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Nucleophilic Addition of Triethyl Phosphite to Acetates of the Baylis-Hillman Adducts: Stereoselective Synthesis of (E)- and (Z)-Allylphosphonates

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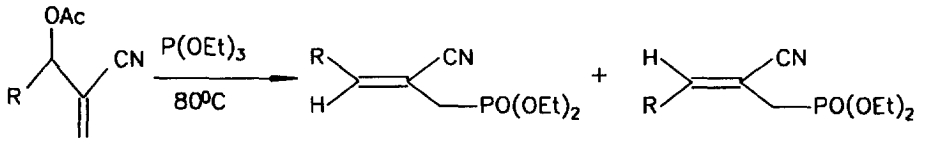
Abstract: Nucleophilic addition of triethyl phosphite to 3-acetoxy-2-methylenealkanenitriles and methyl 3-acetoxy-2-methylenealkanoates provides (2E)-2-(diethoxyphosphorylmethyl)alk-2-enenitriles and methyl (2Z)-2-(diethoxyphosphorylmethyl)alk-2-enoates respectively with good stereoselectivity.

The Baylis-Hillman reaction[1-6] provides synthetically attractive and useful molecules possessing chemospecific functional groups such as hydroxyl, an alkene and an electron withdrawing group in close proximity. The proximity of these functional groups is a key issue for the high utility of the Baylis-Hillman adducts in a variety of stereoselective processes.[7-16] In continuation of our interest[11-15] in the Baylis-Hillman reaction we herein report a simple and convenient stereoselective synthesis of (2E)-2-(diethoxyphosphorylmethyl)alk-2-enenitriles and methyl (2Z)-2-(diethoxyphosphorylmethyl)alk-2-enoates important precursors for synthetically attractive 2-substituted-1,3-butadienes *via* the treatment of acetates of the Baylis-Hillman adducts, 3-acetoxy-2-methylenealkanenitriles (**1**) and methyl 3-acetoxy-2-methylenealkanoates (**2**), with triethyl phosphite.

Recently, we have developed a simple and convenient methodology for stereoselective synthesis of various functionalized trisubstituted alkenes *via* the addition of various nucleophiles to acetates of the Baylis-Hillman adducts.[11-15] We envisaged that the nucleophilic attack of triethyl phosphite to acetates of Baylis-Hillman adducts (**1** & **2**) might provide, after thermal Arbuzov rearrangement, the required (E)- and (Z)-allylphosphonates with high stereoselectivity.

Accordingly we first carried out the reaction of 3-acetoxy-2-methylenealkanenitriles (**1**) (5 mM) with triethyl phosphite (7 mM) at 80°C for

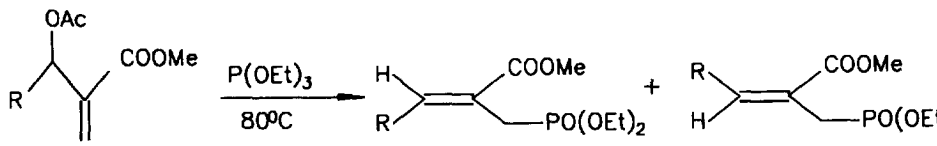
30 min. to provide (2E)-2-(diethoxyphosphorylmethyl)alk-2-enenitriles (**3**) in high stereoselectivity and in excellent yields (Eq.1, Table 1). It is evident from Table 1 that the level of stereocontrol in formation of allylphosphonates **3** depends on the nature of substituent (R) in the substrate **1**. Thus, substrates with alkyl substituents (**1c-1f**, Table 1) afforded the corresponding allylphosphonates with high (E)-stereoselectivity (E\Z = >90\<10) whereas substrates with aryl substituents (**1a**, **1b**) afforded the allylphosphonates in moderate (E)-stereoselectivity (E\Z = 75-80\25-20).



