Reactions of β-oxo-α,β-bis(alkoxycarbonyl)phosphonium ylides based on P-heterocycles

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The title phosphonium ylides give an ylidene oxolactone under reductive conditions and are unreactive towards aldehydes, but are degraded by hydroxylamine to form the simpler acetic ester phosphonium ylides that do react with aldehydes in the normal manner.

Keywords: phosphorus heterocycles, phosphoranes/ylides, lactones, Wittig reactions

It was a surprising discovery that a series of 1-trialkylphenyl cyclic phosphine oxides (*e.g.* **1a**–**c**) entered into reaction with dimethyl acetylenedicarboxylate (DMAD) to afford phosphoranes/ylides (**2a**–**c**) according to an inverse Wittig protocol.[1-3] It is also known that a variety of α-keto-phosphoranes undergo thermolysis to form phosphine oxides and alkynes.[4-8] This transformation is just the reverse of the one involving the interaction of phosphine oxides **1a–c** and DMAD to furnish product **2a–c**.

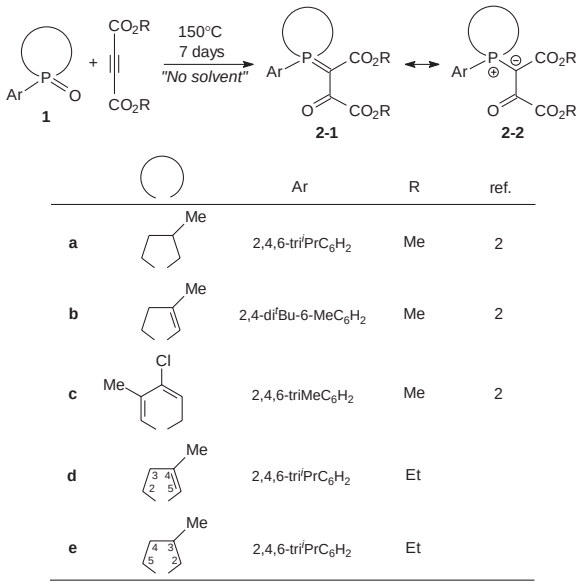
As a further extension of our novel reaction, two additional stabilised phosphonium ylides (**2d** and **2e**) have been synthesised by the reaction of diethyl acetylenedicarboxylate with 2,3-dihydro-1*H*-phosphole oxide **1d** and tetrahydro derivative **1e**, respectively (Scheme 1). From a 7:3 mixture of diastereomers of **1e**, the product (**2e**) was formed in a similar ratio of isomers.

Formation of the stabilised phosphonium ylides (**2**) may involve spirocyclic oxaphosphete intermediates **3** containing the oxygen atom in the equatorial position in the trigonal bipyramid around the phosphorus atom.[9] The oxaphosphetes (**3**) are the unsaturated analogues of the well-known Wittig intermediates.[10] The intermediacy of an azaphosphete has recently been described.[11]

The ^{31}P NMR shifts of 24.0–43.6, the ^{13}C and the ^{1}H NMR spectral data, as well as high level quantum chemical calculations supported the phosphorane/ylide structure of the products (**2**) that are isomers of the oxaphosphete (**3**) intermediates. In this paper, some reactions of the ylides of type **2** are described that are consistent with the β-oxo-α,β-bis(alkoxycarbonyl) moiety of the products (**2**).

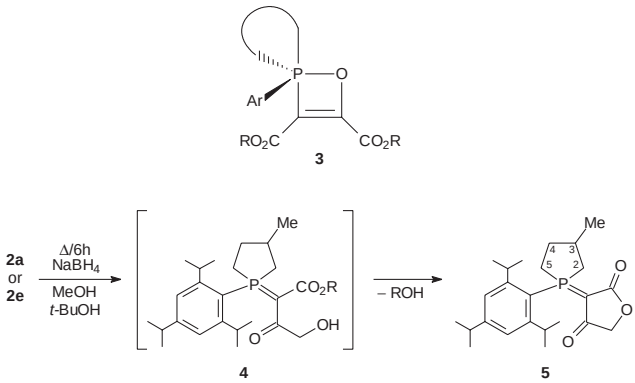
The stabilised phosphonium ylide **2a** was subjected to reduction by an excess of sodium borohydride. A (phospholane-1-ylidene)-furan-2,4-dione (**5**) could be isolated from the mixture in a neat reaction. Starting from a 3:1 mixture of diastereomers of **2a**, product **5** was obtained as a 63:37 mixture of isomers and in 84% yield after purification by chromatography (Scheme 2).

