Organic Chemistry

Regioselectivity in the C-alkylation of triethyl 3-methyl-4-phosphonobut-2-enoate

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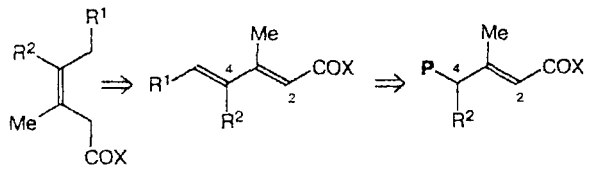
The reaction of triethyl 3-methyl-4-phosphonobut-2-enoate (**1**) with three alkyl halides AlkX (Alk = Pr^i, $Me_2CHCH_2CH_2$, and $c\text{-}C_5H_9$; X = Br, I) in the system KOH(solid)—DMF—Bu^n_4NBr at ~20 °C gives exclusively products of alkylation at C(2) with Δ^2 and/or Δ^3 position of the double bond. Under the same conditions, the reaction of **1** with MeI gives a mixture of products with different substitution patterns. Only the use of an ion pair extraction technique affords 2-methyl-Δ^2-products selectively, albeit in rather moderate yields. The Horner—Emmons olefination of PhCHO with the resulting phosphonates gives ethyl 2-alkyl-3-methyl-5-phenylpenta-2,4-dienoates in high yields.

Key words: triethyl 3-methyl-4-phosphonobut-2-enoate, alkylation; phase transfer catalysis; Horner—Emmons reaction; regioselectivity.

In a continuation of our studies on the use of the Horner—Emmons reaction for the stereocontrolled synthesis of derivatives of 3-methylalka-2,4-dienoic acids (see the review, Ref. 1) and on stereospecific transformation of the latter into *Z*-trisubstituted olefins with a homoallylic type of functional substitution,[2] we intended to use this reaction sequence for the stereospecific construction of trisubstituted double bonds. For this purpose, we needed alkyl 4-alkyl-3-methylalka-2,4-dienoates (as substrates for stereospecific 1,4-*cis*-hydrogenation) and, correspondingly, alkyl 3-methyl-4-phosphonobut-2-enoates.



X = OAlk, NR_2; R^1 = Alk, Ar; R^2 = Alk,
P — phosphonate group

In this communication, we report an attempt to obtain phosphonates of the type specified above by direct C-alkylation of the readily accessible triethyl 3-methyl-4-phosphonobut-2-enoate (**1**) under conditions of phase transfer catalysis. The results of this reaction demonstrate its high regioselectivity, the reaction occurring as an electrophilic attack almost exclusively at the C(2) atom of ester **1**.

It is known that simple phosphonates of the type $(AlkO)_2P(O)CH_2X$ (where X is an electron-withdrawing substituent) form C-alkylation products in both traditional variants of carbanionic reactions[3—5] and under conditions of phase transfer catalysis (using the ion-pair

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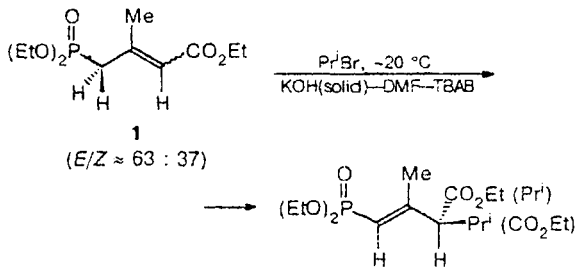
extraction technique, which requires an equimolar amount of the phase transfer catalyst).[6–8] It is also known that under both classical conditions of carbanion generation[9,10] and under conditions of phase transfer catalysis,[11] unsaturated ambident CH-acids of the type $MeC(R)=CHCOR^1$ (where R = H, Alk; R^1 = OAlk, Me) undergo alkylation with electrophiles R^2X in low-polarity media preferentially at the α-position with respect to the carbonyl group to give products of the type $CH_2=C(R)-CH(R^2)COR^1$ and $MeC(R)=CR^2COR^1$, the equilibrium between which depends on a number of factors. This direction of the alkylation is in agreement with the data of quantum-chemical calculations.[12,13]

The methylene moiety at position 4 in phosphonate **1** and in other allylic phosphonates of similar structure is activated both by the adjacent phosphonate group and by the vinylogous group CO_2Et. It is known[14,15] that irrespective of the ratio of the *E*- and *Z*-isomers in phosphonate **1** the action of strong bases rapidly transforms it into a delocalized carbanion (an equilibrium mixture of *E*- and *Z*-isomers), which reacts with aldehydes exclusively through the anionic pole at C(4). In the absence of quantum-chemical calculation data, it was of interest to check experimentally the possibility of C-alkylation of allylic phosphonate **1** at position 4.