(1)

1a-1f **93-75% (E)** **3a-3f** **7-25% (Z)**

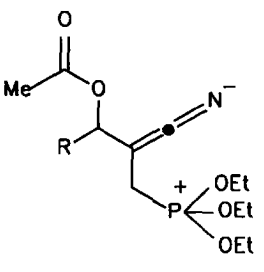
Encouraged by this observation we next studied the reaction of methyl 3-acetoxy-2-methylenealkanoates (**2**) (5 mM) with triethyl phosphite (7 mM) which afforded the corresponding allylphosphonates **4** with predominant (Z)-stereoselectivity and in excellent yields (Eq 2, Table 2). In this case also, the nature of substituent (R) has significant influence on the stereochemical outcome, thus allylphosphonates were obtained with high (Z)-stereoselectivity (Z\E = >90\<10) when R is aryl and with moderate stereoselectivity when R is alkyl.



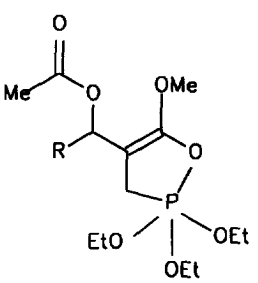
(2)

2a-2f **93-68% (Z)** **4a-4f** **7-32% (E)**

Though the exact mechanism of this reaction is not yet clear we propose two possible transition state models (**I**) and (**II**) for the above stereochemical observations.



(I)



(II)

Table 1: Synthesis of (2E)-2-(diethoxyphosphorylmethyl)alk-2-enenitriles[a-c]

Acetate of Baylis-Hillman adduct	R	Product	b.p. (°C) / mm Hg	Yield[d] (%)	E : Z[e]
1a	Ph	**3a**	190-192/2.8	94	75 : 25
1b	4-MeC_6H_4	**3b**	182-184/1.2	90	80 : 20
1c	Me	**3c**	109-110/1.4	85	91 : 09
1d	n-Pr	**3d**	146-148/1.8	91	92 : 08
1e	n-Hex	**3e**	172-174/2.1	88	93 : 07
1f	i-Pr	**3f**	139-141/1.8	92	91 : 09

a) All reactions were carried out on 5 mM scale of acetate with triethyl phosphite (7 mM) at 80°C for 30 min. b) Satisfactory spectral [IR, ^{1}H NMR (200 MHz), ^{13}C NMR (50 MHz) and ^{31}P NMR (81 MHz)] data and microanalyses were obtained for all products **3a-3f** c) (E)-Stereochemistry was assigned to all major isomers of **3a-3f** on the basis of ^{1}H NMR spectra d) Isolated yield of the products after distillation under reduced pressure. e) Determined by ^{1}H NMR spectral analysis.

Table 2: Synthesis of methyl (2Z)-2-(diethoxyphosphorylmethyl)alk-2-enoates[a-c]

Acetate of Baylis-Hillman adduct	R	Product	b.p. (°C) / mm Hg	Yield[d] (%)	Z : E[e]
2a	Ph	**4a**	168-170/1.6	87	91 : 09
2b	4-MeC_6H_4	**4b**	193-195/3.5	90	90 : 10
2c	4-ClC_6H_4	**4c**	204-207/2.3	92	90 : 10
2d	2-MeOC_6H_4	**4d**	185-186/1.3	95	93 : 07
2e	n-Pr	**4e**	144-146/2.9	93	68 : 32
2f	n-Hex	**4f**	164-166/2.0	90	69 : 31

a) All reactions were carried out on 5 mM scale of acetate with triethyl phosphite (7mM) at 80°C for 30 min. b) Satisfactory spectral [IR, ^{1}H NMR (200 MHz), ^{13}C NMR (50 MHz) and ^{31}P NMR (81 MHz)] data and microanalyses were obtained for all products **4a-4f** c) (Z)-Stereochemistry was assigned to all major isomers of **4a-4f** on the basis of ^{1}H NMR spectra. d) Isolated yield of the products after distillation under reduced pressure. e) Determined by ^{1}H NMR spectral analysis.

It is worth mentioning here that the Janecki and Bodalski[16] have recently described the synthesis of (E)- and (Z)-allylphosphonates *via* thermal Arbuzov rearrangement of allyl phosphites which are obtained by treating the Baylis-Hillman adducts with diethyl phosphorochloridite. According to this report, esters (methyl 3-hydroxy-2-methylenealkanoates) provide the resulting allylphosphonates in high stereoselectivity (Z/E = 95/5) whereas nitriles (3-hydroxy-2-methylenealkanenitriles) provide the allylphosphonates in moderate stereoselectivity (E/Z = 60-75/40-25). In our work, nitriles **1** provide allylphosphonates with high stereoselectivity (E/Z =>90\<10) particularly for aliphatic substituents (R = alkyl) thus complementing the work of Janecki and Bodalski.

