Formation of a P^{III}—$C(sp^2)$ bond by addition of diphenyl(trimethylsilyl)phosphine to activated acetylenes

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Diphenyl(trimethylsilyl)phosphine reacts with alkoxy(alkyl)acetylenes to give mixtures of addition products, (2-alkoxy-2-trimethylsilylalkenyl)diphenylphosphines and (2-alkoxyalkenyl)diphenylphosphines. The reaction is sensitive to the solvent; in MeCN, it gives only nonsilylated products. (1-Alkoxyethenyl)diphenylphosphines were obtained as the main products upon the reaction of Ph_2PSiMe_3 with terminal alkoxyalkynes, irrespective of the reaction conditions.

Key words: diphenyl(trimethylsilyl)phosphine, alkoxy(alkyl)acetylenes, alkoxyacetylenes, addition; (1-alkoxyalkenyl)- and (2-alkoxyalkenyl)diphenylphosphines.

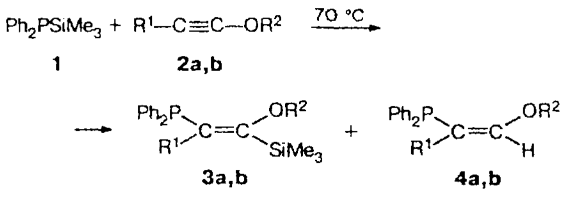
The addition of fragments arising upon cleavage of an element—element bond to unsaturated substrates is a promising method for the formation of an element—carbon bond in the synthesis of functionally substituted heteroorganic alkanes and alkenes that cannot be prepared using the most popular method involving organic derivatives of magnesium or lithium.

Previously we showed that noncatalytic addition of the P^{III}—Hal,[1,2] Si—Hal, and Ge—Hal [3,4] fragments to alkoxy- or aminoacetylenes makes it possible to synthesize functionally substituted alkenyl halides, which are valuable intermediates for the synthesis of heteroorganic di- and polyunsaturated compounds.[5]

To continue the studies on the addition of P^{III}—heteroatom fragments to nucleophilic alkynes, in the present work, we studied the interaction of diphenyl(trimethylsilyl)phosphine (**1**) containing a P—Si bond with internal and terminal alkoxyacetylenes (**2a—h**).

The reaction of compound **1** with alkoxy(alkyl)acetylenes **2a,b** (Scheme 1) occurs on keeping equimolar

Scheme 1



R^1 = Me, R^2 = Et (**a**); R^1 = R^2 = Et (**b**)

Table 1. Conditions of the synthesis and yields and boiling points of the products for the reactions of diphenyl(trimethylsilyl)phosphine **1** with alkoxyacetylenes **2a—h**

Acetylene	R^1	R^2	Solvent	Reaction duration/h	Reaction temperature/°C	Major product	Isolated yield (%)	B.p./°C (*p*/Torr)
2a	Me	Et	—	60	70—80	**3a**[a]	62	160 $(1.5 \cdot 10^{-2})$
2b	Et	Et	—	120	70—80	**3b**[b]	65	170 $(1.5 \cdot 10^{-2})$
2a	Me	Et	MeCN	48	70—80	**4a**	75	125 $(2.5 \cdot 10^{-2})$
2b	Et	Et	MeCN	72	70—80	**4b**	73	130 $(2 \cdot 10^{-2})$
2d	Me	Me	MeCN	36	70—80	**4c**	75	160 (1)
2e	Et	Me	MeCN	72	70—80	**4d**	68	120 $(2 \cdot 10^{-2})$
2f	Pr^i	Et	MeCN	88	70—80	**4e**	77	150 $(3 \cdot 10^{-2})$
2c	H	Et	—	48	20	**5a**[c]	50	105 (0.1)
2c	H	Et	MeCN	10	70—80	**5a**	77	120—130 (0.1)
2g	H	Me	MeCN	12	20	**5b**	68	125 $(2.5 \cdot 10^{-2})$
2h	H	Bu^n	MeCN	14	70—80	**5c**	72	140 $(2 \cdot 10^{-2})$

[a] **4a** is formed as a minor product (17%). [b] **4b** is formed as a minor product (14%). [c] **3c** is formed as a minor product (14%).

