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A convenient synthesis of a sterically protected 1,4-diphosphabutatriene and its pentacarbonyltungsten complexes

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Abstract

1,4-Bis(2,4,6-tri-*t*-butylphenyl)-1,4-diphosphabutatriene was obtained by the copper-mediated coupling reaction of 1-halo-2-phosphaethenyllithiums. The 1,4-diphosphabutatriene formed mono- and bis-pentacarbonyltungsten complexes, and the X-ray analysis of the bis-tungsten complex revealed end-on type coordination with the *trans* configuration. © 1998 Elsevier Science S.A.

Keywords: 1,4-Diphosphabutatriene; Copper-mediated coupling reaction; Tungsten pentacarbonyl complex; X-ray analysis; Steric protection

1. Introduction

Sterically protected and multiple-bonded organophosphorus compounds are currently of interest [1]. Utilizing the 2,4,6-tri-*t*-butylphenyl group (abbreviated to the Ar group) as a protecting group, we have reported the synthesis and characterization of diphosphenes [2,3], phosphaethenes [4], and so on. Phosphacumulenes are also attractive compounds, and we and others have been successful in the synthesis and characterization of 1-phosphaallenes [5–7], 1,3-diphosphaallenes [8–11], and 1-phosphabutatrienes [12,13]. Although 1,4-diphosphabutatriene **1** was synthesized by Märkl and Kreitmeier [14] and ourselves [15], the properties of **1** have little been revealed. Indeed, there exist *trans* and *cis* isomers for **1**, but no X-ray crystallographic analysis has been carried out. We now report on a simple and convenient preparation of **1** and formation of its pentacarbonyltungsten complexes. Furthermore, X-ray analysis of one of the tungsten complexes was performed as the first example of the *E*-1,4-diphosphabutatriene system.

2. Results and discussion

2.1. Copper-mediated coupling reaction of 1-halo-2-phosphaethenyllithiums

Previous methods for the preparation of 1,4-diphosphabutatriene **1** are depicted in Scheme 1. Märkl and Kreitmeier [14] reported the reaction of silylated ethynylphosphine with dichlorophosphine **2** affording the butatriene **1** ([3 + 1] system, Method A), while we reported the preparative method of repeated utilization of the sequence of addition of dichlorocarbene to the P=C bond and rearrangement to allene as the modified Doering–Moore–Skattebøl homologation ([2 + 1 + 1] system, Method B) [15]. These methods, however, require several elaborating steps.

On the other hand, 2,2-dihalo-1-phosphaethenes **3a**, **3b** are easily available from **2** and can be derived to the corresponding 1-halo-2-phosphaethenyllithiums **4a**, **4b** by the halogen–metal exchange reaction at low temperature [16,17]. Halophosphaethenyllithium reagents **4** are the phosphorus analogues of alkylidene carbenoids [18,19] and useful synthons for novel organophosphorus compounds. In fact, van der Sluis et al. [20] and us [21] reported on the reactions of **4a** with several carbonyl compounds affording the multifunctionalized phosphaethenes.

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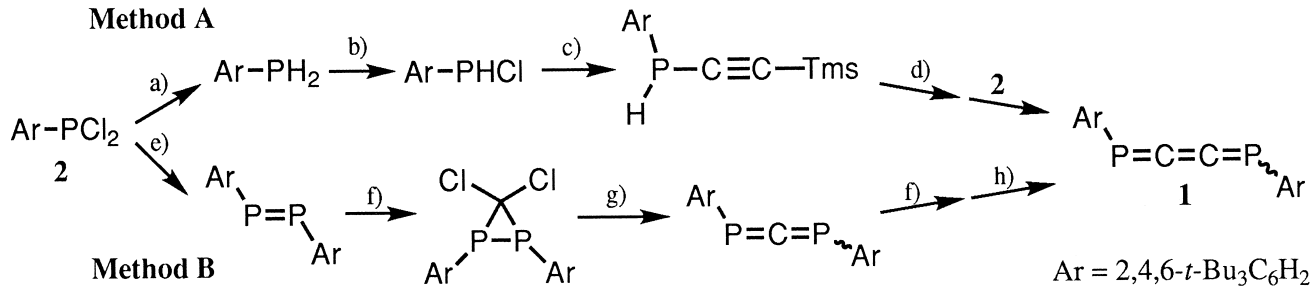


Scheme 1. *Reagents*: (a) LAH; (b) CCl_4, AIBN; (c) LiC≡CTms; (d) MeLi/TMEDA; (e) Mg; (f) $[PhCH_2NEt_3]Cl$, NaOH, $CHCl_3$; (g) *t*-BuLi; (h) $Li^+C_{10}H_8^{-\cdot}$.