In conclusion, our methodology describes the application of triethyl phosphite as a nucleophile thus providing stereoselective synthesis of (E)- and (Z)-allylphosphonates in excellent yields. Our study also describes the influence of alkyl and aryl substituents on the degree of stereoselectivity in the synthesis of the allylphosphonates.

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EXPERIMENTAL

The boiling points were uncorrected. IR spectra were recorded on Jasco-FT-IR model 5300 or Perkin-Elmer model 1310 spectrometer using samples as neat liquid. ^{1}H NMR (200 MHz) and ^{13}C NMR (50 MHz) spectra were recorded on Bruker-AC-200 spectrometer using tetramethylsilane (TMS, δ = 0 ppm) as internal standard. ^{31}P NMR (81 MHz) spectra were recorded on Bruker-AC-200 spectrometer using 85% H_3PO_4 (δ = 0 ppm) as external standard. In ^{1}H and ^{31}P NMR spectra, the underlined chemical shift values are due to minor isomers. ^{13}C NMR spectral data refers to only the major isomer.

General procedure for the preparation (E)- and (Z)-allylphosphonates:

A solution of acetate of the Baylis-Hillman adduct (**1** & **2**) (5 mM) and triethyl phosphite (1.2 mL, 7 mM) was stirred at 80^{o}C for 30 min. Fractional distillation of reaction mixture under reduced pressure afforded the allylphosphonate (**3** & **4**) as viscous liquid in excellent yields.

2-(Diethoxyphosphorylmethyl)-3-phenylprop-2-enenitrile (3a):

Yield: 94%; b.p: 190-192°C/2.8 mm; E : Z = 75 : 25; IR (neat) ν_{max}\ cm^{-1} : 1620, 2216; ^{1}H NMR: δ 1.36 (t, 6H, J = 7.4 Hz), 2.90 & 2.98 (2d, 2H, J = 21 Hz), 4.02-4.34 (m, 4H), 7.12 (d, 1H, J = 5.1 Hz), 7.34-7.84 (m, 5H); ^{13}C NMR: δ 16.17 (d, J = 5.3 Hz) 32.97 (d, J = 140.6 Hz), 62.48 (d, J = 6.3 Hz), 101.05 (d, J = 12.2 Hz), 117.99 (d, J = 4.8 Hz), 128.62 130.29, 133.06 (d, J = 3 Hz), 147.30 (d, J = 10.5 Hz); ^{31}P NMR: δ 21.65 & 21.97; Analysis calculated for $C_{14}H_{18}NO_3P$: C, 60.21; H, 6.49; N, 5.01; found C, 60.19; H, 6.46; N, 5.02.

2-(Diethoxyphosphorylmethyl)-3-(4-methylphenyl)prop-2-enenitrile (3b):

Yield: 90%; b.p: 182-184°C/1.2 mm; E : Z = 80 : 20; IR (neat) ν_{max}\ cm^{-1}: 1608, 2214; ^{1}H NMR : δ 1.36 (t, 6H, J = 7 Hz), 2.38 (s, 3H), 2.88 & 3.02 (2d, 2H, J = 20.8 Hz) 4.08-4.32 (m, 4H), 7.08 (d, 1H, J = 6.2 Hz) 7.16-7.72 (m, 4H); ^{13}C NMR : δ 16.28 (d, J = 5.1 Hz), 21.34, 33.02 (d, J = 140.8 Hz), 62.55 (d, J = 6.2 Hz), 99.63 (d, J = 12.1 Hz), 118.34 (d, J = 4.7 Hz), 128.72, 129.43, 130.46 (d, J = 3.3 Hz), 140.93, 147.41 (d, J = 10.3 Hz); ^{31}P NMR: δ 21.81 & 22.18; Analysis calculated for $C_{15}H_{20}NO_3P$: C, 61.43; H, 6.87; N, 4.77; found: C, 61.37; H, 6.84; N, 4.76.