In order to carry out the reaction of ester **1** with alkyl halides under conditions of phase transfer catalysis, we tested three variants of the two-phase catalytic system solid base—DMF—tetrabutylammonium bromide (TBAB) using K_2CO_3, LiOH, and KOH as the base. Isopropyl bromide was used as the standard AlkX.

In the systems K_2CO_3(solid)—DMF—TBAB and LiOH(solid)—DMF—TBAB at ~20 °C, the ester **1** virtually does not react with Pr^iBr. However, when the system KOH(solid)—DMF is used with an equimolar ratio of the reagents and in the presence of a catalytic amount of TBAB (at ~20 °C), the reaction is completed in 1 h. Judging by GLC and ^1H NMR spectroscopic data, the resulting product is a mixture of two stereoisomers in a ~85 : 15 ratio. The type of its ^1H NMR spectrum agrees with the valence structure of a vinylic phosphonate (**2a**), in which the isopropyl group is located at the C(2) atom of the original substrate. The signal of the proton at the C(4) atom, which is easy to identify from the spin-coupling constants of the H and P nuclei (d, 1 H, J = 16.5 Hz), is observed at δ 5.50 for the major stereoisomer and at δ 5.40 for the minor stereoisomer, whereas in the spectrum of the original ester **1** (E : Z ~ 63 : 37), the signals of the C(4) methylene group resonate at δ 2.60 (*E*-isomer) and 3.40 (*Z*-isomer) and the spin-spin coupling constant of the H and P nuclei is typical of a sp^3-hybridized C atom (J = 23 Hz).[16]

Whereas the signal of the olefinic proton in the spectrum of the original ester **1** is observed at δ 5.71 and has an "allylic" coupling constant (J = 2 Hz), the signal of the proton at C(2) in the spectrum of ester **2a** is a doublet with δ 2.57 and a "vicinal" coupling constant (J = 10.5 Hz). The data obtained from ^1H NMR spectroscopy are insufficient for an unambiguous assignement of the configurations of the major and minor stereoisomers. One can assume that the signal of the allylic Me group of the major isomer (δ 1.98) is shifted downfield relative to the corresponding signal of the minor isomer (δ 1.80), since in the former case the deshielding effect of the O atom in the *cis*-positioned phosphoryl group is exhibited. The vinylic proton of the major isomer is not located within the zone of diamagnetic shielding exhibited by the carbonyl group, and its signal is therefore shifted downfield with respect to the similar signal of the minor isomer. Both effects are in agreement with the assumed *E*-configuration of the major isomer.



1
($E/Z \approx 63 : 37$)

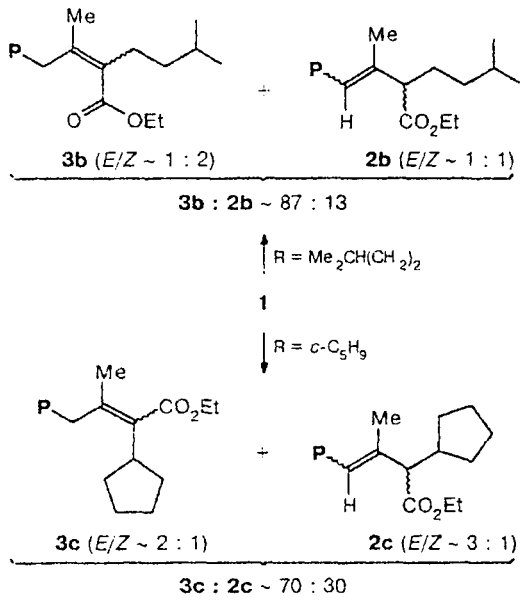
2a
(major isomer?)

Under the same conditions, isopropyl iodide reacts with ester **1** to give also a binary mixture of stereoisomers of vinylic phosphonate **2a** in a yield of 46%.

The reaction of phosphonate **1** with isopentyl bromide and cyclopentyl bromide in the same heterogeneous system also afforded only the products of monoalkylation at the C(2)-atom, although the qualitative composition of the products was different as compared with the binary mixture of stereoisomers **2a**.