Formation of the keto-lactone ring can be explained by assuming the selective reduction of the β-methoxycarbonyl moiety to a hydroxymethyl group to yield intermediate **4** that is then stabilised by an intramolecular alcoholysis. This kind of reaction is the consequence of the 3-oxo-succinic acid diester moiety of starting compound **2a**. The same product (**5**) was obtained after a similar treatment of the diethyl ester (**2e**) by sodium borohydride showing that the reaction is probably general.



		Ar	R	ref.
a		2,4,6-tri^{i}PrC$_6$H$_2$	Me	2
b		2,4-di^{t}Bu-6-MeC$_6$H$_2$	Me	2
c		2,4,6-triMeC$_6$H$_2$	Me	2
d		2,4,6-tri^{i}PrC$_6$H$_2$	Et	
e		2,4,6-tri^{i}PrC$_6$H$_2$	Et	

Scheme 1



Scheme 2

Aiming at the synthesis of the corresponding oxime, β-keto products **2a** and **2e** were reacted with hydroxylamine hydrochloride at the boiling point of the corresponding alcohol. The result of the reaction was, however, another phosphorane/ylide (**6a** and **6e**, respectively) (Scheme 3), formed by the elimination of the C(O)CO$_2$R moiety of substrate **2**. This fragmentation may be brought about by the nucleophilic attack of hydroxylamine on the β-keto group. Another possibility is that the oxime (**7**) is in fact formed, and

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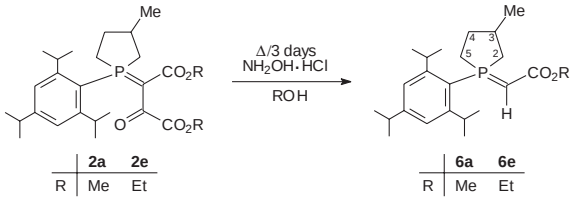
is stabilised subsequently, as shown in Scheme 4. Starting from a *ca* 7:3 isomeric mixture of **2a** or **2e**, the corresponding product (**6a** or **6e**, respectively,) was obtained in a similar ratio.

The reactivity of the keto function is obviously affected by the strong electron delocalisation and by the alkoxycarbonyl group on the carbonyl carbon atom.

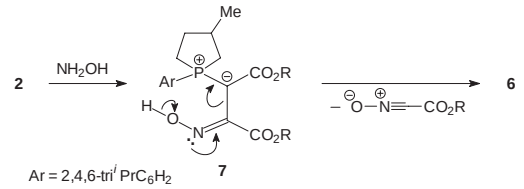
The strong delocalisation also has an effect on the reactivity of the stabilised phosphonium ylides **2** in the Witttig reaction. Thus **2** would not enter into reaction with benzaldehyde. Phosphorane/ylide **6a** underwent, however, the Wittig reaction with benzaldehyde (or with anisaldehyde) to furnish a mixture of isomers of the corresponding cinnamic acid ester **8** and phosphine oxide **1a** (Scheme 5). It is noteworthy that the attempted reaction of **6a** failed with *p*-nitrobenzaldehyde.

All new products (**2d**, **2e**, **5**, **6a** and **6e**) were characterised by ^{31}P, ^{13}C and ^{1}H NMR spectroscopy, as well as HRMS.

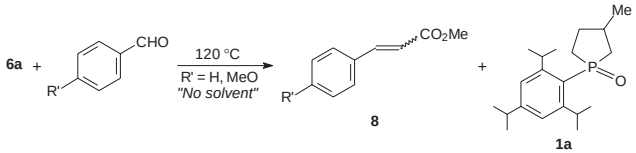
In summary, β-oxo-α,β-bis(alkoxycarbonyl)phosphonium ylides **2** were converted under reductive conditions into an ylidene oxo-lactone (**5**) in a neat reaction. On reaction with hydroxylamine, the stabilised phosphonium ylides (**2**) were transformed into the simpler phosphorane/ylide (**6**) that did undergo the Wittig reaction.