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Table 2. Parameters of the ^{1}H and ^{31}P NMR and IR spectra of (1-alkoxyalkenyl)- and (2-alkoxyalkenyl)diphenylphosphines **3a—c**, **4a—e**, **5a—c**

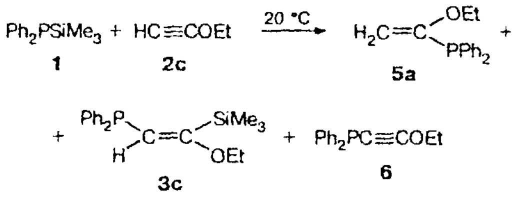
Com- pound	δH (J_{PH}, J_{HH}/Hz)				δP	ν(C=C) /cm^{-1}
	=CH or Me_3Si	O—R^2	R^1	Ph (m)		
3a	0.33 s	1.24 t; 3.70 q	1.60 d	7.32	−12.72	—
3b	0.33 s	1.15 t; 3.70 q	0.58 t; 2.10 t	7.24	−12.36	—
3c	0.12 s; 5.25 d	1.20 t; 4.17 q	—	7.26	−15.20	—
4a	6.72 dq (15.6, 1.6)	1.27 t; 3.92 q	1.55 dd (2.4, 1.6)	7.50	−20.20	1630
4b	6.43 dt (14.0, <1)	0.84 t; 3.43 q	1.16 t; 2.41 m	7.25	−16.62	1620
4c	6.62 dq (15.8, 1.4)	3.67 s	1.49 dd (3.8, 1.4)	7.41	−19.50	1625
4d	6.69 dt (15.0, <1)	3.65 q	1.00 t; 2.10 m	7.30	−16.88	1625
4e	6.72 br.d (11.6)	1.04 t; 3.72 q	1.20 d; 2.45 m (1.5)	7.48	−19.07	1620
5a	4.47 dd (8.3); 4.76 dd (24.5)	1.23 t; 3.81 q	—	7.48	−4.78	1605
5b	4.53 dd (8.4); 4.80 dd (24.4)	3.57 s	—	7.50	−4.60	1605
5c	4.35 dd (9.1); 4.60 dd (26.3)	0.72 t; 1.37 m; 3.61 t	—	7.34	−4.04	1610

amounts of the reactants without a solvent at 70 °C for 60—120 h and gives (according to ^{31}P NMR spectroscopy) a product mixture consisting of *E*-(2-alkoxy-2-trimethylsilylalkenyl)diphenylphosphines (**3a**,**b**, δP −12.35 to −12.72) and *Z*-(2-alkoxyalkenyl)diphenylphosphines (**4a**,**b**, δP −18.08 to −18.38); the **3** : **4** ratio is (5 to 6) : 1 (Table 1). The transformation is quantitative, as indicated by the fact that the ^{31}P NMR spectrum of the reaction mixture does not contain the signal of the starting silylphosphine **1** (δP −57.6), and the IR spectrum does not contain the ν(C≡C) absorption band (2300 cm^{-1}) corresponding to alkoxy(alkyl)acetylene **2**.

The structures of compounds **3** and **4** were established based on the data of ^{1}H and ^{13}C NMR spectroscopy and confirmed by the IR and ^{31}P NMR spectroscopy (see Experimental and Tables 2 and 3).

