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Diastereoselective Synthesis of *meso*-Bisphosphonates from Trialkyl(aryl) Phosphites and Activated Acetylenes in the Presence of 4-Nitrophenol

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Summary. The reaction between trialkyl(aryl) phosphites and dibenzoylacetylene or dimethyl acetylenedicarboxylate in the presence of 4-nitrophenol leads to stable *meso*-bisphosphonate derivatives in good yields.

Keywords. Bisphosphonates; Activated acetylenes; Trialkyl phosphites; Stereoselective synthesis.

Introduction

Organophosphorus compounds are versatile intermediates in synthetic chemistry [1–3]. In particular, organophosphonates that possess a C–P bond have found to be useful reagents, have a varied biological activity, and are useful in material chemistry [4–6]. As a result, a large number of methods have appeared describing novel syntheses of bisphosphonate systems [7–10]. There are also many studies on the reaction between trivalent phosphorus nucleophiles and α,β-unsaturated carbonyl compounds in the presence of a proton source, such as alcohol or phenol [11–13]. A recent paper of *Balaraman* and *Kumaraswamy* [11] intrigued us to present our work on the diastereoselective synthesis of *meso*-bisphosphonates through the reaction of trialkyl(aryl) phosphites and activated acetylenes in the presence of 4-nitrophenol.

Results and Discussion

The reaction of trialkyl(aryl) phosphites **2** with dibenzoylacetylene (*DBA*, **1a**) in the presence of 4-nitrophenol (**3**) as the proton source/nucleophile leads to dialkyl

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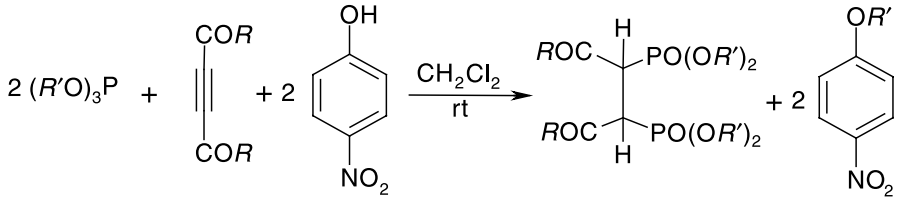


2	R′
a	*Me*
b	*Et*
c	*Ph*

1	R
a	*Ph*
b	*OMe*

3

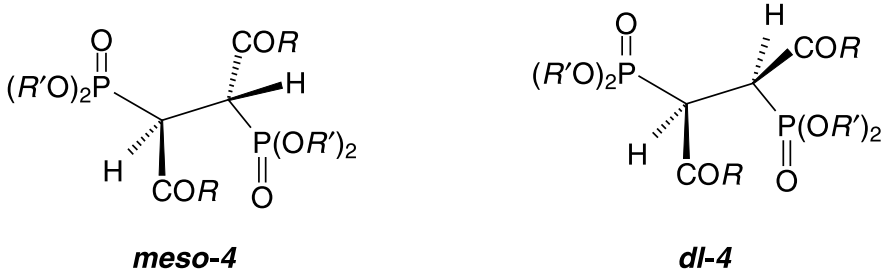
4	R	R′	Yield/%
a	*Ph*	*Me*	86
b	*Ph*	*Et*	88
c	*Ph*	*Ph*	87
d	*OMe*	*Me*	88
e	*OMe*	*Et*	83
f	*OMe*	*Ph*	85