Scheme 3



Scheme 4



Scheme 5

Experimental

The ^{31}P, ^{13}C and ^{1}H NMR spectra were taken on a Bruker DRX-500 spectrometer operating at 202.4, 125.7 and 500 MHz, respectively. Chemical shifts are downfield relative to 85% H_3PO_4 or TMS. The couplings are given in Hz. FAB mass spectrometry was performed on a ZAB-2SEQ instrument. The ratio of the isomers was determined on the basis of relative ^{31}P NMR intensities in all cases.

2-[4-Methyl-1-(2,4,6-triisopropylphenyl)-2,3-dihydro-1H-1λ^5-phosphol-1-ylidene]-3-oxo-succinic acid diethyl ester (**2d**)*:* Prepared from **1d** as described for similar derivatives earlier;[1] Yield: 79% as a thick oil; δ_P ($CDCl_3$) 39.5; δ_C 13.6 (*C*H_3CH$_2$O), 13.9 (*C*H$_3$CH$_2$O), 20.1 (^3J = 17.9, C_4–Me), 23.2 (^4J = 3.8, CH(*C*H$_3$)$_2$), 23.3 (CH(*C*H$_3$)$_2$), 25.0 (CH(*C*H$_3$)$_2$), 25.5 (^1J = 58.9, C_2), 31.3 (^3J = 6.3, *o*-*C*HMe$_2$), 33.8 (*p*-*C*HMe$_2$), 35.2 (^2J = 7.0, C_3), 58.6 (CH$_3$*C*H$_2$O), 60.2 (CH$_3$*C*H$_2$O), 75.9 (^1J = 103.3, C_α), 115.9 (^1J = 88.6, C_5), 122.4 (^3J = 11.4, $C_{3'}$), 122.5 (^1J = 91.7, $C_{1'}$), 152.1 (^2J = 9.0, $C_{2'}$)[a], 152.5 ($C_{6'}$)[a,b], 152.3 ($C_{4'}$)[a,b], 163.2 (^2J = 22.7, C_4), 166.0 (^2J = 14.6, C=O), 167.2 (^2J = 14.9, C=O), 182.4 (^2J = 6.5, C_β), [a,b]may be reversed; δ_H 1.08 (t, $^3J_{HH}$ = 7.1, 3H, C*H*$_3$CH$_2$O), 1.13 (d, $^3J_{HH}$ = 6.5, 6H, CH(C*H*$_3$)$_2$), 1.23 (d, $^3J_{HH}$ = 6.9, 6H, CH(C*H*$_3$)$_2$), 1.30 (t, $^3J_{HH}$ = 7.2, 3H, C*H*$_3$CH$_2$O), 1.34 (d, $^3J_{HH}$ = 6.6, 6H, CH(C*H*$_3$)$_2$), 2.03 (s, 3H, C_4–CH$_3$), 2.84–2.89 (m, 1H, C*H*Me$_2$), 3.49–3.60 (m, 2H, C*H*Me$_2$), 4.03 (q, $^3J_{HH}$ = 7.0, 2H, CH$_3$C*H*$_2$O), 4.23 (q, $^3J_{HH}$ = 7.2, 2H, CH$_3$C*H*$_2$O), 6.28 (d, $^2J_{PH}$ = 27.5, 1H, C_5–*H*), 7.07 (s, 2H, Ar*H*); $(M+H)^+_{found}$ = 489.2642, $C_{28}H_{42}O_5P$ requires 489.2666; IR (film) 1754, 1715, 1669 cm^{-1}.

2-[3-Methyl-1-(2,4,6-triisopropylphenyl)-1λ^5-phospholan-1-ylidene]-3-oxo-succinic acid diethyl ester (**2e**): Prepared from the 7:3 mixture of the isomers of **1e**; Yield: 83% oily product, as a 67:33 mixture of two isomers; $(M+H)^+_{found}$ = 491.2815, $C_{28}H_{44}O_5P$ requires 491.2845; IR (film) 1739, 1714, 1666 cm^{-1}.