The reaction of silylphosphine **1** with a terminal alkyne, ethoxyacetylene **2c**, follows a more complex pathway (Scheme 2). ^{31}P NMR analysis of the mixture obtained after the reaction (48 h, 20 °C) indicates the formation of three compounds: two addition products, (1-ethoxyethenyl)diphenylphosphine (**5a**, δP −4.78) and *Z*-diphenyl(2-trimethylsilyl-2-ethoxyethenyl)phosphine (**3c**, δP −15.2), and the product of substitution of the acetylenic hydrogen atom by a phosphorus-containing residue, (2-ethoxyethenyl)diphenylphosphine (**6**, δP −23.5). Before distillation, the **5a** : **3c** : **6** ratio is 4.0 : 1.5 : 0.5, and after distillation, only phosphine **5a** can be isolated.

Scheme 2



We showed previously[6] that alkoxyethynylphosphine **6** is relatively unstable and rearranges spontaneously to ketenylidenephosphorane 7.

Table 3. Parameters of the ^{13}C NMR spectra of (1-alkoxyalkenyl)- and (2-alkoxyalkenyl)diphenylphosphines **3a—c**, **4a—e**, **5a—c**

Com- pound	δ (J_{PC}/Hz)				
	=$\underline{C}$—O	$\underline{C}$=	O—$\underline{C}$	O—C—$\underline{C}$	$\underline{R}^1$—C=
3a	168.80 (12.9)	105.00 (17.7)	67.15	15.25	16.80
3b	153.55 (14.3)	110.30 (15.9)	66.50	15.30	15.12; 23.30
3c	159.10 (67.2)	97.60 (<1)	65.00	18.19	—
4a	153.55 (19.9)	105.41 (15.3)	68.72	15.73	16.95 (2.3)
4b	152.39 (15.9)	113.32 (16.2)	67.98	15.14	15.26; 24.98 (>1)
4c	154.60 (18.7)	104.62 (13.1)	60.17	—	16.56 (4.3)
4d	153.61 (19.7)	111.55 (16.3)	59.91	—	14.48; 24.03 (>1)
4e	151.90 (5.9)	117.06 (18.7)	68.31	15.29	24.26 (5.2); 31.81 (15.4)
5a	164.06 (8.7)	97.27 (31.5)	64.23 (3.0)	14.44	—
5b	164.02 (6.8)	96.12 (29.9)	55.50	—	—
5c	166.32 (7.4)	99.85 (32.7)	71.13	16.13; 21.93; 33.70	—

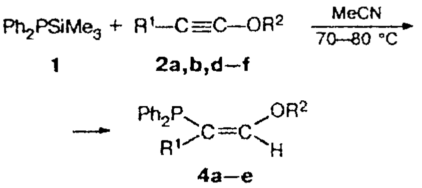
$$\underset{\mathbf{6}}{\mathrm{Ph_2PC{\equiv}COEt}} \longrightarrow \underset{\mathbf{7}}{\mathrm{Ph_2P(Et){=}C{=}C{=}O}}.$$

The structures of compounds **5a** and **3c** were determined using ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy. Phosphine **5a** is responsible for the characteristic low-field doublet of quartets (δH_{trans} 4.76, $^3J_{PH}$ = 24.5 Hz; δH_{cis} 4.47, $^3J_{PH}$ = 8.3 Hz, $^2J_{HH}$ = 2.1 Hz) in the ^{1}H NMR spectrum. The ^{13}C NMR spectrum contains two doublets for vinylic carbon atoms (δC 97.27, $^2J_{PC}$ = 31.5 Hz and δC 164.06, $^1J_{PC}$ = 8.7 Hz). The ^{1}H NMR spectrum of compound **3c** exhibits signals for the trimethylsilyl group (δH 0.12) and the vinylic proton (δH 5.25, $^2J_{PH}$ = 11.2 Hz), and the ^{13}C NMR spectrum contains signals for the vinylic carbon atoms (δC 97.60, $^1J_{PC} <$ 1 Hz and δC 159.10, $^2J_{PC}$ = 67.2 Hz).

We suggested that nonsilylated products are formed due to the rigorous conditions of the reaction (prolonged heating); therefore, we attempted to conduct this process under catalytic conditions by using either radical initiators or palladium complexes as the catalysts. However, the introduction of these catalysts changed neither the process duration nor the structure or the ratio of the products formed.