2-(Diethoxyphosphorylmethyl)but-2-enenitrile (3c):

Yield: 85%; b.p: 109-110°C/1.4 mm; E : Z = 91 : 09; IR (neat) ν_{max}\ cm^{-1} : 1635, 2215 cm^{-1}; ^{1}H NMR: δ 1.30 (t, 6H, J = 7.2 Hz), 1.88 & 1.99 (2m, 3H), 2.65 & 2.70 (2d, 2H, J = 20.6 Hz), 4.10 (m, 4H), 6.41 & 6.59 (2m, 1H); ^{13}C NMR: δ 16.22 (d, J = 5.7 Hz), 17.39, 31.19 (d, J = 141.8 Hz), 62.40 (d, J = 6.7 Hz), 105.91 (d, J = 11.5 Hz), 116.49, 147.44 (d, J = 10.6 Hz); ^{31}P NMR: δ 22.14 & 22.29; Analysis calculated for $C_9H_{16}NO_3P$: C, 49.76; H, 7.42; N, 6.44; found C, 49.65; H, 7.45; N, 6.40.

2-(Diethoxyphosphorylmethyl)hex-2-enenitrile (3d):

Yield: 91%; b.p: 146-148°C/1.8 mm; E : Z = 92 : 08; IR (neat) ν_{max}\ cm^{-1} : 1640, 2220; ^{1}H NMR : δ 0.96 (t, 3H, J = 7.4 Hz), 1.35 (t, 6H, J = 6.6 Hz), 1.51 (sext, 2H, 7.4 Hz), 2.26 & 2.39 (2m, 2H), 2.71 & 2.75 (2d, 2H, J = 20.5 Hz), 4.16 (m, 4H), 6.39 & 6.52 (2m, 1H). ^{13}C NMR : δ 13.16, 16.09 (d, J = 5.7 Hz), 21.40 (d, J = 2.6 Hz), 31.05 (d, J = 141.4 Hz) 33.45, 62.22 (d, J = 6.4 Hz) 104.85 (d, J = 11.4 Hz), 116.56, 152.32 (d, J = 10.4 Hz); ^{31}P NMR : δ 21.91 & 22.24; Analysis calculated for $C_{11}H_{20}NO_3P$: C, 53.87; H, 8.22; N, 5.71; found: C, 53.92; H, 8.20; N, 5.69.

2-(Diethoxyphosphorylmethyl)non-2-enenitrile (3e):

Yield: 88%; b.p: 172-174°C/2.1 mm; E : Z = 93 : 07; IR (neat) ν_{max}\ cm^{-1} : 1620, 2220; ^1H NMR: δ 0.88 (dist t, 3H), 1.18-1.56 (m, 14H), 2.26 & 2.41 (2m, 2H), 2.69 & 2.73 (2d, 2H, J = 20.7 Hz), 4.15 (m, 4H), 6.39 & 6.52 (2m, 1H); ^{13}C NMR: δ 13.72, 16.10 (d, J = 5.7 Hz), 22.24, 28.03, 28.36, 31.08 (d, J = 141.4 Hz), 31.22, 31.60, 62.25 (d, J = 6.7 Hz), 104.67 (d, J = 11.5 Hz), 116.62 (d, J = 4.2 Hz), 152.60 (d, J = 10.5 Hz); ^{31}P NMR: δ 21.92 & 22.24; Analysis calculated for $C_{14}H_{26}NO_3P$: C, 58.52; H, 9.12; N, 4.87; found C, 58.45; H, 9.15; N, 4.86.

2-(Diethoxyphosphorylmethyl)-4-methylpent-2-enenitrile (3f):

Yield: 92%; b.p: 139-141°C/1.8 mm; E : Z = 91 : 09; IR (neat) ν_{max}\ cm^{-1} : 1620, 2210; ^1H NMR : δ 1.03 & 1.05 (2d, 6H, J = 6.6 Hz), 1.33 & 1.34 (2t, 6H, J = 7 Hz), 2.64 & 2.72 (2d, 2H, J = 20.6 Hz), 2.80-2.98 (m, 1H), 4.13 (m, 4H), 6.16 & 6.31 (2m, 1H) ^{13}C NMR: δ 16.07 (d, J = 5.7 Hz), 21.50, 21.54, 30.95 (d, J = 142 Hz), 31.39, 62.22 (d, J = 6.5 Hz), 102.41 (d, J = 11.3 Hz), 116.41, 158.72 (d, J = 10.5 Hz); ^{31}P NMR: δ 21.94 & 22.13; Analysis calculated for $C_{11}H_{20}NO_3P$: C, 53.87; H, 8.22; N, 5.71; found: C, 53.95; H, 8.25; N, 5.70.