5	R	Yield/%
a	*Me*	92
b	*Et*	87

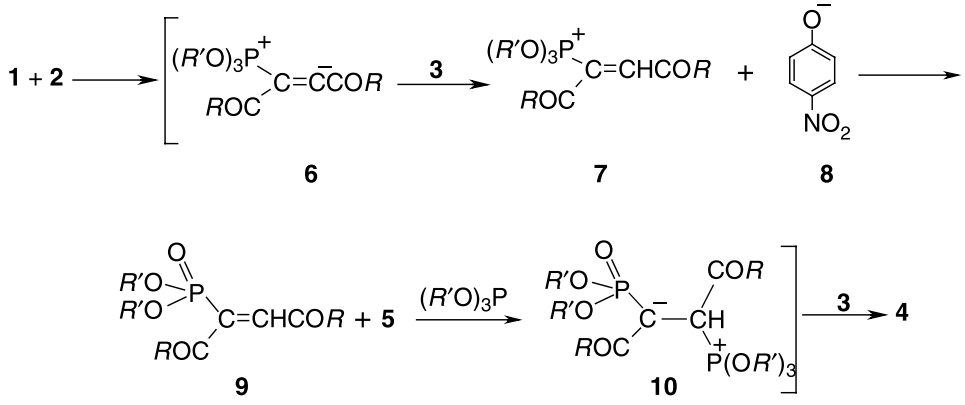
Scheme 1

[1-benzoyl-2- (dialkoxyphosphoryl)-3-oxo-3-phenylpropyl]phosphonates **4a**–**4c** in good yields. Similarly, dimethyl 2,3-bis(dialkoxyphosphoryl)succinates **4d**–**4f** were prepared in good yields using dimethyl acetylenedicarboxylate (*DMAD*, **1b**) as the activated acetylenic compound (Scheme 1).

The structures of bisphosphonates **4a**–**4f** and **5a**–**5b** were deduced from their elemental analyses and their IR, ^{1}H, ^{13}C, and ^{31}P NMR, and mass spectral data. The mass spectra of these compounds displayed molecular ion peaks at appropriate m/z values. The ^{1}H NMR spectrum of **4a** in $CDCl_3$ showed two doublets at $\delta = 3.42$ ($^{3}J_{HP} = 10.1$ Hz) and 3.64 ($^{3}J_{HP} = 9.1$ Hz) for the diastereotopic methoxy groups and a doublet of doublet at $\delta = 5.22$ ($^{2}J_{HP} = 10.6$ Hz and $^{3}J_{HH} = 7.9$ Hz) for the methine protons, along with multiplets at $\delta = 7.46$–8.06 ppm for the aromatic moieties. A single resonance at $\delta = 194.2$ ppm is observed in the ^{13}C NMR spectrum of **4a**, which is attributed to the carbonyl groups. The ^{31}P NMR signal of **4a** was found at $\delta = 23.23$ ppm. The ^{1}H and ^{13}C NMR spectra of **4b** and **4c** are similar to those of **4a** except for the phosphoranyl moieties. The ^{1}H NMR spectrum of **4d** in $CDCl_3$ showed two doublets at $\delta = 3.22$ ($^{3}J_{HP} = 8.7$ Hz) and 3.41 ($^{3}J_{HP} = 8.2$ Hz) for the diastereotopic methoxy groups and a doublet of doublet at $\delta = 4.75$ ($^{2}J_{HP} = 11.2$ Hz and $^{3}J_{HH} = 8.1$ Hz) for the methine moieties, along with a singlet at $\delta = 3.81$ ppm for the two CO_2Me groups. The ester carbonyl resonances in the ^{13}C NMR spectra of **4d** appear as a singlet at $\delta = 167.4$ ppm in the ^{13}C NMR



Scheme 2



Scheme 3

spectrum. The ^{31}P NMR signal of **4d** was found at $\delta = 19.58$ ppm. The ^{1}H and ^{13}C NMR spectra of **4e** and **4f** are similar to those of **4d** except for the phosphoranyl moieties.

Assignment of the *meso* structure to compounds **4** (Scheme 2) is based on the ^{13}C NMR spectra [11]. Whereas the P–CH carbon atoms of the *meso* diastereoisomer exhibits a 5-line pattern (A part of AXX′ system; X = X′ = phosphorus), a simple doublet is expected for the *dl* form [11]. The *meso* structure represents a high field limiting case which does not give a ''first order'' spectrum. Thus, some mixing of the basic functions is involved [14]. The *R*C=O carbon of the *meso* form appears essentially as a singlet (fairly small $^{2,3}J_{PC}$ values), but the *dl* form exhibits a multiplet (a doublet of doublet due to the higher $^{2,3}J_{PC}$ values).

