactions. On the other hand, prolonged heating of chloromethylphosphonic acids in alkaline solution also leads to the competitive hydrolysis product of the chlorine–carbon bond to give hydroxymethylphosphonic acid.[15] As a part of our efforts to introduce novel methods for the synthesis of organophosphorus compounds,[18] we present here a new method for the synthesis of bis[1-diethoxyphosphorylalkyl]amines is described. Recently, we reported that reaction of aromatic aldehydes with ammonia solution followed by reaction with diethyl phosphite, gave diethyl aryl[(arylmethylidene)amino]methylphosphonates in good yields that can be easily hydrolyzed to diethyl 1-aminoarylmethylphosphonates.[19] We have now found that the reaction of diethyl phenyl[(phenylmethylidene)amino]methylphosphonate (**1a**), as a model compound, with diethyl phosphite in the presence of chlorotrimethylsilane (0.5 equiv) gives bis[1-diethoxyphosphorylphenylmethyl]amine (**2a**) as sole product (Scheme 1). The reaction can be carried out in 0.2 equivalent of chlorotrimethylsilane involving a long reaction period (12 h). The reaction failed even after 48 hours when carried out without any catalyst. Use of other Lewis acid catalysts such as $AlCl_3$, $FeCl_3$, $TiCl_4$, $LiClO_4$ and $ZnCl_2$ did not give any product.

The ^{31}P NMR spectrum of **2a** exhibited two peaks at δ = 22.78 and 23.08 due to diastereoisomers. An excellent diastereoselection can be obtained in this reaction (93:7).



Scheme 1

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Table 1 Reaction of **1** with Diethyl Phosphite Using Chlorotrimethylsilane as Catalyst

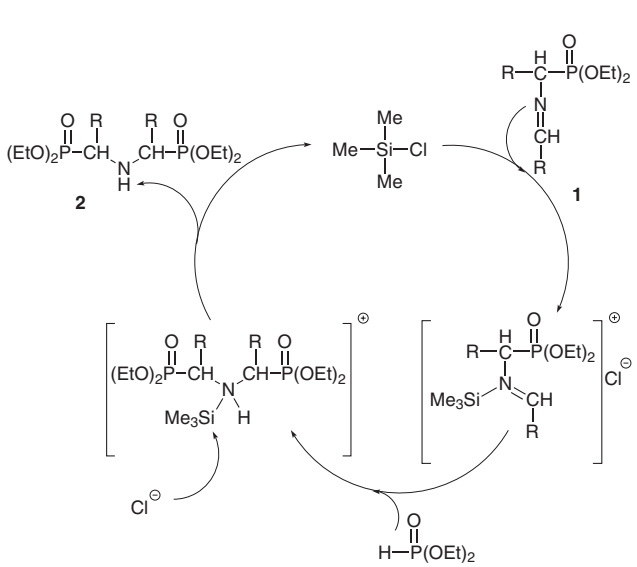
Entry	R	Time (h)	Yield of **2** (%)[a]	Diastereoselection (%)[b]	^{31}P NMR, δ	
					Major	Minor
a	Ph	3	78	93: 7	22.78	23.08
b	4-MeOCHC$_6$H$_4$	5	80	81:19	23.21	23.44
c	4-ClC$_6$H$_4$	2	65	94: 6	22.03	22.40
d	4-BrC$_6$H$_4$	2	73	84:16	21.83	22.20
e	4-FC$_6$H$_4$	2	76	88:12	22.36	22.68
f	4-MeC$_6$H$_4$	5	65	73:27	23.21	23.48
g	3-MeOCHC$_6$H$_4$	3	73	84:16	22.78	22.99
h	3-BrC$_6$H$_4$	2	70	87:13	21.52	21.98
i	3-MeC$_6$H$_4$	5	70	79:21	22.93	23.22
j	3-FC$_6$H$_4$	3	73	89:11	21.71	22.17
k	β-Naphthyl	5	63	89:11	22.75	23.09

[a] Isolated yield.
[b] Diastereoselection was calculated from ^{31}P NMR spectra.

Due to the presence of two stereogenic carbons bonded to the nitrogen atom, this compound exists as two diastereomeric forms: one meso (*syn*) and one racemic pair (*anti*).