Methyl 2-(diethoxyphosphorylmethyl)-3-phenylprop-2-enoate (4a):

Yield: 87%; b.p: 168-170°C/1.6 mm; Z : E = 91 : 09; IR (neat) ν_{max}\ cm^{-1}: 1615, 1710; ^1H NMR : δ 1.26 & 1.31 (2t, 6H, J = 7 Hz), 2.99 & 3.23 (2d, 2H, J = 22.5 Hz), 3.63 & 3.84 (2s, 3H), 3.95-4.28 (m, 4H), 6.90 & 7.75 (2d, 1H, J = 5.5 Hz), 7.18-7.62 (m, 5H); ^{13}C NMR : δ 16.05 (d, J = 6.1 Hz), 25.98 (d, J = 139.5 Hz), 52.02, 61.83 (d, J = 6.4 Hz), 123.71 (d, J = 11.6 Hz), 128.35, 128.73, 129.17, 134.64 (d, J = 2.8 Hz), 141.18 (d, J = 11 Hz), 167.73; ^{31}P NMR : δ 24.31 & 24.89; Analysis calculated for $C_{15}H_{21}O_5P$: C, 57.68; H, 6.77; found C, 57.72; H, 6.80.

Methyl 2-(diethoxyphosphorylmethyl)-3-(4-methylphenyl)prop-2-enoate (4b)

Yield: 90%; b.p: 193-195°C/3.5 mm; Z : E = 90 : 10; IR (neat) ν_{max}\ cm^{-1} : 1610, 1640, 1705; ^1H NMR: δ 1.24 & 1.34 (2t, 6H, J = 7 Hz), 2.32 & 2.34 (2s, 3H), 2.98 & 3.22 (2d, 2H, J = 22.4 Hz), 3.66 & 3.81 (2s, 3H), 4.06 (quint, 4H, J = 7 Hz), 6.88 & 7.74 (2d, 1H, J = 5.6 Hz), 7.11-7.59 (m, 4H); ^{13}C NMR: δ 16.07 (d, J = 6.1 Hz), 21.08, 26.03 (2d, J = 139.7 Hz), 51.98, 61.82 (d, J = 6.6 Hz), 122.64 (d, J = 11.6 Hz), 129.07, 129.34, 131.75 (d, J = 3.1 Hz), 138.98, 141.29 (d, J = 10.9 Hz), 167.88; ^{31}P NMR: δ 24.39 & 24.99; Analysis calculated for $C_{16}H_{23}O_5P$: C, 58.89; H, 7.10; found C, 58.95; H, 7.14.

Methyl 3-(4-chlorophenyl)-2-(diethoxyphosphorylmethyl)prop-2-enoate (4c)

Yield: 92%; b.p: 204-207°C/2.3 mm; Z : E = 90 : 10; IR (neat) ν_{max}\ cm^{-1}: 1615, 1645, 1708; ^{1}H NMR: δ 1.26 & 1.31 (2t, 6H, J = 6.9 Hz), 3.01 & 3.18 (2d, 2H, J = 22.6 Hz), 3.64 & 3.83 (2s, 3H), 4.08 (quint, 4H, J = 7 Hz), 6.88 & 7.72 (2d, 1H, J = 7.4 Hz), 7.18 & 7.32 (2d, 2H, J = 8 Hz), 7.28 & 7.56 (2d, 2H, J = 8 Hz); ^{13}C NMR: δ 16.15 (d, J = 6 Hz), 26.15 (d, J = 139.5 Hz), 52.19, 62.00 (d, J = 6.7 Hz), 124.35 (d, J = 11.7 Hz), 128.67, 130.67, 133.10 (d, J = 3 Hz), 134.88, 139.95 (d, J = 11.1 Hz), 167.59; ^{31}P NMR: δ 24.01 & 24.47 Analysis calculated for $C_{15}H_{20}O_5PCl$: C, 51.95; H, 5.81; found: C, 51.85; H, 5.81.