Although we have not yet established the mechanism of the reaction between trialkyl(aryl) phosphites and acetylenes in the presence of 4-nitrophenol in an experimental manner, a plausible explanation is proposed in Scheme 3. On the basis of the well established chemistry of phosphorus nucleophiles [1–3], it is reasonable to assume that compounds **4** result from initial addition of phosphite to the activated acetylene and subsequent protonation of the reactive 1:1 adduct by 4-nitrophenol, followed by attack of the phenoxide ion at the alkyl group of the phosphite to generate **9** and 1-alkoxy-4-nitrobenzene **5**. Because of the higher nucleophilicity of phosphites compared to the conjugate base of 4-nitrophenol, intermediate **9** is attacked by a second phosphorus nucleophile.

The present method carries the advantage that, not only is the reaction performed under neutral conditions, but the educts can be mixed without any activation or modification. The simplicity of the present procedure for diastereoselective synthesis of bisphosphonates makes it an interesting alternative to complex multistep approaches.

Experimental

Dibenzoylacetylene was prepared according to Refs. [15, 16]. Other chemicals were purchased from Fluka and used without further purification. Melting points were measured on an Electrothermal 9100 aparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. The results agreed favorably with the calculated values. Mass spectra were recorded on a FINNIGAN-

MATT 8430 spectrometer operating at an ionization potential of 70 eV. IR spectra were measured on a Shimadzu IR-460 spectrometer. ^{1}H, ^{13}C, and ^{31}P NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500.1, 125.8, and 202.4 MHz.

Dimethyl [1-benzoyl-2-(dimethoxyphosphoryl)-3-oxo-3-phenylpropyl]phosphonate (**4a**, $C_{20}H_{24}O_8P_2$)

Typical procedure: To a stirred solution of 0.23 g *DBA* (1 mmol) and 0.28 g 4-nitrophenol (2 mmol) in 10 cm^3 CH_2Cl_2 was added 0.25 g trimethyl phosphite (2 mmol) at room temperature. The reaction mixture was then stirred for 24 h. The solvent was removed under reduced pressure and the viscous residue was purified by preparative TLC on silica gel column chromatography (Merck 230–400 mesh) using *n*-hexane-*Et*O*Ac* as eluent to give **4a** as white powder, mp 158–160°C, yield 0.39 g, 86%; IR (KBr): $\bar{\nu} = 2950$, 1667, 1584, 1258, 1015 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.42$ (6 H, d, $^{3}J_{HP} = 10.1$ Hz, 2 OMe), 3.64 (6 H, d, $^{3}J_{HP} = 9.1$ Hz, 2 OMe), 5.22 (2 H, dd, $^{2}J_{HP} = 10.6$ Hz and $^{3}J_{HH} = 7.9$ Hz, 2 CH), 7.47 (4 H, t, $^{3}J_{HH} = 7.2$ Hz, 4 CH_{meta} of 2 C_6H_5), 7.55 (2 H, t, $^{3}J_{HH} = 6.4$ Hz, 2 CH_{para} of 2 C_6H_5), 8.05 (4 H, d, $^{3}J_{HH} = 7.4$ Hz, 4 CH_{ortho} of 2 C_6H_5) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 45.9$, 46.2, 46.6, 47.2, 47.5 (5 lines for P–CH), 53.4 (d, $^{2}J_{CP} = 3.1$ Hz, 2 OCH_3), 53.5 (d, $^{2}J_{CP} = 3.3$ Hz, 2 OCH_3), 128.6 (s, 4 CH of C_6H_5), 128.7 (s, 4 CH of 2 C_6H_5), 133.3 (s, 2 CH of 2 C_6H_5), 137.1 (s, 2 C_{ipso} of 2 C_6H_5), 194.2 (s, C=O) ppm; ^{31}P NMR (202 MHz, $CDCl_3$): $\delta = 23.23$ (P=O) ppm; MS (EI, 70 eV): m/z (%) = 454 (M^+, 10), 423 (15), 349 (64), 344 (38), 227 (10), 196 (24), 110 (58), 105 (100), 31 (86).