However, the reaction carried out in a polar solvent, acetonitrile, was substantially accelerated and, in the case of internal alkoxyacetylene, gave (2-alkoxyalkenyl)diphenylphosphines **4a—e** as the major products (Scheme 3).

Scheme 3

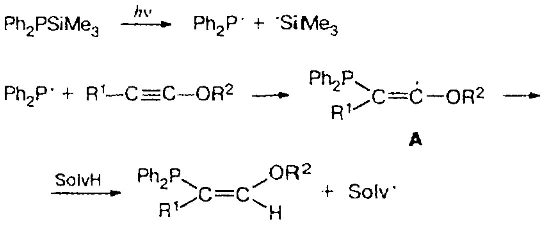


R^1 = Me (**2a,d**, **4a,c**); Et (**2b,e**, **4b,d**); Pr^i (**2f**, **4e**)
R^2 = Me (**2d,e**, **4c,d**); Et (**2a,b,f**, **4a,b,e**)

The yields of phosphines **4a—e** determined by ^{31}P NMR spectroscopy were 85—90%. After distillation, compounds **4a—e** were isolated in 68—77% yields (see Table 1). It can be seen from Table 1 that the reaction duration depends on the nature of the radical R^1, decreasing in the series Pr^i > Et > Me.

The formation of a nonsilylated compound as the only product in acetonitrile may be due to the fact that the reaction follows a radical pathway (Scheme 4) in which the arising alkenyl radical (**A**) is stabilized by abstracting a hydrogen atom from, for example, a solvent molecule (SolvH).

Scheme 4

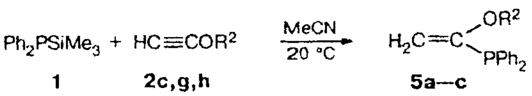


The possibility of radical cleavage of the P—Si bond was confirmed by performing the addition of silylphosphine **1** in toluene. In the case of relatively nonreactive ethoxy(ethyl)acetylene **2b** at 20 °C, the ^{31}P NMR spectrum of the reaction mixture recorded after 72 h, apart from the signal of the initial phosphine **1**, contained only one signal with δP −41 corresponding to diphenylphosphine **8**. The addition of silylphosphine **1** to a more reactive alkoxyacetylene, namely ethoxyacetylene **2c**, in toluene at room temperature affords both nonsilylated product **5a** and compound **3c** containing a trimethylsilyl group.

The reaction presented in Scheme 3 is stereoselective, and phosphine (**4a—e**) is formed as a single geometrical isomer, whose configuration was determined based on the criterion proposed in our previous study,[7,8] namely, the magnitude of the geminal spin-spin coupling constant ($^2J_{PC}$) of the phosphorus atom with the vinylic carbon atom attached to the alkoxy group. For compounds **4a—e**, this constant is 5.9—19.9 Hz, *i.e.*, it falls into the range typical of the *cis*-arrangement of the P atom and the alkoxy group; hence, the isomer formed upon the addition is characterized by the *Z*-configuration of the double bond.

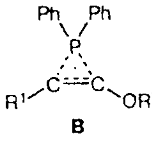
Unlike alkoxy(alkyl)acetylenes, terminal alkoxyacetylenes **2c,g,h** react with silylphosphine **1** in acetonitrile under mild conditions; the reactions occur at room temperature over a period of 24—72 h (Scheme 5).

Scheme 5



R^2 = Me (**2g**, **5b**); Et (**2c**, **5a**); Bu^n (**2h**, **5c**)

The main products of these reactions are phosphines **5a—c**, which also contain no trimethylsilyl groups, but the regioselectivity of the addition differs from that observed with internal alkoxyacetylenes. This difference between the regiochemistry of the reactions of terminal and internal alkoxyacetylenes (see Schemes 3 and 5) can be due either to different stabilities of the corresponding radical intermediates (kinetic or thermodynamic control) or to the formation of a bridged radical of type **B**.

