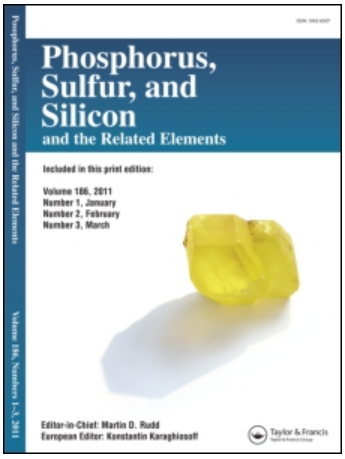


Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

OLEFIN SYNTHESIS VIA THE LITHIUM DERIVATIVES OF THE N,N,N,N'-TETRAMETHYLDIAMIDES OF ARYLMETHANEPHOSPHONIC ACIDS. 2. SYNTHESIS OF SOME (Z)-ORTHO-AND PARA-SUBSTITUTED STILBENES

Jordanka Petrova[a]; Snejana Momchilova[a]; Marko Kirilov[a]
[a] Faculty of Chemistry, University of Sofia, Sofia, Bulgaria

To cite this Article Petrova, Jordanka , Momchilova, Snejana and Kirilov, Marko(1985) 'OLEFIN SYNTHESIS VIA THE LITHIUM DERIVATIVES OF THE N,N,N,N'-TETRAMETHYLDIAMIDES OF ARYLMETHANEPHOSPHONIC ACIDS. 2. SYNTHESIS OF SOME (Z)-ORTHO-AND PARA-SUBSTITUTED STILBENES', Phosphorus, Sulfur, and Silicon and the Related Elements, 24: 3, 243 — 250
To link to this Article: DOI: 10.1080/03086648508074235
URL: http://dx.doi.org/10.1080/03086648508074235

Phosphorus and Sulfur, 1985, Vol. 24, pp. 243–250
0308-664X/85/2403-0243/$15.00/0

OLEFIN SYNTHESIS VIA THE LITHIUM DERIVATIVES OF THE N,N,N′,N′-TETRAMETHYLDIAMIDES OF ARYLMETHANEPHOSPHONIC ACIDS. 2. SYNTHESIS OF SOME (Z)-ORTHO- AND PARA-SUBSTITUTED STILBENES

JORDANKA PETROVA, SNEJANA MOMCHILOVA and MARKO KIRILOV*

Faculty of Chemistry, University of Sofia, 1 Anton Ivanov Av., 1126 Sofia, Bulgaria

(*Received February 19, 1985*)

The reaction of the lithium derivatives of N,N,N′,N′-tetramethyldiamides of arylmethanephosphonic acids (**1**-Li) with ortho- and para-substituted benzaldehydes **2** is studied. Reaction conditions are found for erythro-stereoselective addition of **1** to **2** with high diastereomeric purity (95–99%) of the erythro adducts **3**, **4** (yields 35–72%). By thermolysis of the adducts in neutral medium the corresponding (Z)-ortho and para-substituted stilbenes **5**, **6** are obtained. Some factors causing the predominant formation of the erythro adducts **3**, **4**, as well as the influence of the ortho-substituents on the equilibrium threo-**3**, **4** ⇌ erythro-**3**, **4** are discussed.

INTRODUCTION

It is known that the Horner-Emmons reaction with stabilized phosphonate carbanions proceeds in a highly (E)-stereoselective manner, thus rendering the (Z)-alkenes rather difficultly accessible if this synthetic approach is followed.[1-3] A comparatively high (Z)-stereoselectivity with phosphonate carbanions has been achieved in the preparation of esters[4-8] and nitriles[9-11] of some, mainly α-substituted, (Z)-unsaturated acids. The "salt-free" Wittig reaction with unstable (alkyl substituted) ylids[12] is a convenient method for the preparation of (Z)-alkenes, while the semi-stabilized (aryl substituted) ylids afford stilbenes with low stereoselectivity or such ones in which the (E)-isomers predominate.[12]