When chlorophosphaethenyllithium **4a** (*Z*-isomer) was generated in the presence of copper(II) chloride, 1,4-diphosphabutatriene **1** and 1,4-diphospha-1,3-butadiene **5a** were obtained [22], while Niecke et al. [23] reported the dimerization reaction of **4a** to 1,3-diphosphacyclobutane-2,4-diyl as shown in Scheme 2. However, the yields of **1** and **5a** were dependent on the quenching time and temperatures as well as on the presence or absence of oxygen. Although **1** was obtained in the absence of oxygen [22], the yields of **1** were strongly dependent on quenching time. [1] In this reaction, copper(II) chloride might first react with 2 mol of **4a** to form an organocopper intermediate, and upon warming, the reductive elimination from the intermediate occurs to afford **1**. Furthermore, butatriene **1** was obtained always as a mixture of $E/Z = 4{:}1$ isomers and almost insoluble in most common organic solvents. [2]

1-Bromo-2-phosphaethenyllithium **4b** was derived from **3b** as a mixture of *E*/*Z*-isomer (1:5), and afforded **1** and/or butadiene **5b** (Scheme 2, Table 1). [3] In contrast, in the case of **4b**, butatriene **1** was the major product at −78°C, accompanied with **5b** as the minor product. Because of the high leaving ability of bromide compared to chloride, butatriene **1** might be formed mainly from **4b**. At −95°C, however, **4b** afforded only butadiene **5b** in 22% yield. Thus the present copper-mediated coupling reaction of 1-halo-2-phosphaethenyllithiums **4a**, **4b** is a simple and efficient method for the preparation of 1,4-diphosphabutatriene **1** ([2 + 2] system). Moreover, halophosphaethenyllithiums **4a**, **4b** are useful for preparation of 2,3-dihalo-1,4-diphospha-1,3-butadienes **5a**, **5b**, which might serve as another type of novel intermediates in the organophosphorus compounds [24].

2.2. Complex formation and structural analysis

1,4-Diphosphabutatriene **1** is an attractive ligand for metal complexes because of its having various kinds of possible coordination modes. Previously, we reported on an end-on type coordinated pentacarbonyltungsten complex of 4,4-diphenyl-1-(2,4,6-tri-*t*-butylphenyl)-1-phosphabutatriene [25].

Butatriene **1** ($E/Z = 4{:}1$) was allowed to react with an excess amount of $W(CO)_5(THF)$ to give the dicoordinated complex **6**. When **1** was allowed to react with 1 equivalent of $W(CO)_5(THF)$, a peak due to monocoordinated complex **7** was observed by ^{31}P NMR together with that due to **6** (Scheme 3). Although **7** could not be isolated, probably due to its instability, **6** was purified by silica-gel column chromatography. Both *E*- and *Z*-isomers were considered to be formed, but only **6** of *E*-configuration was obtained as a single isomer even if starting from an $E/Z = 4{:}1$ mixture of **1**, thus the *Z*-isomer of **6** was not detected. In the ^{31}P NMR spectrum of complex **6**, a peak appeared at δ_P 105.0 ($CDCl_3$) accompanied by satellite peaks due to ^{183}W of 14% natural abundance.

The structure of **6** was unambiguously established by the X-ray crystallographic analysis. Fig. 1 shows an ORTEP drawing of the molecular structure for **6** and

Table 1
Copper-mediated coupling reaction of **4** giving **1** and/or **5** (isolated yields)