In the former case, a mixture of monoalkylated phosphonates (elemental analysis data), was obtained in 57% yield; it contained, judging by ^1H NMR spectroscopic data, conjugated [(*E*)-**3b** and (*Z*)-**3b**] and nonconjugated products of alkylation at the C(2) atom [(*E*,*Z*)-**2b**] in a ~87 : 13 ratio. The spectrum did not contain a signal around δ 5.7 typical of an olefinic proton at the α-position to the alkoxycarbonyl group, which suggests that products of alkylation at C(4) are not formed in this case, either. The ratio of integral intensities of the signals from the proton at C(4) and the methyl at C(3) (presumably, δ 3.15 and 1.88 for (*Z*)-**3b**, and 2.60 and 2.0 for (*E*)-**3b**, respectively) indicates that the conjugated *Z*-isomer, (*Z*)-**3b**, is the dominating component of the mixture. The ratio of stereoisomers in the minor component (*E*,*Z*)-**2b** is close to 1 : 1, as judged by the intensity of signals of the olefinic proton at C(4) (δ 5.35 and 5.45, J_{H-P} = 14 Hz).

In the latter case, judging by GLC and ^1H NMR data, the isolated reaction product (yield 44%) is a mixture of both structural isomers (**2c** and **3c**), in which the allylic phosphonate **3c** predominates; the ratio of



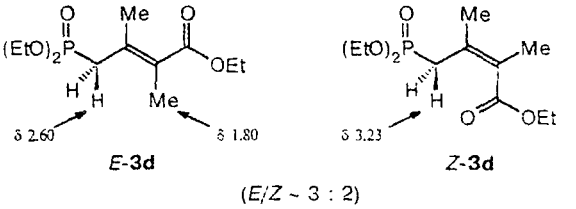
P = $(EtO)_2P(O)$

Reagents and conditions: RBr, KOH(solid)—DMF—TBAB, ~20 °C.

E- and *Z*-stereoisomers in the latter is close to 2 : 1 (Table 1). The fraction of vinylic phosphonate **2c** is ~30% of the overall weight of the monoalkylation products, while the ratio of *E*- and *Z*-stereoisomers in it is ~3 : 1.

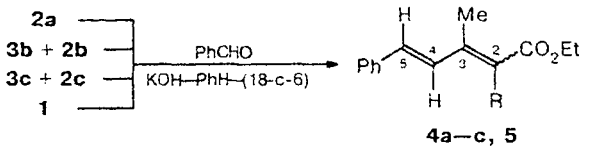
The results of alkylation of ester **1** with one primary ($Me_2CHCH_2CH_2Br$) and two secondary alkyl halides (Me_2CHX and *cyclo*-C_5H_9Br) allow one to suggest that the prototropic equilibrium between phosphonates of the types **2** and **3** is more strongly shifted towards the latter the weaker the steric congestion created by the substituent entering at position C(2). These results are consistent with the data on the prototropic equilibrium[17,18] and C-alkylation of monofunctional allylic and vinylic phosphonates.[18]

Methylation of phosphonate **1** with methyl iodide in the chosen system, KOH(solid)—DMF—TBAB, at both ~20 °C and 0—5 °C, affords multicomponent mixtures of mono- and bis-alkylation products, which could not be identified either in the resulting mixture of C-methylated phosphonates or from the ^1H NMR spectrum of the dienes formed upon the reaction of a mixture of these phosphonates with benzaldehyde (see below). An attempt to perform selective methylation of ester **1** with MeI in the presence of an equimolar amount of sodium hydride in 1,2-diethoxyethane (DEE), by analogy with the known procedure,[3] proved unsuccessful: after 12 h of exposure at 30 °C, ~65% of the original ester **1** was recovered. Prolonged methylation of ester **1** under conditions of ion pair extraction[19] resulted only in a partial conversion of the original substrate. In the ^1H NMR spectrum of a mixture of compound **1** with methylation products, which was difficult to separate, we were able to reveal signals corresponding unambiguously to one of the possible structures, *viz.*, **3d**: the methyl group at C(2) (singlet, δ 1.80), the allylic methylene group in the *E*-isomer of **3d** (a doublet at δ 2.60 with J_{P-H} = 23 Hz), and the same group in the *Z*-isomer of **3d** (a doublet at δ 3.23 with J_{P-H} = 23 Hz). However, the integral intensity of these signals is rather low in comparison with the signals for the original ester **1**, and corresponds to a ~30% conversion of the substrate.