Diethyl [1-benzoyl-2-(diethoxyphosphoryl)-3-oxo-3-phenylpropyl]phosphonate (**4b**, $C_{24}H_{32}O_8P_2$)

White powder, mp 167–169°C, yield 0.45 g, 88%; IR (KBr): $\bar{\nu} = 2925$, 1675, 1584, 1254, 1024 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.25$ (12 H, t, $^{3}J_{HH} = 7.0$ Hz, 4 Me), 4.03 (8 H, m, 4 OCH_2), 4.72 (2 H, dd, $^{2}J_{HP} = 14.7$ Hz and $^{3}J_{HH} = 10.2$ Hz, 2 CH), 7.41 (4 H, t, $^{3}J_{HH} = 7.7$ Hz, 4 CH_{metha} of 2 C_6H_5), 7.55 (2 H, t, $^{3}J_{HH} = 6.4$ Hz, 2 CH_{para} of 2 C_6H_5), 8.03 (4 H, d, $^{3}J_{HH} = 7.9$ Hz, 4 CH_{ortho} of 2 C_6H_5) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 16.0$ (d, $^{3}J_{CP} = 6.5$ Hz, 2 CH_3), 16.2 (d, $^{3}J_{CP} = 5.97$ Hz, 2 CH_3), 47.5, 47.8, 48.6, 49.3, 40.5 (5 lines for P–CH), 62.9 (d, $^{2}J_{CP} = 6.7$ Hz, 2 OCH_2), 63.5 (d, $^{2}J_{CP} = 5.9$ Hz, 2 OCH_2), 128.5 (s, 4 CH of C_6H_5), 128.6 (s, 4 CH of 2 C_6H_5), 133.3 (s, 2 CH of 2 C_6H_5), 135.8 (s, 2 C_{ipso} of 2 C_6H_5), 195.23 (s, C=O) ppm; ^{31}P NMR (202 MHz, $CDCl_3$): $\delta = 22.39$ (P=O) ppm; MS (EI, 70 eV): m/z (%) = 510 (M^+, 5), 465 (25), 405 (52), 373 (28), 255 (24), 137 (10), 105 (100), 45 (75).

Diphenyl [1-benzoyl-2-(diphenoxyphosphoryl)-3-oxo-3-phenylpropyl]phosphonate (**4c**, $C_{40}H_{32}O_8P_2$)

Colorless crystals, mp 185–187°C, yield 0.61 g, 87%; IR (KBr): $\bar{\nu} = 2920$, 1673, 1581, 1252, 1014 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.23$ (2 H, dd, $^{2}J_{HP} = 11.6$ Hz and $^{3}J_{HH} = 10.7$ Hz, 2 CH), 7.08 (4 H, t, $^{3}J_{HH} = 8.0$ Hz, 4 CH_{para} of 4 OC_6H_5), 7.09 (8 H, d, $^{3}J_{HH} = 8.5$ Hz, 8 CH_{ortho} of 4 OC_6H_5), 7.17 (8 H, dd, $^{3}J_{HH} = 7.6$ Hz, $^{3}J_{HH} = 7.9$ Hz, 8 CH_{meta} of 4 OC_6H_5), 7.37 (4 H, t, $^{3}J_{HH} = 7.4$ Hz, 4 CH_{meta} of 2 C_6H_5), 7.43 (2 H, t, $^{3}J_{HH} = 7.2$ Hz, 2 CH_{para} of 2 C_6H_5), 7.65 (4 H, d, $^{3}J_{HH} = 7.3$ Hz, 4 CH_{ortho} of 2 C_6H_5) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 44.7$, 45.4, 45.9, 46.4, 46.8 (5 lines for P–CH), 120.8 (d, $^{3}J_{CP} = 4.9$, 8 CH of 4 OC_6H_5), 125.0 (s, 8 CH of 4 OC_6H_5), 128.2 (s, 4 CH of 2 OC_6H_5), 129.5 (s, 4 CH of 2 C_6H_5), 129.7 (s, 4 CH of 2 C_6H_5), 130.5 (s, 2 CH of 2 C_6H_5), 150.4 (s, 2 C_{ipso} of 2 C_6H_5), 162.4 (d, $^{2}J_{PC} = 15.0$ Hz, 2 C_{ipso} of 2 OC_6H_5), 162.6 (d, $^{2}J_{PC} = 15.2$ Hz, 2 C_{ipso} of 2 OC_6H_5), 194.2 (s, C=O) ppm; ^{31}P NMR (202 MHz, $CDCl_3$): $\delta = 11.02$ (P=O) ppm; MS (EI, 70 eV): m/z (%) = 702 (M^+, 5), 469 (15), 351 (35), 236 (38), 233 (38), 105 (100), 93 (85), 77 (86).