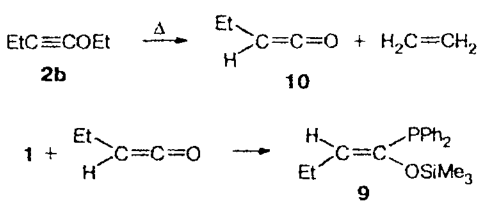


B

In the ^1H NMR spectra of phosphines **5a—c**, the vinylic protons are responsible for two doublets of doublets, typical of an ABX system, with $^3J_{PH}$ = 8.3—9.1 Hz for the *cis*-proton and $^3J_{PH}$ = 24.4—26.3 Hz for the *trans*-proton.

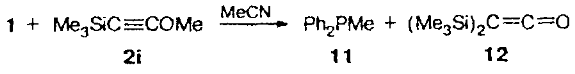
Since Ph_2PSiMe_3 slowly reacts with internal alkoxyacetylenes without a solvent, we attempted to reduce the reaction time by conducting the process at a higher temperature (130—140 °C). In the case of ethoxy(ethyl)acetylene, this gave the only product with δP −4.93, which was identified based on the data of ^1H and ^{13}C NMR spectroscopy as *E*-diphenyl(1-trimethylsilyloxybut-1-en-1-yl)phosphine (**9**). The formation of this product can be explained by assuming that at 140 °C, ethoxy(ethyl)acetylene eliminates ethylene, being thus converted into ethyl ketene **10** (Scheme 6).

Scheme 6



A special experiment showed that ethoxy(ethyl)acetylene actually decomposes at temperatures above 100 °C with evolution of ethylene. At 140 °C, decomposition occurs quantitatively over a period of 10 h, as indicated by the disappearance of the absorption band due to the triple bond, ν(C≡C) 2300 cm^{-1}, from the IR spectrum. Thus, adduct **9** results from the addition of silylphosphine to the ketene formed *in situ*; the addition occurs similarly to the known[9] reactions of trimethylsilyldiethylphosphine with ketene and diphenyl ketene.

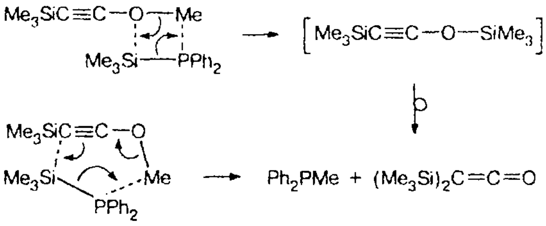
An interesting result was obtained in the reaction of silylphosphine **1** with methoxy(trimethylsilyl)acetylene **2i** in acetonitrile.



$$\mathbf{1} + \underset{\mathbf{2i}}{Me_3SiC{\equiv}COMe} \xrightarrow{MeCN} \underset{\mathbf{11}}{Ph_2PMe} + \underset{\mathbf{12}}{(Me_3Si)_2C{=}C{=}O}$$

The ^{31}P NMR spectrum recorded after completion of the reaction contained only one signal with δP −27.9. Fractionation of the reaction mixture afforded methyldiphenylphosphine (**11**, yield 82%) and bis(trimethylsilyl) ketene (**12**, yield 65%), which were identified by comparison of their physicochemical parameters with the published data;[10,11] the structures of these products were confirmed by spectroscopy. A four- or six-membered transition state can be proposed for this transformation (Scheme 7).

Scheme 7



The unusual pathway of this reaction of silylphosphine can be due to the large size of the substituent at the β-C atom in compound **2i**, which prevents the addition to the triple bond as shown in Scheme 3.

Experimental

All reactions and operations associated with the synthesis and isolation of compounds of tricoordinated phosphorus were carried out under dry argon using anhydrous solvents.