2e/*major isomer*: δ_P ($CDCl_3$) ^{31}P NMR ($CDCl_3$) δ 33.0 (67%); ^{13}C NMR ($CDCl_3$) δ 13.6 (*C*H$_3$CH$_2$O), 13.8 (*C*H$_3$CH$_2$O), 19.8 (^3J = 11.5, C_3–Me), 23.3 (CH(*C*H$_3$)$_2$), 24.0 (CH(*C*H$_3$)$_2$), 24.9 (CH(*C*H$_3$)$_2$), 31.5 (^3J = 5.6, *o*-*C*HMe$_2$), 31.7 (^1J = 54.7, C_5), 33.3 (^2J = 7.7, C_4), 33.6 (^1J = 54.8, C_2), 33.8 (*p*-*C*HMe$_2$), 34.2 (^2J = 5.4, C_3), 58.7 (CH$_3$*C*H$_2$O), 60.1 (CH$_3$*C*H$_2$O), 73.1 (^1J = 98.2, C_α), 120.7 (^1J = 85.4, $C_{1'}$), 122.8 (^3J = 11.3, $C_{3'}$), 152.0 ($C_{4'}$), 152.9 (^2J = 11.0, $C_{2'}$), 166.0 (^2J = 14.7, C=O), 166.8 (^2J = 14.5, C=O), 182.6 (^2J = 5.9, C_β); δ_H 1.18 (t, $^3J_{HH}$ = 7.1, 3H, C*H*$_3$CH$_2$O), 1.24 (d, $^3J_{HH}$ = 6.9, 6H, CH(C*H*$_3$)$_2$, 1.25 (d, $^3J_{HH}$ = 7.0, 6H, CH(C*H*$_3$)$_2$), C_3–C*H*$_3$ overlapped by the signals of CH(C*H*$_3$)$_2$, C*H*$_3$CH$_2$O overlapped by the signals of CH(C*H*$_3$)$_2$, 1.32 (d, $^3J_{HH}$ = 6.4, 6H, CH(C*H*$_3$)$_2$), 2.85–2.90 (m, 1H, C*H*Me$_2$), 3.53–3.62 (m, 2H, C*H*Me$_2$), 4.09 (q, $^3J_{HH}$ = 7.1, 2H, MeC*H*$_2$O), 4.23 (q, $^3J_{HH}$ = 7.0, 2H, MeC*H*$_2$O), 7.10 (s, 2H, Ar*H*).

2e/*minor isomer*: δ_P ($CDCl_3$) ^{31}P NMR ($CDCl_3$) δ 32.4 (33%); ^{13}C NMR ($CDCl_3$) δ 13.7 (*C*H$_3$CH$_2$O), 14.1 (*C*H$_3$CH$_2$O), 20.2 (^3J = 15.7, C_3–Me), 23.3 (CH(*C*H$_3$)$_2$), 24.0 (CH(*C*H$_3$)$_2$), 24.9 (CH(*C*H$_3$)$_2$), 27.1 (^1J = 55.0, C_5), 32.1 (C_4), 38.3 (^1J = 55.6, C_2), 72.8 (^1J = 98.8, C_α), 120.5 (^1J = 86.3, $C_{1'}$).

3-[3-Methyl-1-(2,4,6-triisopropylphenyl)-1λ^5-phospholan-1-ylidene]-furan-2,4-dione (**5**): A solution of a *ca* 7:3 isomeric mixture of 1.08 mmol of phosphorane/ylide **2a** (or **2e**) in 30 ml of *t*-butanol was treated with five portions of 0.40 g sodium borohydride in 5 ml of ethanol (in total, 2.0 g (50 mmol) of $NaBH_4$), at 26°C on stirring. Then, the mixture was stirred at the boiling point for 6h and the volatile components were evaporated. The residue was taken up in 50 ml of chloroform and the resulting mixture was treated with the solution of 6 ml of 37% hydrochloric acid in 30 ml of water. The organic phase was separated and dried (Na_2SO_4). The crude product obtained after concentration *in vacuo* was purified by column chromatography (silica gel, 2% methanol in chloroform and 1:1 ethyl-acetate/*n*-hexane as eluants) to give 0.36 g (84%) [or 0.30 g (63%)] of **5** as a thick oil that was a 63–37% (74–26%) mixture of isomers; $(M+H)^+_{found}$ = 403.2382, $C_{24}H_{35}O_3P$ requires 403.2402.