Methyl 2-(diethoxyphosphorylmethyl)-3-(2-methoxyphenyl)prop-2-enoate (4d):

Yield: 95%; b.p: 185-186°C/1.5 mm; Z : E = 93 : 07; IR (neat) ν_{max}\ cm^{-1}: 1633, 1716; ^{1}H NMR: δ 1.26 & 1.28 (2t, 6H, J = 7 Hz), 3.08 & 3.18 (2d, 2H, J = 22.4 Hz), 3.61 & 3.84 (2s, 6H), 4.06 (quint, 4H, J = 7 Hz), 6.82-7.06 (m, 2H), 7.28-7.38 (m, 1H), 7.73 (d, 1H, J = 6.6Hz), 7.94 (d, 1H, J = 6Hz). ^{13}C NMR: 16.03 (d, J = 6.1 Hz), 26.08 (d, J = 139.5 Hz), 51.92, 55.20, 61.73 (d, J = 6.3 Hz), 110.25, 120.22, 123.43, 123.65, 129.77, 130.27, 137.32 (d, J = 11.1 Hz), 157.26, 167.77; ^{31}P NMR: δ 24.39 & 25.19; Analysis calculated for $C_{16}H_{23}O_6P$: C, 56.13; H, 6.77; found C, 56.08; H, 6.73.

Methyl 2-(diethoxyphosphorylmethyl)hex-2-enoate (4e):

Yield: 93%; b.p: 144-146°C/2.9 mm; Z : E = 68 : 32; IR (neat) ν_{max}\ cm^{-1}: 1645, 1716; ^{1}H NMR: δ 0.90 & 0.93 (2t, 3H, J = 6.5 Hz), 1.28 & 1.34 (2t, 6H, J = 7 Hz), 1.50 (m, 2H), 2.28 & 2.50 (2m, 2H), 2.84 & 2.98 (2d, 2H, J = 24 Hz) 3.76 (s, 3H), 4.08 (quint, 4H, J = 7 Hz), 6.14 & 6.91 (2q, 1H, J = 6 Hz); ^{13}C NMR: δ 13.06, 15.54 (d, J = 5.9 Hz), 20.95 (d, J = 1.5 Hz), 24.19 (d, J = 139.7 Hz), 30.46 (d, J = 1.5 Hz), 51.07, 61.07 (d, J = 6.4 Hz), 122.33 (d, J = 11.3 Hz), 145.06 (d, J = 10.2 Hz), 166.28; ^{31}P NMR: δ 25.04 & 25.24; Analysis calculated for $C_{12}H_{23}O_5P$: C, 51.79; H, 8.33; found C, 51.74; H, 8.31.

Methyl 2-(diethoxyphosphorylmethyl)non-2-enoate (4f):

Yield: 90%; b.p: 164-166°C/2.0 mm; Z : E = 69 : 31; IR (neat) ν_{max}\ cm^{-1}: 1645, 1718; ^{1}H NMR: δ 0.87 (dist. t, 3H), 1.12-1.58 (m, 14H), 2.24 & 2.52 (2m, 2H), 2.85 & 2.95 (2d, 2H, J = 24 Hz), 3.72 (s, 3H), 4.08 (quint, 4H, J = 7 Hz), 6.16 & 6.92 (2q, 1H, J = 5.8 Hz). ^{13}C NMR: δ 13.55, 15.89 (d, J = 5.9 Hz), 22.11, 24.53 (d, J = 139.8 Hz), 27.98,

28.65, 28.86, 31.21, 51.44, 61.42 (d, J = 6.5 Hz), 122.42 (d, J = 11.3 Hz), 145.79 (d, J = 10.1 Hz), 166.68; ^{31}P NMR: δ 25.30 & 25.46; Analysis calculated for $C_{15}H_{29}O_5P$: C, 56.23; H, 9.12; found: C, 56.25; H, 9.13.

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