(Z)-alkenes can also be obtained by resolving diastereoisomeric mixtures of hydroxy phosphonate[13] or phosphine oxide[14,15] adducts followed by olefin formation from the erythro isomers or, in some cases, their alkaline salts. This approach is unsuitable in the case of (Z)-stilbenes, because the corresponding phosphine oxide adducts decompose to alkenes affording (Z)- and (E)-isomers even at −50°C.[15]

We demonstrated earlier that some (Z)-stilbenes and α-substituted styrenes can be easily obtained in high diastereomeric purity. A high erythro-stereoselectivity is achieved by the interaction between N,N,N′,N′-tetramethyldiamides of arylmeth-

*Author to whom all correspondence should be addressed.

anephosphonic acids and carbonyl compounds followed by a thermal olefination of the erythro-adducts.[16]

RESULTS

In the present work we wish to report the results from the further elaboration of our method mentioned above leading to (Z)-ortho- or para-substituted stilbenes.[16]

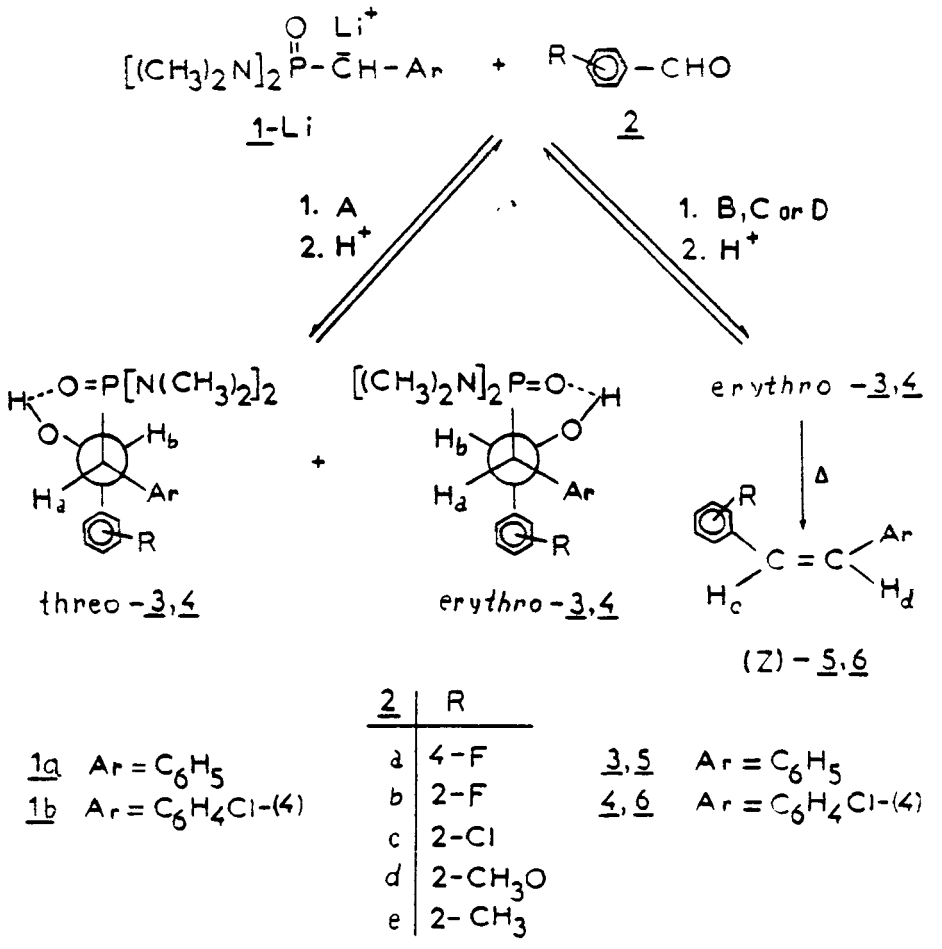
We studied the interaction between the Li-derivatives of the N,N,N′,N′-tetramethyldiamides of the arylmethanephosphonic acids **1a, b** (**1a**: Ar = C_6H_5; **1b**: Ar = C_6H_4—Cl—(4))[16] with ortho- and para-substituted benzaldehydes **2a–e**. The starting phosphonamides **1** were metallated with BuLi in THF at −70°C, while the reaction of the obtained Li-reagents **1**-Li with the aldehydes **2** was carried out according to the procedures A, B, C and D[16] (see Scheme 1). The ratios of the erythro/threo adducts **3, 4** were determined in the crude reaction mixtures by means of ^{1}H-NMR spectroscopy in $CDCl_3$. The integral intensities of the H_a and H_b proton signals in the erythro and threo adducts were determined (see Table I). We found that procedure A led in all cases to diastereomeric mixtures of the N,N,N′,N′-tetramethyldiamides of 1-aryl-2-aryl-2-hydroxyethanephosphonic acids **3, 4** in which the erythro/threo ratio is approximately 50 : 50 or the threo isomer predominates. Procedure B employing 2-methoxybenzaldehyde (**2d**) and 2-methylbenzaldehyde (**2e**) affords only erythro phosphonamide adducts **3d, 4d** and **3e, 4e**. If halogen-substituted aldehydes (**2a, 2b** and **2c**) are used diastereomeric mixtures are obtained in which the erythro adducts predominate in the following erythro/threo ratios: 80 : 20 (**3a**), 70 : 30 (**3b**), 66 : 34 (**3c**), 90 : 10 (**4b**) and 92 : 8 (**4c**). Monitoring the reaction course with thin-layer chromatography and ^{1}H-NMR showed that the erythro/threo ratio in the interaction between **1a, b** and 4-fluorobenzaldehyde **2a** grew with the increase of the reaction time of 1 hr at room temperature from 80 : 20 to 95 : 5 (procedure C) (see Table I).