5/*major isomer*: δ_P ($CDCl_3$) 25.8 (63%); δ_C 20.1 (^3J = 12.2, C_3–Me), 23.8 (CH(*C*H$_3$)$_2$), 24.6 (CH(*C*H$_3$)$_2$), 25.4 (CH(*C*H$_3$)$_2$), 30.2 (^1J = 55.0, C_5), 31.3 (^1J = 57.4, C_2), 33.0 (^3J = 5.3, *o*-*C*HMe$_2$), 33.6 (^2J = 7.1, C_4), 34.4 (*p*-*C*HMe$_2$), 34.9 (^2J = 5.6, C_3), 66.0 (^1J = 106.6, C_α), 72.4 (^3J = 13.3, C_γ), 119.0 (^1J = 84.6, $C_{1'}$), 123.5 (^3J = 11.4, $C_{3'}$), 153.7 (^2J = 5.3, $C_{2'}$), 153.8 ($C_{4'}$), 175.6 (^2J = 18.0, C_δ), 194.0 (^2J = 8.9, C_β); δ_H 1.19 (d, $^3J_{HH}$ = 5.7, 3H, C_3–CH$_3$), 1.24 (d, $^3J_{HH}$ = 6.3, 6H, CH(C*H*$_3$)$_2$), 1.27 (d, $^3J_{HH}$ = 7.5, 6H, CH(C*H*$_3$)$_2$), 1.36 (d, $^3J_{HH}$ = 6.3, 6H, CH(C*H*$_3$)$_2$), 2.90–2.93 (m, 1H, C*H*Me$_2$), 3.43–3.49 (m, 2H, C*H*Me$_2$), 4.35 (s, 2H, COC*H*$_2$O), 7.15 (s, 2H, Ar*H*).

5/minor isomer: δ_P 32.0 (37%); δ_C 20.3 (^3J = 15.6, Me), 23.8 (CH(*C*H$_3$)$_2$), 24.6 (CH(*C*H$_3$)$_2$), 25.3 (CH(*C*H$_3$)$_2$), 30.2 (^1J = 55.0, C_5), 33.0 (^3J = 5.3, *o*-*C*HMe$_2$), 34.2 (^1J = 9.6, C_3), 34.4 (*p*-*C*HMe$_2$), 37.1 (^2J = 56.0, C_2), 65.9 (^1J = 106.7, C_α), 72.4 (^3J = 13.4, C_γ), 119.1 (^1J = 84.8, $C_{1'}$), 123.5 (^3J = 11.8, $C_{3'}$), 153.7 (^2J = 5.3, $C_{2'}$), 153.8 ($C_{4'}$), 175.6 (^2J = 18.1, C_δ), 194.0 (^2J = 5.0, C_β).

*General procedure for the preparation of phosphoranes/ylides **6a** and **6e**:* 2.0 g (28.8 mmol) of hydroxylamine hydrochloride was added to a solution of a *ca* 7:3 isomeric mixture of 2.55 mmol of phosphorane/ylide **2a** or **2e** in 20 ml of methanol or ethanol, respectively, and the mixture was stirred at the boiling point for 3 or 2 days, respectively. The solvent was evaporated and the residue extracted with 30 ml of chloroform to give the products (**6a** or **6e**) as oils, in a purity of *ca* 95% according to ^{31}P NMR.

[3-Methyl-1-(2,4,6-triisopropylphenyl)-1λ^5-phospholan-1-ylidene]-acetic acid methyl ester (**6a**): Yield: 95% oily product, as a 77:23 mixture of two isomers; $(M+H)^+_{found}$ = 377.2591, $C_{23}H_{38}O_2P$ requires 377.2609; IR (film) 1735 cm^{-1}.