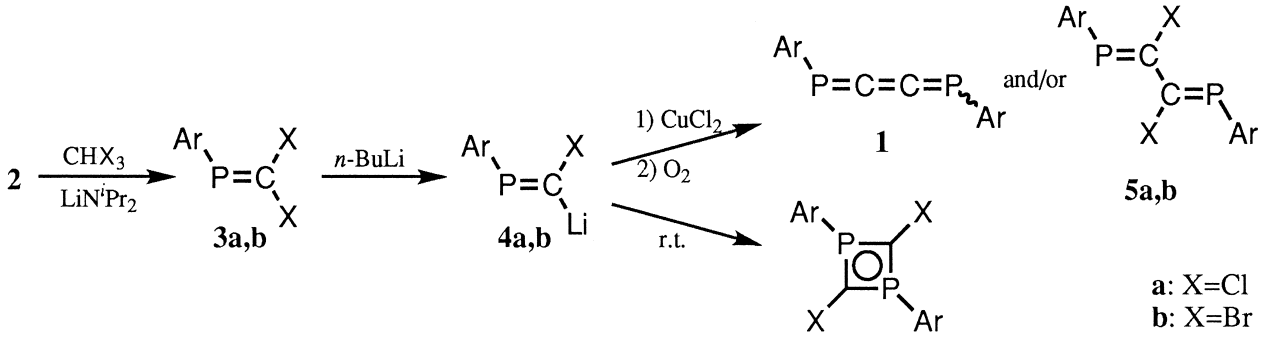
Substrate	Temperature (°C)	Yield of **1** (%)	Yield of **5** (%)
4a	0	54	7
4a	−78	0	46
4b	−78	47	13
4b	−95	0	22

a: X=Cl. **b**: X=Br. See Section 3 for the reaction conditions.

[1] In the previous communication [22], we reported that the yield of **1** was 63% using **3a** after immediate quenching in the absence of oxygen, but the yields depend on the reaction conditions especially on the quenching time. For example, no **1** was obtained after 30-min stirring at −78°C instead of immediate warming. Under the oxidative conditions described in this report, the yields of **1** and **5** were reproducible without any tricky reaction conditions, although in some cases the yields were slightly lower than those reported before.

[2] The *E*/*Z* ratio of 4:1 seems to be an equilibrium ratio in chloroform at 295 K. The ^{31}P NMR peak due to the *Z*-isomer disappeared upon heating at 318 K and it became visible again in the same *E*/*Z* ratio as before heating, when the mixture was cooled down at 295 K.

[3] In ^{31}P NMR (81 MHz), **4b** was observed as $E/Z = 1{:}5$ mixture at 210 K; δ_P (THF-d_8) *E*-**4b**, 369.7; *Z*-**4b**, 254.6.



Scheme 2.

Table 2 shows some selected bond lengths and angles. The molecule has a center of symmetry and the structure was solved as a half of the molecule. Complex **6** clearly shows end-on type coordination at both phosphorus atoms of *E*-butatriene configuration. The P–W bond distance is 2.492(2) Å. The P1–C1 and C1–C1* distances are 1.656(6) Å and 1.25(1) Å, respectively, which are very similar to those for the tungsten complex of 4,4-diphenyl-1-(2,4,6-tri-*t*-butylphenyl)-1-phosphabutatriene [25]. The W, P, C1, C2 atoms are coplanar (within 0.0 Å), and the plane makes an angle of 83.1° with the mean aromatic ring of the 2,4,6-tri-*t*-butylphenyl group.

Since the structure of the free ligand **1** has not been solved yet, this is the first example of structural analysis of 1,4-diphosphabutatriene system, showing that the system is very similar to that of 1-phosphabutatriene.

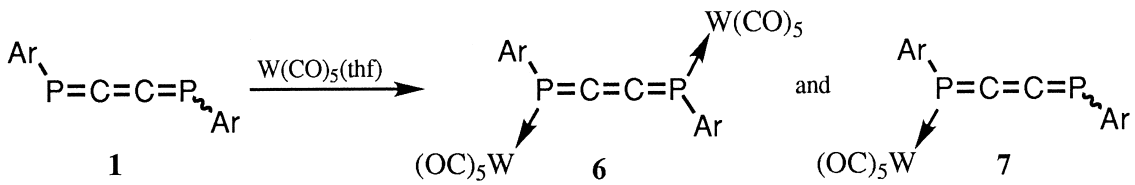
3. Experimental

All experiments were carried out under an argon atmosphere with dry solvents, unless otherwise specified. The melting points were determined with a Yanagimoto micromelting-point apparatus MP-J3 and are not corrected. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AC200P or a Bruker AM600 spectrometer. ^{31}P NMR spectra were obtained with a Bruker AC200P spectrometer using 85% H_3PO_4 as an external standard. NMR spectra were recorded at room temperature unless otherwise noted. MS spectra were taken on a JEOL HX-110. IR spectra were recorded on a Horiba FT-300 spectrometer. UV–vis spectra were obtained with a Hitachi U-3210 spectrometer. Microanalyses were performed at the Instrumental Analysis Center of Chemistry, Faculty of Science, Tohoku University. X-ray diffraction data were collected on a Rigaku AFC-7S four-circle diffractometer. The starting phosphaethenes **3a** and **3b** were prepared according to literature methods [16,17].