Procedure C leads to high stereoselectivity too in the case of the reaction of **1a** with 2-fluorobenzaldehyde (**2b**). Thus the erythro/threo ratio for **3b** becomes 89 : 11, compared with 70 : 30 (procedure B). An even higher stereoselectivity with respect to erythro-**3b** (95 : 5) was found with procedure D, the yields and purity of **3b** and **4b** however sharply deteriorated.†

2-Chlorobenzaldehyde (**2c**) was found to be the only case in which the erythro/threo ratio remained approximately unchanged when the reaction was conducted under the various above described conditions.

The newly synthesized products **3, 4a–e** were subjected to thermal olefination using Corey's procedure.[13] Table II shows that the highest (Z)-diastereomeric purity (95–99%) is observed for (Z)-2-methoxystilbene, (Z)-2-methoxy-4′-chlorostilbene (**5d, 6d**), for (Z)-2-methylstilbene and (Z)-2-methyl-4′-chlorostilbene (**5e, 6e**). (Z)-4-fluorostilbene, (Z)-4-fluoro-4′-chlorostilbene (**5a, 6a**), (Z)-2-fluorostilbene, (Z)-2-fluoro-4′-chlorostilbene (**5b, 6b**) and (Z)-2-chloro-4′-chlorostilbene (**6c**) are obtained in a

† The yields of the olefins **5b** and **6b** sharply decreases because of the presence of **1** in **3b** and **4b** which are subjected to thermal olefination.



SCHEME 1

Procedure A: THF, 5 hrs, −70°C.
Procedure B: THF, 5 hrs, −70°C, 1/2 hr to room temperature.
Procedure C: THF, 5 hrs, −70°C, 1/2 hr to room temperature, 1 hr at room temperature.
Procedure D: THF + HMPT(85 : 15), 5 hrs, −70°C, 1/2 hr to room temperature, 1 hr at room temperature.

relative high purity (90–95%) by the procedures B, C or D. Recrystallization was required only for the adduct **3c** obtained according to the procedure B.

DISCUSSION

In the present studies, similarly to our earlier investigations,[16] it appears that the erythro isomers are formed under thermodynamic control. This assumption is corroborated by the results obtained from extending the reaction time and/or increasing the reaction temperature (procedures B, C) which led to higher erythro/threo ratios (see Table I). The same is observed when the reaction is conducted in a more polar medium (THF : HMPT 85 : 15, procedure D), while in the less polar ether used as solvent the amount of the threo adduct is greater. This is an

TABLE I

Yields and constants of compounds **3** and **4** obtained from **1** and **2** according to procedures A, B, C and D