6a/major isomer: δ_P ($CDCl_3$) 40.7 (77%); δ_C 19.0 (^3J = 14.6, C_3–Me), 23.4 (CH(*C*H$_3$)$_2$), 24.5 (CH(*C*H$_3$)$_2$), 25.6 (CH(*C*H$_3$)$_2$), 30.0 (^1J = 48.8, C_5), 33.0 (^2J = 5.6, C_4), 33.5 (^3J = 4.7, *o*-*C*HMe$_2$), 34.3 (*p*-*C*HMe$_2$), 35.0 (^1J = 46.4, C_2), 36.1 (^2J = 6.9, C_3), 53.3 (MeO), 52.9 (^1J = 47.0, C_α), 112.6 (^1J = 75.7, $C_{1'}$), 124.4 (^3J = 11.8, $C_{3'}$), 154.7

(^2J = 11.4, $C_{2'}$), 155.9 ($C_{4'}$), 165.8 (^2J = 3.0, C=O); δ_H 1.28 (d, $^3J_{HH}$ = 6.9, 6H, CH(CH_3)$_2$), 1.32 (d, $^3J_{HH}$ = 6.6, 6H, CH(CH_3)$_2$), 1.35 (d, $^3J_{HH}$ = 6.1, 6H, CH(CH_3)$_2$), 1.40 (d, $^3J_{HH}$ = 6.0, 3H, C_3–CH_3), 2.93–2.96 (m, 1H, C*H*Me$_2$), 3.13–3.16 (m, 2H, C*H*Me$_2$), 3.94 (s, 3H, CH_3O), 7.21 (s, 2H, Ar*H*).

6a/*minor isomer*: δ_P δ 40.6 (23%); δ_C 20.1 (^3J = 15.1, C_3–Me), 23.4 (CH(*C*H$_3$)$_2$), 24.6 (CH(*C*H$_3$)$_2$), 25.5 (CH(*C*H$_3$)$_2$), 28.5 (^1J = 51.5, C_5), 33.5 (^3J = 4.7, o-*C*HMe$_2$), 34.3 (p-*C*HMe$_2$), 36.5 (^1J = 50.0, C_2), 53.5 (MeO), 112.7 (^1J = 74.8, $C_{1'}$), 124.4 (^3J = 11.5, $C_{3'}$), 154.7 (^2J = 11.0, $C_{2'}$), 164.5 (^2J = 3.0, C=O).

[3-Methyl-1-(2,4,6-triisopropylphenyl)-1λ^5-phospholan-1-ylidene]-acetic acid ethyl ester (**6e**): Yield: 95%, as a 72:28 mixture of two isomers; $(M+H)^+_{found}$ = 391.2745, $C_{24}H_{40}O_2P$ requires 391.2766; IR (film) 1732 cm^{-1}.

6e/*major isomer*: δ_P ($CDCl_3$) ^{31}P NMR ($CDCl_3$) δ 41.0 (72%); ^{13}C NMR ($CDCl_3$) δ 13.8 (*C*H$_3$CH$_2$O), 18.9 (^3J = 14.5, C_3–Me), 23.3 (CH(*C*H$_3$)$_2$), 24.3 (CH(*C*H$_3$)$_2$), 25.4 (CH(*C*H$_3$)$_2$), 29.9 (^1J = 48.6, C_5), 32.9 (^2J = 5.2, C_4), 33.3 (^3J = 4.2, *o*-*C*HMe$_2$), 34.2 (*p*-*C*HMe$_2$), 34.9 (^1J = 52.0, C_2), 35.8 (^2J = 6.5, C_3), 62.5 (CH$_3$*C*H$_2$O), 61.7 (^1J = 49.0, C_α), 112.4 (^1J = 76.2, $C_{1'}$), 124.2 (^3J = 11.6, $C_{3'}$), 154.6 (^2J = 11.2, $C_{2'}$), 155.7 ($C_{4'}$), 165.2 (^2J = 3.0, C=O); δ_H 1.18–1.43 (m, 24H, 3xCH(CH_3)$_2$, CH_3CH$_2$C$_3$CH$_3$), 2.91–3.00 (m, 1H, C*H*Me$_2$), 3.08–3.18 (m, 2H, C*H*Me$_2$), 4.22–4.32 (m, 2H, CH$_2$O), 7.21 (s, 2H, Ar*H*).