This process was successfully applied to other diethyl aryl[(arylmethylidene)amino]methylphosphonates **1b**–**k** as summarized in Table 1. Methylphosphonates **1b**–**k**[19] react with diethyl phosphite in the presence of chlorotrimethylsilane to afford the desired products **2b**–**k** in good yields. The mechanism of formation of **2** is illustrated in Scheme 2. Reaction between ammonium hydroxide and aliphatic aldehydes failed with diethyl phosphite and did not give any imines **1**.

In summary, fast reaction rates, mild reaction conditions, good yields, simple work-up, and relatively clean reactions with no tar formation make this method an attractive and useful contribution to present methodologies. Indeed, a wide range of imines **1** was converted into the corresponding bis[1-diethoxyphosphorylalkyl]amines using this method. Further investigations on this reaction are now in progress.



Scheme 2

All chemicals were commercial products and distilled or recrystallized before use. All melting points were obtained on a Büchi 510 melting point apparatus and are uncorrected. The IR spectra were determined using a FT-IR Bruker-Vector 22 spectrophotometer. NMR spectra were taken with a 250 Bruker Avance instrument with the chemical shifts being reported as δ in ppm and coupling constants expressed in Hz. Silica gel column chromatography was carried out with silica gel 100 (Merck No. 10184). Merck silica gel 60 F254 plates (No. 5744) were used for the preparative TLC.

Imines 1; General Procedure

The aldehyde (15 mmol) was added to NH_4OH (30%, 15 mL) and the solution was stirred for 5 h at reflux. During this time, a white precipitate formed. The precipitate was removed by filtration and dried. Diethyl phosphite (828 mg, 6 mmol) was added to this solid and the resulting solution was stirred for 2–5 h at 70 °C. Chromatography on silica gel with EtOAc–*n*-hexane (5:5) gave the pure products **1** as oils.[19]

Bis[1-diethoxyphosphorylalkyl]amines 2; General Procedure

Chlorotrimethylsilane (217 mg, 2 mmol) was added to compound **1** (4 mmol) in CH_2Cl_2 (20 mL) and the mixture was stirred for 2–5 h at r.t. The solvent was evaporated and the residue was chromatographed on a plug of silica gel using EtOAc–MeOH (9:1) as eluent. Evaporation of the solvent under reduced pressure gave the pure product as colorless oil in 63–80% yields. All products gave satisfactory spectral data in accord with the assigned structures.

Bis(1-diethoxyphosphorylphenylmethyl)amine (2a)

Colorless oil; yield: 78%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.10 (6 H, t, *J* = 7.1 Hz), 1.26 (6 H, t, *J* = 7.1 Hz), 2.93 (1 H, br, NH), 3.65–4.35 (10 H, m), 7.29 (10 H, s).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 134.3, 128.8, 128.2, 128.1, 62.6–62.9 (O*C*H_2CH$_3$), 57.4 (dd, $J_{P,C}$ = 155.1, 17.9 Hz), 16.1–16.3 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 22.78, 23.08 (93:7).

Anal. Calcd for $C_{22}H_{33}NO_6P_2$: C, 56.29; H, 7.08; N, 2.98. Found: C, 56.20; H, 7.10; N, 2.78.

Bis[1-diethoxyphosphoryl(4-methoxyphenyl)methyl]amine (2b)
Colorless oil; yield: 80%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.14 (6 H, t, *J* = 7.1 Hz), 1.29 (6 H, t, *J* = 7.1 Hz), 1.69 (1 H, br, NH), 3.79 (3 H, s), 3.68–4.35 (10 H, m), 6.87 (4 H, d, *J* = 8.5 Hz), 7.22 (4 H, d, *J* = 8.5 Hz).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 159.4, 129.9, 125.9, 113.8, 62.4–62.8 (O*C*H_2CH$_3$), 56.4 (dd, $J_{P,C}$ = 156.7, 17.9 Hz), 55.1, 16.1–16.3 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 23.21, 23.44 (81:19).

Anal. Calcd for $C_{24}H_{37}NO_8P_2$: C, 54.44; H, 6.99; N, 2.65. Found: C, 54.32; H, 7.08; N, 2.73.