Dimethyl 2,3-bis(dimethoxyphosphoryl)succinate (**4d**, $C_{10}H_{20}O_{10}P_2$)

White powder, mp 73–74°C, yield 0.32 g, 88%; IR (KBr): $\bar{\nu} = 2922$, 1720, 1583, 1259, 1154, 1024 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.22$ (6 H, d, $^{3}J_{HP} = 8.7$ Hz, 2 OMe), 3.41 (6 H, d,

$^3J_{HP} = 8.2$ Hz, 2 OMe), 3.81 (6 H, s, 2 CO_2Me), 4.75 (2 H, dd, $^2J_{HP} = 11.2$ Hz and $^3J_{HH} = 8.1$ Hz, 2 CH) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 42.9$, 43.2, 43.7, 44.2, 44.5 (5 lines for P–CH), 52.4 (d, $^2J_{CP} = 3.5$ Hz, 2 $POCH_3$), 52.8 (d, $^2J_{CP} = 3.8$ Hz, 2 $POCH_3$), 55.6 (s, 2 CO_2CH_3), 167.4 (s, C=O) ppm; ^{31}P NMR (202 MHz, $CDCl_3$): $\delta = 19.58$ (P=O) ppm; MS (EI, 70 eV): m/z (%) = 362 (M^+, 5), 331 (35), 253 (20), 181 (48), 110 (100), 31 (78).

Dimethyl 2,3-bis(diethoxyphosphoryl)succinate (**4e**, $C_{14}H_{28}O_{10}P_2$)
White powder, mp 85–87°C, yield 0.35 g, 83%; IR (KBr): $\bar{\nu} = 2920$, 1731, 1589, 1261, 1158, 1029 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.21$ (12 H, t, $^3J_{HH} = 7.0$ Hz, 4 Me), 3.60 (2 H, dd, $^2J_{HP} = 10.2$ Hz and $^3J_{HH} = 8.1$ Hz, 2 CH), 3.83 (6 H, s, 2 OMe), 4.01 (8 H, m, 4 OCH_2) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 15.8$ (d, $^3J_{CP} = 6.9$ Hz, 2 CH_3), 15.9 (d, $^3J_{CP} = 6.7$ Hz, 2 CH_3), 39.2, 39.5, 40.3, 41.2, 41.8 (5 lines for P–CH), 54.6 (s, 2 OCH_3), 62.9 (d, $^2J_{CP} = 5.9$ Hz, 2 OCH_2), 63.5 (d, $^2J_{CP} = 5.8$ Hz, 2 OCH_2), 168.6 (s, C=O) ppm; ^{31}P NMR (202 MHz, $CDCl_3$): $\delta = 21.09$ (P=O) ppm; MS (EI, 70 eV): m/z (%) = 418 (M^+, 6), 387 (10), 251 (20), 223 (96), 209 (10), 138 (48), 113 (100), 45 (78).