No	Ar	R	Procedure	Yield %, **3**, **4***	er/thr **3**, **4****	m.p.°C***	^{1}H-NMR, **3**, **4** erythro H_a	H_b	threo H_a	H_b
3a	C_6H_5	F-(4)	A	66	40 : 60		3.28	5.45	3.50	—
3a	C_6H_5	F-(4)	B	64	80 : 20					
3a	C_6H_5	F-(4)	C	50	95 : 5	127–129				
4a	C_6H_4—Cl-(4)	F-(4)	A	62	50 : 50		3.20	5.34	3.40	4.95
4a	C_6H_4—Cl-(4)	F-(4)	C	62	96 : 4	138–139				
3b	C_6H_5	F-(2)	B	57	70 : 30		3.45	5.65	3.76	5.43
3b	C_6H_5	F-(2)	C	52	89 : 11					
3b	C_6H_5	F-(2)	D	50	95 : 5	141–143				
4b	C_6H_4—Cl-(4)	F-(2)	A	59	66 : 34		3.48	5.63	3.78	5.35
4b	C_6H_4—Cl-(4)	F-(2)	B	72	90 : 10					
4b	C_6H_4—Cl-(4)	F-(2)	D	47	99 : 1	111–113				
3c	C_6H_5	Cl-(2)	A		66 : 34		3.53	5.62	3.77	5.32
3c	C_6H_5	Cl-(2)	B	71	66 : 34					
3c	C_6H_5	Cl-(2)	C		68 : 32	132–134				
3c	C_6H_5	Cl-(2)	D	34	70 : 30					
4c	C_6H_4—Cl-(4)	Cl-(2)	B	68	92 : 8	112–114	3.60	5.65	3.83	5.35
3d	C_6H_5	OCH_3-(2)	A	50	50 : 50		3.65	5.68	—	—
3d	C_6H_5	OCH_3-(2)	B	55	99 : 1	150–152				
4d	C_5H_4—Cl-(4)	OCH_3-(2)	B	67	99 : 1	145–147	3.60	5.67	—	—
3e	C_6H_5	CH_3-(2)	B	35	95 : 5	142–144	3.17	5.45	—	—
4e	C_6H_4—Cl-(4)	CH_3-(2)	B	56	99 : 1	133–135	3.20	5.47	—	—

*The yields are given for crude products after washing with ether/hexane 1 : 2.

**The erythro/threo ratio is determined on crude reaction mixtures.

***The m.p. are of recrystallised compounds **3** and **4**. The elemental analyses for **3** and **4** (erythro or a mixture of erythro + threo adducts) are in good agreement with the theoretical values. **3**, **4**: IR(nujol) 975–990 $cm^{-1}/\nu_{P-N}/$, 1140–1200 $cm^{-1}/\nu_{P=O}/$, 3200–3350 cm^{-1}/ν_{OH} bonded/. Erythro **3a** ^{1}H-NMR($CDCl_3$): δ 2.20(d, J = 8 Hz) and 2.80(d, J = 8 Hz, NCH_3, 12 H); 3.28(dd, 1 H, H_a, $^3J_{H_aH_b}$ = 2 Hz, $^2J_{H_aP}$ = 14 Hz); 5.45(dd, 1 H, H_b, $^3J_{H_aH_b}$ = 2 Hz, $^3J_{H_bP}$ = 7 Hz); 5.72(s, 1 H, OH); 6.60–7.25 (m, 9 H, C_6H_5, C_6H_4—).

TABLE II

Yields and Z/E ratios of the olefins **5** and **6** obtained from the erythro adducts **3**, **4**

No	Ar	R	Yield %*	|Z| : |E|	Ref.
5a	C_6H_5	F-(4)	66	92 : 8	21, 22
6a	C_6H_4—Cl-(4)	F-(4)	60	94 : 6	21
5b	C_6H_5	F-(2)	66	89 : 11	
5b	C_6H_5	F-(2)	14	95 : 5**	
6b	C_6H_4—Cl-(4)	F-(2)	55	91 : 9	
6b	C_6H_4—Cl-(4)	F-(2)	25	99 : 1**	
5c	C_6H_5	Cl-(2)	86	99 : 1***	22
6c	C_6H_4—Cl-(4)	Cl-(2)	60	91 : 9	
5d	C_6H_5	OCH_3-(2)	77	99 : 1	22
6d	C_6H_4—Cl-(4)	OCH_3-(2)	78	99 : 1	
5e	C_6H_5	CH_3-(2)	60	95 : 5	23
6e	C_6H_4—Cl-(4)	CH_3-(2)	60	99 : 1	

*The crude products **3**, **4** obtained by procedure B or C were subjected to thermal olefination[13] after being washed with ether/hexane 1 : 2.