Bis[1-diethoxyphosphoryl(4-chlorophenyl)methyl]amine (2c)
Colorless oil; yield: 65%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.15 (6 H, t, *J* = 7.1 Hz), 1.29 (6 H, t, *J* = 7.1 Hz), 2.19 (1 H, br, NH), 3.65–4.35 (10 H, m), 7.23 (4 H, d, *J* = 8.25 Hz), 7.33 (4 H, d, *J* = 8.25 Hz).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 134.1, 132.9, 130.1, 128.9, 62.9–63.2 (O*C*H_2CH$_3$), 56.9 (dd, $J_{P,C}$ = 155.4, 17.6 Hz), 16.3–16.4 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 22.03, 22.40 (94:6).

Anal. Calcd for $C_{22}H_{31}Cl_2NO_6P_2$: C, 52.17; H, 6.12; N, 2.76. Found: C, 52.30; H, 5.98; N, 2.65.

Bis[1-diethoxyphosphoryl(4-bromophenyl)methyl]amine (2d)
Colorless oil; yield: 73%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.15 (6 H, t, *J* = 7.1 Hz), 1.29 (6 H, t, *J* = 7.1 Hz), 2.45 (1 H, br, NH), 3.72 (2 H, d, $J_{P,C}$ = 22 Hz), 3.75–4.25 (8 H, m), 7.17 (4 H, dd, *J* = 8.25, 1.5 Hz), 7.47 (4 H, d, *J* = 8.25 Hz).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 133.5, 131.8, 130.5, 122.3, 62.9–63.2 (O*C*H_2CH$_3$), 56.9 (dd, $J_{P,C}$ = 155.4, 17.6 Hz), 16.2–16.4 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 21.83, 22.20 (84:16).

Anal. Calcd for $C_{22}H_{31}Br_2NO_6P_2$: C, 44.38; H, 5.21; N, 2.35. Found: C, 44.40; H, 5.32; N, 2.25.

Bis[1-diethoxyphosphoryl(4-fluorophenyl)methyl]amine (2e)
Colorless oil; yield: 76%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.04 (6 H, t, *J* = 7.0 Hz), 1.18 (6 H, t, *J* = 7.0 Hz), 2.82 (1 H, br, NH), 3.57–4.22 (10 H, m), 6.80–6.97 (4 H, m), 7.10–7.22 (4 H, m).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 162.0 (d, $J_{C,F}$ = 246.5 Hz), 130.4, 115.7, 115.3, 62.6–63.0 (O*C*H_2CH$_3$), 56.6 (dd, $J_{P,C}$ = 156.6, 17.6 Hz), 16.1–16.3 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 22.36, 22.68 (88:12).

Anal. Calcd for $C_{22}H_{31}F_2NO_6P_2$: C, 55.81; H, 6.55; N, 2.96. Found: C, 55.72; H, 6.50; N, 3.15.

Bis[1-diethoxyphosphoryl(4-methylphenyl)methyl]amine (2f)
Colorless oil; yield: 65%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.04 (6 H, t, *J* = 7.1 Hz), 1.19 (6 H, t, *J* = 7.1 Hz), 2.24 (3 H, s), 2.85 (1 H, br, NH), 3.60–4.25 (10 H, m), 6.95–7.15 (8 H, m).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 137.7, 131.1, 129.2, 128.7, 62.4–62.9 (O*C*H_2CH$_3$), 57.0 (dd, $J_{P,C}$ = 156.0, 18.2 Hz), 21.1, 16.1–16.3 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 23.21, 23.48 (73:27).

Anal. Calcd for $C_{24}H_{37}NO_6P_2$: C, 61.93; H, 7.95; N, 3.01. Found: C, 61.75; H, 7.79; N, 2.92.

Bis[1-Diethoxyphosphoryl(3-methoxyphenyl)methyl]amine (2g)
Colorless oil; yield: 73%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.08 (6 H, t, *J* = 7.1 Hz), 1.22 (6 H, t, *J* = 7.1 Hz), 2.78 (1 H, br, NH), 3.72 (3 H, s), 3.64–4.23 (10 H, m), 6.70–6.87 (4 H, m), 7.08–7.21 (4 H, m).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 159.7, 135.9, 129.9, 125.4, 114.0, 62.7–63.0 (O*C*H_2CH$_3$), 57.3 (dd, $J_{P,C}$ = 155.0, 17.9 Hz), 55.1, 16.1–16.4 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 22.78, 22.99 (84:16).