All the stable reagents used in the reactions were freshly distilled. The purity of the alkoxyacetylenes used was checked by spectroscopy (IR, ^1H NMR). IR spectroscopy (in thin films) and ^1H, ^{13}C, and ^{31}P NMR spectroscopy were used to monitor the course of the reactions and to identify the reaction products.

IR spectra were recorded on an IKS-22 spectrometer (NaCl). ^1H NMR spectra were measured on Tesla BS-467 and Varian VXR-400 instruments (60 and 400 MHz, respectively) using Me_4Si and HMDS as internal standards. ^{31}P NMR spectra were run on a Varian FT-80A instrument (32.2 MHz) using 85% H_3PO_4 as the external standard. ^{13}C NMR spectra were recorded on a Varian VXR-400 spectrometer (100.6 MHz) with dichloromethane-d_2 and chloroform-d_1 as internal standards. The chemical shifts are given in the δ scale in relation to Me_4Si (^1H, ^{13}C) or 85% H_3PO_4 (^{31}P).

The reactions with diphenyl(trimethylsilyl)phosphine **1** were carried out until its signal (δP −57.6) disappeared from the ^{31}P NMR spectrum of the reaction mixture.

Reaction of diphenyl(trimethylsilyl)phosphine (1) with alkoxyacetylenes and alkoxy(alkyl)acetylenes (general procedure). A mixture of alkoxyacetylene **2a—h** (0.01 mol) and compound **1** (0.01 mol), neat or in MeCN, was held in a sealed tube at room temperature or at 70—80 °C. After completion of the reaction, the mixture was kept at 20 °C under a pressure of 0.1 Torr for 1 h, and the remaining oil was distilled. The reaction conditions and the yields and boiling points of the products are listed in Table 1, and the spectral data are presented in Tables 2 and 3.

The reactions without a solvent gave the following compounds: from ethoxy(methyl)acetylene **2a**, ***E*-(1-methyl-2-trimethylsilyl-2-ethoxyethenyl)diphenylphosphine (3a)** and ***Z*-(1-methyl-2-ethoxyethenyl)diphenylphosphine (4a)** in 5 : 1 ratio; from ethoxy(ethyl)acetylene **2b**, ***E*-diphenyl(2-trimethylsilyl-1-ethyl-2-ethoxyethenyl)phosphine (3b)** and ***Z*-(1-ethyl-2-ethoxyethenyl)diphenylphosphine (4b)** in 6 : 1 ratio; from ethoxyacetylene **2c**, **(1-ethoxyethenyl)diphenylphosphine (5a)** (65%), ***Z*-diphenyl(2-trimethylsilyl-2-ethoxyethenyl)phosphine (3c)** (20%), and **(2-ethoxyethynyl)diphenylphosphine (6)** (15%); distillation of the mixture gave a mixture of compounds **3c** and **5a** in 6 : 1 ratio (yield 50%).

The reactions in MeCN gave the following products: from ethoxyacetylene **2c**, **(1-ethoxyethenyl)diphenylphosphine (5a)** (found (%): C, 74.35; H, 6.48; $C_{14}H_{17}OP$; calculated (%): C, 74.99; H, 6.69); from methoxyacetylene **2g**, **(1-methoxyethenyl)diphenylphosphine (5b)**; from butoxyacetylene **2h**, **(1-butoxyethenyl)diphenylphosphine (5c)**; from methoxy(methyl)acetylene **2d**, ***Z*-(1-methyl-2-methoxyethenyl)diphenylphosphine (4c)**; from ethoxy(methyl)acetylene **2a**, ***Z*-(1-methyl-2-ethoxyethenyl)diphenylphosphine (4a)** (by passing air through a benzene solution of phosphine **4a** for 24 h at 70 °C, this compound was converted into *Z*-(1-methyl-2-ethoxyethenyl)diphenylphosphine oxide, whose ^{31}P NMR spectrum contained a signal with δP 28.0; found (%): C, 71.70; H, 7.26; $C_{17}H_{19}O_2P$; calculated (%): C, 71.32; H, 6.69); from ethoxy(ethyl)acetylene **2b**, ***Z*-(1-ethyl-2-ethoxyethenyl)diphenylphosphine (4b)**; from ethyl(methoxy)acetylene **2e**, ***Z*-(2-methoxy-1-ethylethenyl)diphenylphosphine (4d)**; from ethoxy(isopropyl)acetylene **2f**, ***Z*-(1-isopropyl-2-ethoxyethenyl)diphenylphosphine (4e)** (found (%): C, 76.08; H, 8.06; $C_{19}H_{23}OP$; calculated (%): C, 76.49; H, 7.77).