The olefins were obtained from the adducts **3b and **4b** synthesized by procedure D.

***(Z)-**5c** was obtained via olefination of recrystallized **3c**.

indication of the greater stability of the erythro zwitter ion also in the presence of a solvent with a powerful solvation of the lithium ion; the favoured complexation of this ion with the phosphonamide group is in a conformation with antiperiplanar oxyanion (open transition state)[16] (cf. 17).

The data listed in Table I indicate that the threo-**3**, **4** ⇌ erythro-**3**, **4** equilibrium is shifted more rapidly to the right (5 hrs at −70°C and 1/2 hr at room temperature, procedure B) in the case of the reaction between **1** and aldehydes containing electron-donating substituents (**2d** and **2e**, R = CH_3O—, CH_3—), than with halo-substituted aldehydes. In the reaction of **1a** with 2-chlorobenzaldehyde (**2c**) the equilibrium remains unaffected even after prolonging the reaction time up to 15 hrs in THF as well as in THF/HMPT (see Table III). With an excess of the aldehyde (**2c**) which should hinder the threo → erythro isomerisation through the initial **1a** and **2c** no effect was detected on the interaction. One reason for the rapid establishment of equilibrium even at low temperatures can be the retroaldol decomposition facilitated by the ortho-effect of the chlorine atom.

The (considerably) greater preponderance of the threo adduct **3c** in the equilibrium mixture (65 : 35) in comparison with the other adducts **3** and **4** under the

TABLE III

Ratios of the diastereomeric **3c** obtained under different reaction conditions from **1a** and **2c**

No	Solvent	Procedure	Ratio of **1a** : **2c**	erythro vs. threo **3c**
1	THF	A	1 : 1	66 : 34
2	THF	B	1 : 1	66 : 34
3	THF	B	1 : 2	65 : 35
4	THF	C	1 : 1	68 : 32
5	THF/HMPT	D	1 : 1	70 : 30
6	THF	15 hrs, −70°C	1 : 1	65 : 35
7	THF/HMPT	15 hrs, −70°C	1 : 1	64 : 36

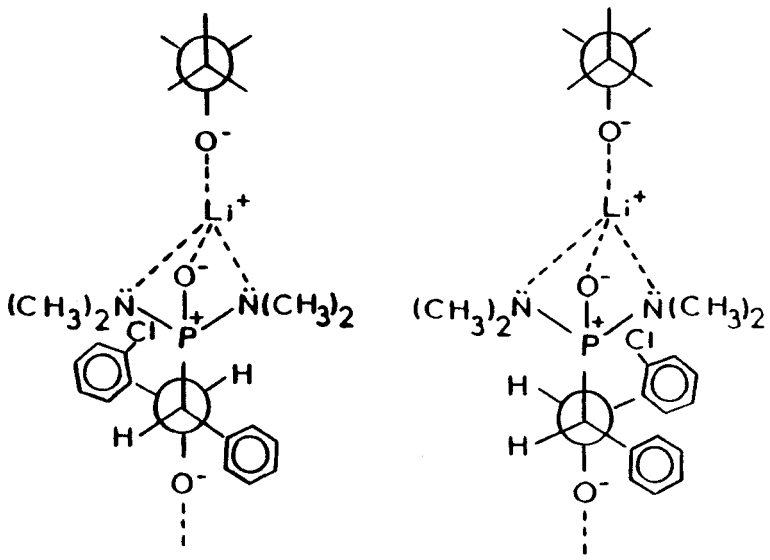
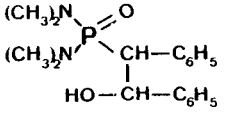


FIGURE 1

same conditions can be ascribed to the smaller difference in thermodynamic stability between the erythro **3c** and threo-**3c** lithium salts. If a bulky and electron rich substituent is in the ortho position of the phenyl ring the repulsive interaction with the dimethylamido group nitrogens in both diastereo-isomers is greater than the gauche interactions with the other aromatic ring (Figure 1).