Anal. Calcd for $C_{24}H_{37}NO_8P_2$: C, 54.44; H, 6.99; N, 2.65. Found: C, 54.60; H, 7.10; N, 2.81.

Bis[1-diethoxyphosphoryl(3-bromophenyl)methyl]amine (2h)
Colorless oil; yield: 70%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.01–1.29 (12 H, m), 1.52 (1 H, br, NH), 3.55–4.20 (10 H, m), 7.06–7.48 (10 H, s).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 136.8, 131.5, 131.4, 130.1, 127.5, 122.7, 62.9–63.3 (O*C*H_2CH$_3$), 57.1 (dd, $J_{P,C}$ = 155.1, 17.9 Hz), 16.2–16.3 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 21.52, 21.98 (87:13).

Anal. Calcd for $C_{22}H_{31}Br_2NO_6P_2$: C, 44.38; H, 5.21; N, 2.35. Found: C, 44.51; H, 5.35; N, 2.48.

Bis[1-diethoxyphosphoryl(3-methylphenyl)methyl]amine (2i)
Colorless oil; yield: 70%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.00 (6 H, t, *J* = 7.0 Hz), 1.17 (6 H, t, *J* = 7.0 Hz), 2.21 (3 H, s), 2.82 (1 H, br, NH), 3.63–4.20 (10 H, m), 6.97–7.13 (8 H, m).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 137.9, 134.2, 129.2, 128.8, 128.2, 126.0, 62.2–62.8 (O*C*H_2CH$_3$), 57.3 (dd, $J_{P,C}$ = 154.9, 17.9 Hz), 21.3, 16.0–16.3 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 22.93, 23.22 (79:21).

Anal. Calcd for $C_{24}H_{37}NO_6P_2$: C, 61.93; H, 7.95; N, 3.01. Found: C, 61.82; H, 7.83; N, 2.89.

Bis[1-diethoxyphosphoryl(3-fluorophenyl)methyl]amine (2j)
Colorless oil; yield: 73%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.11 (6 H, t, *J* = 7.0 Hz), 1.24 (6 H, t, *J* = 7.0 Hz), 2.92 (1 H, br, NH), 3.65–4.25 (10 H, m), 6.85–7.09 (6 H, m), 7.15–7.32 (2 H, m).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 162.9 (d, $J_{C,F}$ = 246.8 Hz), 137.1, 130.1, 124.5, 115.7, 115.3, 62.9–63.2 (O*C*H_2CH$_3$), 57.2 (dd, $J_{P,C}$ = 155.1, 17.4 Hz), 16.1–16.3 (OCH$_2$*C*H$_3$).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 21.71, 22.17 (89:11).

Anal. Calcd for $C_{22}H_{31}F_2NO_6P_2$: C, 55.81; H, 6.55; N, 2.96. Found: C, 55.68; H, 6.42; N, 3.10.

Bis[1-diethoxyphosphoryl(β-naphthyl)methyl]amine (2k)
Colorless oil; yield: 63%.

^{1}H NMR ($CDCl_3$/TMS, 250 MHz): δ = 1.10 (6 H, t, *J* = 7.0 Hz), 1.31 (6 H, t, *J* = 7.0 Hz), 3.03 (1 H, br, NH), 3.70–4.57 (10 H, m), 7.35–7.92 (14 H, m).

^{13}C NMR ($CDCl_3$/TMS, 62.9 MHz): δ = 133.3, 132.0, 131.9, 128.4, 127.9, 127.7, 126.2, 62.8–63.1 (OCH_2CH_3), 57.7 (dd, $J_{P,C}$ = 155.1, 17.8 Hz), 16.1–16.3 (OCH_2CH_3).

^{31}P NMR ($CDCl_3$/H_3PO_4): δ = 22.75, 23.09 (89:11).

Anal. Calcd for $C_{30}H_{37}NO_6P_2$: C, 67.04; H, 6.89; N, 2.61. Found: C, 66.88; H, 6.78; N, 2.49.

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