(*E*/*Z* ~ 3 : 2)

The resulting alkylated phosphonates of the types **2** and **3** (or their mixtures) were then used in the Horner—Emmons reaction with benzaldehyde under conditions of phase transfer catalysis developed previously.[15] Since both types of phosphonates form the same delocalized carbanion in the presence of bases,[14,15] all of the alkyl 2-alkyl-3-methyl-5-phenylpenta-2,4-dienoates (**4a—c**) obtained by this procedure turned out to be structurally uniform, as follows from their ^1H NMR spectra (Table 2; the spectrum of diene[5] obtained by the reaction of phosphonate **1** with PhCHO is given for comparison). The high yields of dienes **4a—c** make the two-step sequence of transformations **1** → **2/3** → **4** a convenient procedure for synthesizing conjugated dienes.



4: R = Pr^i (**a**), $Me_2CH(CH_2)_2$ (**b**), *c*-C_5H_9 (**c**)
5: R = H

The reaction of a phosphonate mixture **1**+**3d** (the product isolated after methylation of ester **1** by ion pair extraction) with benzaldehyde under the conditions speci-

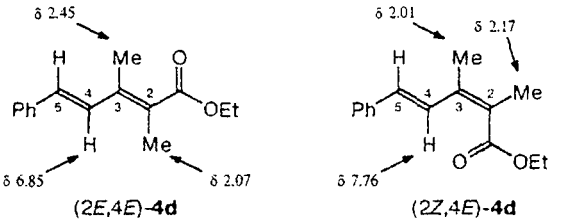
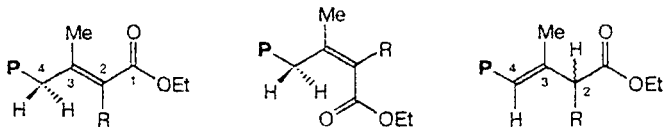


Table 1. ^{1}H NMR spectral data for the starting phosphonate **1** and its monoalkylation products



(*E*)-**1**, (*E*)-**3** (*Z*)-**1**, (*Z*)-**3** (*E*)-**2**

P = $(EtO)_2P(O)$

Phosphonate (R)	δ (*J*/Hz) 2-H	4-H	3-Me	OCH_2Me	R	Signal assignment	Isomer ratio
1 (H)		2.60 (d, *J* = 23)[a]	2.23 (d, *J* = 2)		—	(*E*)-**1**	
	5.71 (q, *J* = 2)			1.15—1.30 (m, 9 H)[b]; 3.95—4.15 (m, 6 H)[c]			*E* : *Z* ≈ 63 : 37
		3.40 (d, *J* = 23)[a]	1.96 (d, *J* = 2)		—	(*Z*)-**1**	
2a (Pr^i)		5.50 (d, *J* = 16.5)	1.98 (d, *J* = 2)	1.15—1.25 (m, 9 H)[b]		(*E*)-**2a**	
	2.57 (d, *J* = 10.5)				0.84+0.90 (both d, *J* = 6.7; J_{AB} = 20.5); 2.05 (hept, *J* = 6.7)[d]		*E* : *Z* ≈ 85 : 15
		5.40 (d, *J* = 16.5)	1.80 (d, *J* = 1.5)	3.85—4.10 (m, 6 H)[c]		(*Z*)-**2a**	
3b+2b ($(CH_2)_2CHMe_2$)	—	3.15 (d, *J* = 25)	1.88 (d, *J* = 2)		0.83 (br.d, 6 H, *J* = 6.7)	(*Z*)-**3b**	
	—			1.15—1.30 (m, 9 H)[b]	1.05—1.20 (m, 2 H)		(*E*)-**3b** : (*Z*)-**3b** : : (*E*,*Z*)-**2b** ≈ ≈ 28 : 59 : 13
		2.60 (d, *J* = 25)	2.0 (d, *J* = 2)	3.89—4.15 (m, 6 H)[c]	1.45 (hept, *J* = 6.7); 2.7—2.8 (m, ~0.9 H)	(*E*)-**3b**	
	[e]	5.35+5.45 (both d, *J* = 14.5)	1.78 (br.s)		1.4—1.7 (m, ~0.1 H)	(*E*,*Z*)-**2b**	
3c+2c ($c\text{-}C_5H_9$)	—	2.58 (d, *J* = 29)	2.03 (d, *J* = 2)			(*E*)-**3c**	
				1.10—1.25 (m, 9 H)[b]; 3.90—4.15 (m, 6 H)[c]	1.35—1.75 (m, 8 H); 2.25 (m, 1 H)[f]		(*E*)-**3c** : (*Z*)-**3c** : : (*E*)-**2c** : (*Z*)-**2c** ≈ ≈ 28 : 59 : 13
	—	2.73 (d, *J* = 29)	1.82 (d, *J* = 2)			(*Z*)-**3c**	
		5.50 (d, *J* = 20.5)				(*E*)-**2c**	
	2.69 (d, *J* = 11)		1.85 (d, *J* = 2)				
		5.38 (d, *J* = 17)				(*Z*)-**2c**	