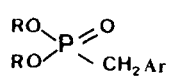
As mentioned in our earlier investigations[16] the isolated hydroxy adducts afford olefins on thermal treatment in good yields. This reaction proceeds to a much lesser extent in the case of their alkaline salts. Thus, after a 5 hour reflux the salts **7**-Li and **7**-Na lead to stilbene in yields of 9% and 25%, respectively (see Experimental), while the thermal treatment of the hydroxy adduct **7** afforded the stilbene in 74% yield.[16]



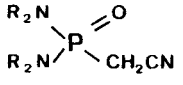
7

These observations indicate that the Horner-Emmons olefination reaction is affected significantly not only by stabilising the carbanion electron-withdrawing α-substituents (CN, CO, Ar)[19] but also by the nature of the substituents on phosphorus. In this respect, suitable starting compounds in this reaction are, for

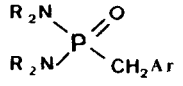
instance, the esters **8**[20] and the amides **9**.[10] (In the case of the Corey olefination procedure: **1** and **10**).[13]



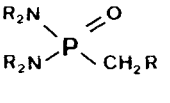
8



9



1



10

It can thus be concluded that the reagents of type **1**, to which the presently investigated phosphonamides, belong can be characterised by a reduced electrophilicity at phosphorus and a medium activating effect due to the aryl group. These compounds occupy an interposition i.e. they are inefficient reagents in the Horner-Emmons reaction, but good starting materials for the Corey elimination procedure.

EXPERIMENTAL

The reactions with **1** and **2** are conducted under dry argon. The THF was treated with $LiAlH_4$, distilled and then refluxed over sodium in the presence of benzophenone. The aldehydes were distilled prior to use. The olefins were determined using ^{1}H-NMR, UV and IR spectroscopy. The (Z)/(E) ratios were determined by gas chromatography (8% PEGA Chromosorb P/NAW) making use of standards prepared as required.[24] The olefins were purified by column chromatography on alumina eluting with hexane. The qualitative TLC studies were conducted on Merck Kieselgel 60 F_{254} Alufolio using a · a mobile phase ethyl acetate/heptane 2 : 1 for the adducts and hexane for the olefins.

The ^{1}H-NMR spectra were recorded on a JEOL-JNM-100 spectrometer with TMS or HMDSO as internal standard and using $CDCl_3$ as solvent. The presently obtained values for $J_{H_cH_d}$ of the olefins **5b**, **6b**, **6c**, **6d** and **6e** are within the limits of 11–12 Hz which is in agreement with the available data in the literature regarding similarly substituted stilbenes.[25]

Synthesis of N,N,N′,N′-tetramethyldiamides of 1-aryl-2-aryl′-2-hydroxyethanephosphonic acids **3**, **4**. Procedure A and B–according to ref. 16.

Procedure C. Butyllithium (10 mmol, 1.6 M in hexane, diluted with 8 ml of THF) is added to the solution of **1** (10 mmol) in anhydrous THF (20 ml) cooled to −70°C. The mixture is stirred 1/2 hr, the aldehyde **2** (10 mmol) dissolved in THF (4 ml) is added and stirring continued for another 5 hrs at −70°C, then within 1/2 hr the reaction mixture is allowed to rise to room temperature and kept there for one hour. The mixture is then hydrolysed with water (5 ml), extracted with methylene chloride or chloroform (3 × 20 ml), the extracts washed with water and dried (magnesium sulfate). The crude reaction products **3**, **4** remaining after the removal of the solvent were studied by ^{1}H-NMR and tlc.

The crude **3**, **4** were purified by washing with ether/hexane 1 : 2 and subjected to thermal olefination. The washed crude **3**, **4** were recrystallized from ether or CCl_4 prior to determining their physical constants and elemental analysis.