*3.1. Copper-mediated coupling reaction of 1-chloro-2-phosphaethenyllithium **4a***

To a solution of **3a** (108.3 mg, 0.301 mmol) in THF (8 ml) was added butyllithium (0.319 mmol, 1.68 M solution in hexane, 1 M = 1 mol dm^{-3}) at −78°C. After being stirred for 5 min, copper(II) chloride (0.270 mmol) was added to the reaction mixture and the mixture was warmed to −30°C and stirred for 1 h. The mixture was then warmed to 0°C and oxygen gas cooled at −78°C was bubbled through the mixture for 5 min (ca. 32 mmol), and a concentrated aqueous $NaHSO_3$ solution was then added. After being warmed to room temperature, ammonia (10% NH_3 in concentrated aqueous NH_4Cl solution) was added to the reaction mixture. The reaction mixture was extracted with chloroform, dried with $MgSO_4$, and the solvent was evaporated in vacuo. The residue was washed with hexane and butatriene **1** was obtained as yellow crystals (44.0 mg). Moreover, 0.6 mg of butatriene **1** and 6.8 mg of butadiene **5a** were obtained from the washings in a similar manner; butatriene **1**, 44.6 mg (54% yield) [14,15], butadiene **5a**, 6.8 mg (7% yield) [22].

When this reaction was carried out at −78°C as described in Section 3.2 for **4b**, only butadiene **5a** was



Scheme 3.

obtained as a coupling compound (46% yield). **1**: Mixture of $E/Z = 4{:}1$ isomers; yellow crystals, mp 250°C (decomp.); $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$, 298 K) *E*-**1**: $\delta = 180.6$, *Z*-**1**: $\delta = 170.0$. [2] **5a**: Yellow prisms (toluene), mp 253–254°C (decomp.); ^1H NMR (200 MHz, $CDCl_3$) $\delta = 1.34$ (18H, s, *p*-*t*-Bu), 1.51 (36H, s, *o*-*t*-Bu), and 7.42 (4H, m, *m*-Ar); $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$) $\delta = 31.3$ (s, *p*-C(CH_3)$_3$), 32.7 (brs, *o*-C(CH_3)$_3$), 35.1 (s, *p*-*C*(CH_3)$_3$), 37.9 (brs, *o*-*C*(CH_3)$_3$), 122.0 (brs, *m*-Ar), 135.5 (dd, $^1J_{PC} = 27.6$ Hz, $^4J_{PC} = 25.3$ Hz, *ipso*-Ar), 151.0 (s, *p*-Ar), 154.0 (brs, *o*-Ar), and 167.4 (dd, $^1J_{PC} = 26.5$ Hz, $^2J_{PC} = 18.0$ Hz, P=*C*); $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$) $\delta = 248.0$; IR (KBr) 1595 cm^{-1}; UV (hexane) 242 (log ε 4.44) and 366 nm (4.28); MS (70 eV, EI) *m*/*z* (rel. intensity) 650 ($M^+ + 4$; 2), 648 ($M^+ + 2$; 8), 646 (M^+; 12), 611 ($M^+ - Cl$; 4), 589 ($M^+ - {}^t$Bu; 5), 575 ($M^+ - 2Cl - 1$; 7), 401 ($ArP_2C_2Cl_2^+$; 90), 335 ($ArPC_2Cl^+$; 14), 299 ($ArPC_2^+ - 1$; 6), 275 ($ArP^+ - 1$; 34), and 57 ($^t Bu^+$; 100). Found: *m*/*z* 646.3373. Calcd. for $C_{38}H^{35}_{58}Cl_2P_2$: M, 646.3391.