Note. The compositions of isomer mixtures were determined from the ratio of integral intensities of diagnostic signals of protons at the C(2) and C(4) atoms and of those of the methyl group at C(3); in the case of compound **2**, the ratio found by this method equals the ratio of *E*- and *Z*-isomers determined by GLC (*cf.* Ref. 9).

[a] The overall integral intensity corresponds to 2H. [b] Superposition of three triplets with *J* ≈ 6.5 Hz. [c] Superposition of three quartets with *J* ≈ 6.5 Hz. [d] Doubled heptet with an additional coupling constant *J'* ≈ 10.5 Hz. [e] Overlapping of two doublets with *J* = 14.5 Hz (integral intensity ratio ≈ 1 : 1). [f] Doubled quintet with an additional coupling constant *J'* ≈ 11 Hz.

Table 2. ^{1}H NMR spectra of the products of olefination of PhCHO with phosphonates **1**, **2**, and **3**

Diene (R)	δ (*J*/Hz) 2-H	4-H	5-H	3-Me	OCH_2Me	R	Ph	Signal-assign-ment	Isomer ratio
5 (H)	5.90 (br.s)	5.85 (d, J_{AB} = 17)	6.90 (d, J_{AB} = 17)	2.4 (d, J = 1.2)				2*E*,4*E*	
					4.22 (q, 2 H); 1.3 (t, 3 H)		7.25—7.51 (m, 5 H)		2*Z* : 2*E* ≈ ≈ 15 : 85
	5.75 (br.s)	7.84 (d, J_{AB} = 17)	6.87 (d, J_{AB} = 17)	2.11 (d, J = 1.2)				2*Z*,4*E*	
4a (Pr^i)	—	7.23 (d, J_{AB} = 18)	6.75 (d, J_{AB} = 18)	1.98 (s)		1.20 (d, J = 6.5); 3.24 (hept, J = 6.5)		2*Z*,4*E*	
					1.35 (t, 3 H); 4.24—4.40 (q, 2 H)[a]		7.3—7.5 (m, 5 H)		2*Z* : 2*E* ≈ ≈ 85 : 15
	—	6.94 (d, J_{AB} = 18)	6.68 (d, J_{AB} = 18)	2.02 (s)		1.18 (d, J = 6.5); 3.0 (hept, J = 6.5)		2*E*,4*E*	
4b (*c*-C_5H_9)	—	7.25 (d, J_{AB} = 18)	6.75 (d, J_{AB} = 18)	1.98 (s)		3.24 (qt, J = 6.5)		2*Z*,4*E*	
					1.25—1.4 (m, 3 H)[b]; 4.2—4.3 (m, 2 H)[a]	1.5—1.9 (m, 8 H)	7.3—7.5 (m, 5 H)		2*Z* : 2*E* ≈ ≈ 80 : 20
	—	6.95 (d, J_{AB} = 18)	6.67 (d, J_{AB} = 18)	2.03 (s)		3.24 (qt, J = 6.5)		2*E*,4*E*	
4c ($Me_2CH(CH_2)_2$)	—	7.25 (d, J_{AB} = 16.5)	6.83 (d, J_{AB} = 16.5)	2.16 (s)		2.57 (t, J = 6.5)		2*E*,4*E*	
					1.35 (t, 3 H); 4.25 (m, 2 H)[a]	0.95 (d, J = 6.5); 1.4 (m, 2 H); 1.65 (m, 1 H)	7.3—7.5 (m, 5 H)		2*Z* : 2*E* ≈ ≈ 12 : 88
	—	7.53 (d, J_{AB} = 16.5)	6.65 (d, J_{AB} = 16.5)	2.05 (s)		2.47 (t, J = 6.5)		2*Z*,4*E*	

Note. The compositions of isomer mixtures were determined from the ratio of integral intensities of diagnostic signals of protons at the C(4) and C(5) atoms, of the C(3)-methyl group, and of the alkyl group nearest to C(2) (allylic moiety).
[a] The overall integral intensity of overlapping quartets with $J \approx 7$ Hz. [b] The integral intensity of two overlapping triplets with $J \approx 7$ Hz.

fied above afforded a mixture of diene **5** with both stereoisomers of diene **4d**, which was difficult to separate. Its chromatographic purification gave a sample of diene **4d** of about 90—95% purity. From comparison of the integral intensities of methyl group singlets in the ^{1}H NMR spectrum of this sample it is evident that stereoisomers (2*E*,4*E*)-**4d** and (2*Z*,4*E*)-**4d** are present in the ratio of ~3 : 2.