Procedure D. The reaction between **1**, BuLi and **2** is conducted as described in procedure C: **1** is dissolved in a 18.2 ml of THF and 4.8 ml of HMPT mixture, the butyllithium is added dissolved in 5 ml of THF and the aldehyde in 4 ml of THF.

Conversion of the hydroxyphosphonamide adducts into olefins. 1. Thermal elimination reaction in the phosphonamide adducts **3**, **4** to the olefins **5**, **6**.

General procedure: the mixture of **3**, **4** (2 mmol), silicagel (1.400 g) and toluene (16 ml) is refluxed with stirring 3—5 hrs. The reaction mixture is cooled, filtered, the silicagel filtered and washed with ether (2 × 30 ml), the combined filtrates washed with water, dried over magnesium sulfate and the solvent distilled off to give a residue which was subjected to column chromatography on alumina using hexane as eluent.

2. Olefination of the sodium salt of N,N,N′,N′-tetramethyldiamide of 1,2-diphenyl-2-hydroxyethanephosphonic acid **7-Na**. The mixture of **1a** (1.15 g, 5 mmol), sodium amide (0.500 g) and anhydrous THF (5 ml) is stirred 1 hr at room temperature under argon. Benzaldehyde (0.530 g, 5 mmol) dissolved in THF (7 ml) is then added and the mixture refluxed for 5 hrs. After hydrolysis with water (10 ml) the organic layer is extracted with ether (3 × 15 ml), the extracts washed with water, dried over magnesium sulfate, the solvants distilled off and the residue subjected to column chromatography (alumina, hexane) to give 0.23 g (25%) of stilbene (Z + E).

3. Olefination of **7-Li**. The reaction mixture obtained according to procedure B from **1a** (5 mmol), butyllithium and benzaldehyde (1 : 1) is refluxed in THF solvent for 5 hrs. It is then worked up as described in paragraph 2 for compound **7-Na** to give 0.040 g of stilbene (Z + E) in 9% yield.

(Z)-1-Fluoro-2(2-phenylethenyl)benzene; *2-fluoro-(Z)-stilbene* **5b**. $C_{14}H_{11}F$ (198.2) Calc. %: C, 84.83; H, 5.61. Found: C, 84/72; H, 6.00. IR (nujol): 698 cm^{-1} (δ CH—cis); λ_{max} 280 nm (95% ethanol). ^{1}H-NMR ($CDCl_3$); δ 6.45 (d, 1 H, H_c, $J_{H_cH_d}$ = 12 Hz); 6.58 (d, 1 H, H_d, $J_{H_dH_c}$ = 12 Hz).

(Z)-1-Chloro-4[2-(2-fluorophenyl)ethenyl]benzene; *4-chloro-2′-fluoro-(Z)-stilbene* **6b**. $C_{14}H_{10}ClF$ (232.7) Calc. %: C, 72.27; H 4.30. Found: C, 71.94; H, 4.21. IR (nujol): 758 cm^{-1} (δ CH-cis); λ_{max} 286 nm (95% ethanol). ^{1}H-NMR ($CDCl_3$): δ 6.45 (s, 2 H, H_c and H_d).

(Z)-1-Chloro-4[2-(2-chlorophenyl)ethenyl]benzene; *2-chloro-4′-chloro-(Z)-stilbene* **6c**. $C_{14}H_{10}Cl_2$ (249.1) Calc. % C, 67.49; H, 4.05. Found: C, 67.77; H, 4.26. IR (nujol): 740 cm^{-1} (δ CH–cis); λ_{max} 276 nm (95% ethanol). ^{1}H-NMR ($CDCl_3$); δ 6.51 (s, 2 H, H_c and H_d).

(Z)-1-Chloro-4-[2-(2-methoxyphenyl)ethenyl]benzene; *4-chloro-2′-methoxy-(Z)-stilbene* **6d**. $C_{15}H_{13}ClO$ (244.7) Calc. %: C, 73.62; H, 5.36. Found: C, 73.39; H, 5.23. IR (nujol): 750 cm^{-1} (δ CH-cis); λ_{max} 282 nm (95% ethanol). ^{1}H-NMR ($CDCl_3$): δ 3.69 (s, 3 H, CH_3), 6.40 (d, 1 H, H_c, $J_{H_cH_d}$ = 11 Hz), 6.55 (d, 1 H, H_d, $J_{H_cH_d}$ = 11 Hz).