6e/*minor isomer*: ^{31}P NMR ($CDCl_3$) δ 40.8 (28%); ^{13}C NMR ($CDCl_3$) δ 13.9 (*C*H$_3$CH$_2$O), 20.0 (^3J = 14.9, C_3-Me), 23.3 (CH(*C*H$_3$)$_2$), 24.4 (CH(*C*H$_3$)$_2$), 25.3 (CH(*C*H$_3$)$_2$), 30.6 (^1J = 49.3, C_5), 33.3 (^3J = 4.2, *o*-*C*HMe$_2$), 34.2 (*p*-*C*HMe$_2$), 36.4 (^1J = 49.0, C_2), 62.5 (CH$_3$*C*H$_2$O), 112.5 (^1J = 75.0, $C_{1'}$), 124.2 (^3J = 11.6, $C_{3'}$), 154.6 (^2J = 11.0, $C_{2'}$), 164.2 (^2J = 3.0, C=O).

Wittig reaction of phosphorane/ylide **6a** *with aldehydes*: A mixture of 0.1 g (0.25 mmol) of phosphorane/ylide **6a** and 0.3 ml (2.5 mmol) of benzaldehyde was heated in a sealed tube for 7 days. The excess of benzaldehyde was removed *in vacuo* and the residue was passed through a short column (silica gel, 3% methanol in chloroform) to give 0.30 g of an oil containing ~50% of **8** (R' = H) and ~50% of **1a** according to GC–MS. **7** (R' = H) consisted of a 3:1 mixture of the *trans* and *cis* isomers $(M+H)^+_{found}$ = 163.0750, $C_{10}H_{11}O_2$ requires 163.0759), while **1a** was a 2:1 mixture of two diastereomers (δ_P 59.9 and 60.6; M^+ = 320). A similar reaction of **4a** and anisaldehyde provided **8** (R' = MeO) as a 3:1 mixture of the *trans*:*cis* geometrical isomers (M^+ = 192).

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References

1 Gy. Keglevich, H. Forintos, T. Körtvélyesi and L. Töke, *J. Chem. Soc., Perkin Trans. 1*, 2002, 26.
2 Gy. Keglevich, T. Körtvélyesi, H. Forintos, Á. Gy. Vaskó, V. Izvekov and L. Töke, *Tetrahedron*, 2002, **58**, 3721.
3 Gy. Keglevich, *Curr. Org. Chem.* 2002, **6**, 891.
4 R.A. Aitken, H. Herion, A. Janosi, S.V. Raut, S. Seth, I.J. Shannon and F.C. Smith, *Tetrahedron Lett.*, 1993, **34**, 5621.
5 R.A. Aitken, H. Herion, A. Janosi, N. Karodia, S.V. Raut, S. Seth, I.J. Shannon and F.C. Smith, *J. Chem. Soc., Perkin Trans. 1*, 1994, 2467.
6 R.A. Aitken, N. Karodia and P. Lightfoot, *J. Chem. Soc., Perkin Trans. 2*, 2000, 333.
7 R.A. Aitken, N.A. Al-Awadi, M.E. Balkovich, H.J. Bestmann, O. Clem, S. Gibson, A. Groß, A. Kumar and T. Röder, *Eur. J. Org. Chem.*, 2003, 840.
8 R.A. Aitken and A.W. Thomas, in *Chemistry of the Functional Groups, Supplement A3*, ed. S. Patai, Wiley, Chichester, 1997, pp.473–536.
9 Gy. Keglevich, T. Körtvélyesi, H. Forintos and S. Lovas, *J. Chem. Soc., Perkin Trans. 2*., 2002, **10**, 1645.
10 E. Vedejs and C.F. Marth, In *Phosphorus-31 NMR Spectral Properties in Compound Characterization and Structural Analysis*; L.D. Quin and J.G. Verkade, Eds.; VHC, New York, 1994; Ch. 23, p. 297.
11 T. Uchiyama, T. Fujimoto, A. Kakehi and I. Yamamoto, *J. Chem. Soc., Perkin Trans. 1*., 1999, 1577.