The yield of the sample of compound **4d** obtained from phosphonate **1** was 21% (over the two stages).

Experimental

GLC analyses were carried out on an LKhM-8 MD-5 instrument with a flame ionization detector using nitrogen as the carrier gas and a glass column (1.4×0.003 m) with 5% SE-30 on chromaton N-AW-DMCS. ^{1}H NMR spectra were obtained on a Bruker WM-250 instrument (250 MHz) for solutions in $CDCl_3$.

The starting phosphonate 1 was obtained by the procedure reported previously.[15]

Reaction of phosphonate 1 with RHal (R — isopropyl, cyclopentyl, isopentyl). Phosphonate **1** (2.64 g, 10 mmol) and then RHal (R = isopropyl, isopentyl, cyclopentyl; Hal = Br, I, see the text) (12 mmol) were added to a stirred mixture of powdered KOH (1.2 g) and TBAB (0.1 g) in DMF (10 mL). The resulting reaction mixture was stirred for 1—2 h at ~20 °C. When the reaction was completed (GLC monitoring of disappearance of the starting phosphonate **1**), the mixture was poured into cold water and extracted with ether. The ethereal extracts were washed twice with water and dried ($MgSO_4$). The solvent was evaporated, and the product was isolated by vacuum distillation or used in the next stage without additional purification. The ^1H NMR spectra are presented in Table 1.

Compound **2a** (R = Pr^i), yield 46%; b.p. 137—139 °C (0.4 Torr); n_D^{20} 1.4550. **2b+3b** (R — isopentyl), mixture ~13 : 87, yield 57%; b.p. 155—160 °C (0.5 Torr); n_D^{20} 1.465. Found (%): C, 57.33; H, 9.17; P, 8.88. $C_{16}H_{31}O_5P$. Calculated (%): C, 57.48; H, 9.33; P, 9.26. **2c+3c** (R — cyclopentyl), mixture ~1 : 1, yield 42%; b.p. 160—170 °C (0.5 Torr); n_D^{20} 1.4720.

Reaction of phosphonate 1 with MeI. A solution of phosphonate **1** (2.6 g, 10 mmol) and MeI (0.72 mL, 12 mmol) in CH_2Cl_2 (40 mL) was added to a solution of TBAB (3.2 g, 10 mmol) in 0.5 *N* aqueous NaOH (20 mL). The mixture was stirred for 3 h at 35 °C. The organic layer was separated, washed with water, and dried with $MgSO_4$. Distillation at 126— 130°C (0.7 Torr) gave a product (1.8 g) as a mixture of phosphonates **1** and **3d** (~68 : 32, according to GLC and ^1H NMR data).

Reaction of phosphonates 2 and 3 (or their mixtures) with PhCHO. To a mixture of a phosphonate (10 mmol), powdered KOH (1.1 g), and 18-crown-6-ether (0.2 g) in dry benzene (30 mL) PhCHO (1.06 g, 10 mmol) was added dropwise with stirring at ~20 °C, and the reaction mixture was stirred for 2—3 h. After the reaction was completed (GLC monitoring of disappearance of the starting phosphonate **1**), the mixture was poured into ice-cold water (20 mL) and extracted with benzene. The combined organic extract was washed with water and dried ($MgSO_4$). The solvent was evaporated, and the resulting dienes **4a—d** were purified on a column with Al_2O_3 using hexane as the eluent. The ^1H NMR spectra are presented in Table 1. Compound **4a** (R = Pr^i), yield 90%; n_D^{20} 1.5600; **4b** (R — cyclopentyl), yield 95%; n_D^{20} 1.5718; **4c** (R — isopentyl), yield 94%; n_D^{20} 1.5590.

The reaction of phosphonate 1 with PhCHO was carried out as reported previously[15] to give compound **5** (R = H), yield 74%; b.p. 132—136 °C (0.8 Torr); n_D^{20} 1.6094.

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