(Z)-1-Chloro-4-[2-(2-methylphenyl)ethenyl]benzene; *4-chloro-2′-methyl-(Z)-stilbene* **6e**. $C_{15}H_{13}Cl$ (228.7) Calc. %: C, 78.77; H, 5.72. Found: C, 78.89; H, 5.92. IR (nujol): 740 cm^{-1} (δ CH-cis); λ_{max} 271 nm (95% ethanol). ^{1}H-NMR ($CDCl_3$): δ 2.22 (s, 3 H, CH_3), 6.48 (d, 1 H, H_c, $J_{H_cH_d}$ = 11 Hz) 6.64 (d, 1 H, H_d, $J_{H_dH_c}$ = 11 Hz).

REFERENCES

1. D. Wadsworth, O. E. Schupp, III, E. J. Seus and J. A. Ford, *J. Org. Chem.*, **30**, 680 (1965).
2. M. Schlosser and H. Ba Tuong, *Chimia*, **30**, 197 (1976).
3. J. I. G. Cadogan, "Organophosphorus Reagents in Organic Chemistry," Acad. Press, 1979, p. 174.
4. R. K. Huff, C. E. Moppett and J. K. Sutherland, *J. Chem. Soc.* (*C*), 2725 (1968).
5. E. Breuer and D. M. Bannet, *Tetrahedron Lett.*, 1141 (1977).
6. T. B. Strzalko, G. E. Moghadam and J. Seyden-Penne, *Nouveau J. de Chimie*, **7**, 155 (1983).
7. W. C. Still and K. R. Shaw, *Tetrahedron Lett.*, **22**, 3725 (1981).
8. H. Hagaoka and Y. Kishi, *Tetrahedron*, **37**, 3873 (1981).
9. A. Redjal and J. Seyden-Penne, *Tetrahedron Lett.*, 1733 (1974).
10. B. Deschamps, J. P. Lampin, F. Mathey and J. Seyden-Penne, *Tetrahedron Lett.*, 1137 (1977).
11. B. Deschamps, G. Lefebvre, A. Redjal and J. Seyden-Penne, *Tetrahedron*, **29**, 2437 (1973).
12. M. Schlosser and K. Christmann, *Liebigs Ann. Chem.*, **708**, 1 (1967).
13. E. J. Corey and G. T. Kwiatkowski, *J. Amer. Chem. Soc.*, **90**, 6816 (1968).
14. A. D. Buss and S. Warren, *J. Chem. Soc.*, Chem. Commun. 100 (1981).
15. A. D. Buss and S. Warren, *Tetrahedron Lett.*, **24**, 111 (1983).
16. J. Petrova, M. Kirilov and S. Momchilova, *Phosphorus and Sulfur*, **17**, 29 (1983).
17. S. Trippett, *Pure and Appl. Chem.*, **9**, 255 (1964).
18. C. F. Hauser, T. W. Brooks, M. L. Miles, M. A. Raymond and G. B. Butler, *J. Org. Chem.*, **28**, 372 (1963).
19. F. J. Corey, *J. Amer. Chem. Soc.*, **88**, 5654 (1966).
20. L. Horner, H. Hoffmann, W. Klink, H. Ertel, V. Toscano, *Chem. Ber.*, **95**, 581 (1962).
21. M. Gascoyne, P. J. Mitchell and L. Phillips, *J. Chem. Soc.*, Perkin Trans 2, 1051 (1977).
22. O. H. Wheelor and H. N. Patlle de Pabon, *J. Org. Chem.*, **30** 1473 (1965).
23. E. B. Merkushew and T. S. Skorohodowa, *Zh. Org. Chem.*, **18**, 355 (1982).
24. L. Zechmeister and Mc. Neely, *J. Amer. Chem. Soc.*, **64**, 1919 (1942).
25. H. Güsten and M. Salzwedel, *Tetrahedron*, **23**, 187 (1967).