Reaction of diphenyl(trimethylsilyl)phosphine (1) with ethoxy(ethyl)acetylene 2b. A mixture of ethoxy(ethyl)acetylene **2b** (0.01 mol) and phosphine **1** (0.01 mol) was placed in a 25-mL two-neck flask equipped with a reflux condenser and an inlet for argon and heated for 15 h at 130—140 °C. The gas evolved during the reaction was passed through a solution of Br_2 (0.01 mol) in CCl_4 (10 mL), which became colorless by the instant the reaction was completed. Vacuum distillation gave ***E*-diphenyl(1-trimethylsilyloxybut-1-en-1-yl)phosphine (9)**, yield 79%, b.p. 145 °C ($5 \cdot 10^{-2}$ Torr). ^{31}P NMR (MeCN), δ: −4.93. IR, ν/cm^{-1}: 1620 (C=C). ^{1}H NMR (CD_2Cl_2), δ: 0.06 (s, 9 H, $(CH_3)_3Si$); 0.95 (t, 3 H, CH_3); 2.36 (m, 2 H, CH_2); 5.42 (t, 1 H, HC=, *J* = 6 Hz); 8.0 (m, 10 H, Ph). ^{13}C NMR (CD_2Cl_2), δ: 1.60 ($(CH_3)_3Si$); 14.79 ($CH_2\underline{C}H_3$); 20.52 (d, $\underline{C}H_2CH_3$, *J* = 4.2 Hz); 126.32 (d, $\underline{C}$=C—O, *J* = 23.5 Hz); 151.56 (d, C=$\underline{C}$—O, *J* = 8.3 Hz); 136.78 (d, C_{ipso}, *J* = 10.7 Hz).

Reaction of diphenyl(trimethylsilyl)phosphine (1) with methoxy(trimethylsilyl)acetylene (2i). A mixture of methoxy(trimethylsilyl)acetylene **2i** (0.01 mol) and compound **1** (0.01 mol) in 4 mL of MeCN was kept for 48 h at room temperature. The ν(C≡C) absorption band at 2100 cm^{-1} disappeared from the IR spectrum, and the ν(C=C=O) band at 2030 cm^{-1} appeared instead. Fractionation gave **methyldiphenylphosphine (11)**, yield 82%, b.p. 138—140 °C (1 Torr) and **bis(trimethylsilyl) ketene (12)**, yield 65%, b.p. 60 °C (30 Torr). Compound 11. ^{31}P NMR (MeCN), δ: −27.90. ^{1}H NMR (CD_2Cl_2), δ: 6.80 (d, 3 H, CH_3, *J* = 1.1 Hz); 7.30 (m, 10 H, PPh_2). ^{13}C NMR ($CDCl_3$), δ: 12.63 (d, CH_3, *J* = 5.1 Hz); 140.80 (d, C_{ipso}, *J* = 5.1 Hz); 134.50 (d, C_o, *J* = 14.0 Hz); 128.66 (C_m); 128.77 (C_p). Published data:[10] ^{31}P NMR (C_6D_6), δ: 27.90. Compound 12. IR, ν/cm^{-1}: 2030 (C=C=O). ^{1}H NMR ($CDCl_3$), δ: 0.12 (s, Me_3Si). The spectral parameters of compound **12** correspond to the published data.[11]

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