Dimethyl 2,3-bis(diphenoxyphosphoryl)succinate (**4f**, $C_{30}H_{28}O_{10}P_2$)
Colorless crystals, mp 173–175°C, yield 0.52 g, 85%; IR (KBr): $\bar{\nu} = 2950$, 1739, 1581, 1277, 1246, 1184, 1155 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.75$ (6 H, s, OMe), 4.33 (2 H, dd, $^2J_{HP} = 12$ Hz and $^3J_{HH} = 6.0$ Hz, CH), 7.16 (4 H, t, $^3J_{HH} = 8.0$ Hz, 4 CH_{para} of 4 C_6H_5), 7.25 (8 H, d, $^3J_{HH} = 5.7$ Hz, 8 CH_{ortho} of 4 C_6H_5), 7.31 (8 H, dd, $^3J_{HH} = 8.4$ Hz and $^3J_{HH} = 7.5$ Hz, 8 CH_{meta} of 4 C_6H_5) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 44.4$, 44.7, 45.2, 45.7, 46.0 (5 lines for P–CH), 53.3 (2 OCH_3), 120.8 [d, $^3J_{CP} = 7.5$, 8 CH_{ortho} of $(C_6H_5O)_2PO$], 125.6 [d, $^4J_{PC} = 2.5$ Hz, CH_{meta} of $(C_6H_5O)_2PO$], 129.8 [s, 4 CH_{para} of $(C_6H_5O)_2PO$], 150.0 [d, $^2J_{PC} = 14.6$ Hz, 2 C_{ipso} of $(C_6H_5O)_2PO$], 150.1 [d, $^2J_{PC} = 14.4$ Hz, 2 C_{ipso} of $(C_6H_5O)_2PO$], 166.3 (s, C=O) ppm; ^{31}P NMR (202 MHz, $CDCl_3$): $\delta = 11.73$ (P=O) ppm; MS (EI, 70 eV): m/z (%) = 610 (M^+, 6), 579 (20), 517 (100), 485 (10), 318 (10), 285 (80), 223 (58), 140 (46), 94 (40), 77 (100).

1-Methoxy-4-nitrobenzene (**5a**, $C_7H_7NO_3$)
Colorless crystals, mp 75–77°C, yield 0.14 g, 92%; IR (KBr): $\bar{\nu} = 1683$, 1492, 1333, 1261, 1173, 1105, 1016 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.89$ (3 H, s, OMe), 6.93 (2 H, d, $^3J_{HH} = 9.1$ Hz, 2 CH of C_6H_5), 8.17 (2 H, d, $^3J_{HH} = 9.2$ Hz, 2 CH of C_6H_5) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 55.9$ (s, CH_3), 114.0 (s, 2 CH), 125.9 (s, 2 CH), 141.5 (s, C_{ipso} of Ph), 164.6 (s, C_{ipso} of Ph) ppm.

1-Ethoxy-4-nitrobenzene (**5b**, $C_8H_9NO_3$)
Yellow crystals, mp 79–81°C, yield 0.13 g, 87%; IR (KBr): $\bar{\nu} = 1585$, 1490, 1329, 1257, 1177, 1102, 1033 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.45$ (3 H, t, $^3J_{HH} = 6.9$ Hz, Me), 4.12 (2 H, q, $^3J_{HH} = 6.9$ Hz, OCH_2), 6.93 (2 H, d, $^3J_{HH} = 9.2$ Hz, 2 CH of C_6H_5), 8.18 (2 H, d, $^3J_{HH} = 9.2$ Hz, 2 CH of C_6H_5) ppm; ^{13}C NMR (125.7 MHz, $CDCl_3$): $\delta = 14.5$ (s, CH_3), 64.4 (s, OCH_2), 114.4 (s, 2 CH of Ph), 125. 9 (s, 2 CH of Ph), 141.4 (s, C_{ipso} of Ph), 164.0 (s, C_{ipso} of Ph) ppm.

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