3.2. Copper-mediated coupling reaction of 1-bromo-2-phosphaethenyllithium ***4b***

To a solution of **3b** (173.1 mg, 0.386 mmol) in THF (14 ml) was added butyllithium (0.391 mmol) at −100°C. After being stirred for 10 min, copper(II) chloride (0.200 mmol) was added to the reaction mixture and stirred for 1 h at −78°C. Then oxygen gas was similarly bubbled through the reaction mixture for 5 min and then a concentrated aqueous solution of $NaHSO_3$ was added. After being warmed to room temperature, ammonia (10% NH_3 in a concentrated aque-

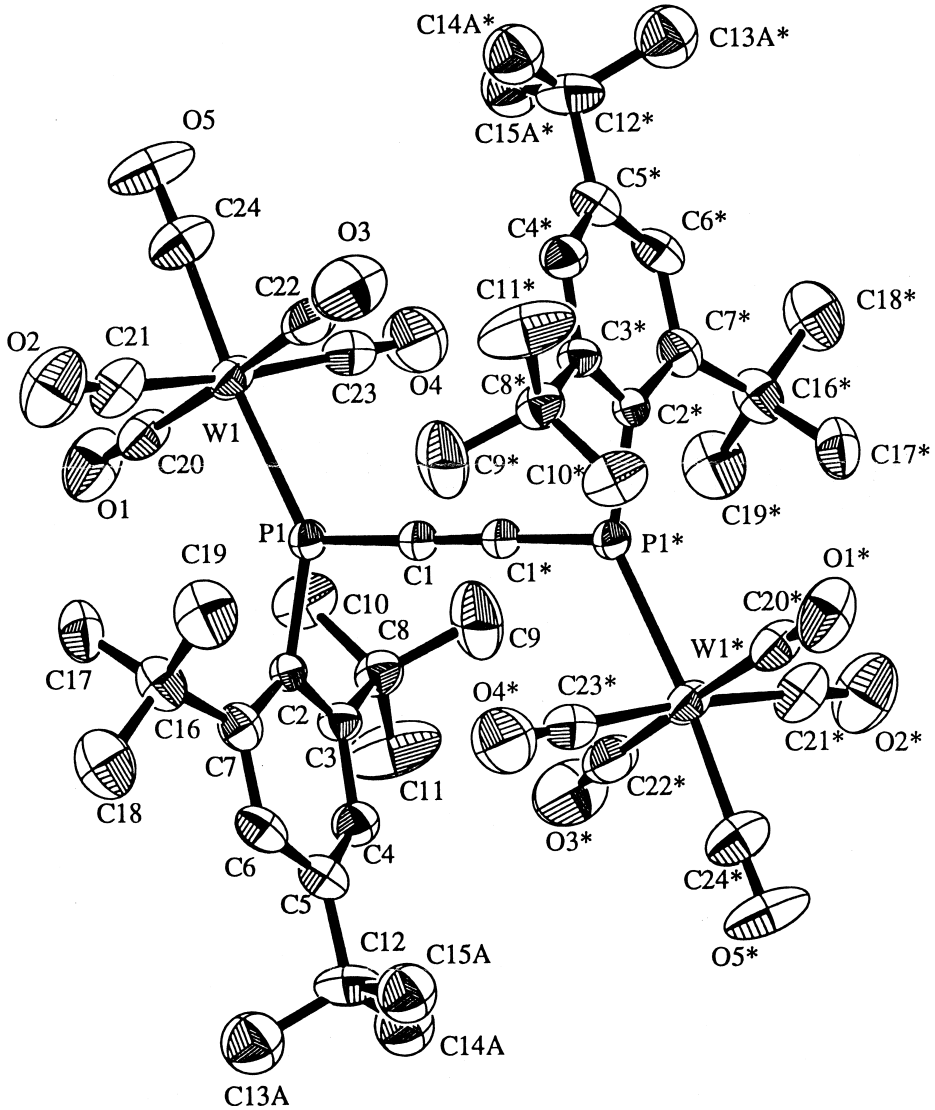


Fig. 1. Molecular structure of **6**. Hydrogen atoms are omitted for clarity. As for the disordered carbon atoms, C(13)–C(15), only those with the higher occupancy factor (0.51) are shown.

Table 2
Some selected bond lengths and angles for **6**

Bond length (Å)		Bond angle (°)	
P1–W	2.492(2)	C1–P1–C2	100.4(3)
P1–C2	1.840(6)	P1–C1–C1*	178.4(8)
P1–C1	1.656(6)	W–P1–C1	117.6(2)
C1–C1*	1.25(1)	W–P1–C2	141.9(2)
W–C20	2.049(8)	P1–W–C20	92.2(2)
W–C21	2.038(9)	P1–W–C21	97.8(2)
W–C22	2.036(9)	P1–W–C22	88.5(2)
W–C23	2.056(9)	P1–W–C23	85.8(2)
W–C24	1.997(9)	P1–W–C24	174.9(3)
		C20–W–C21	91.0(3)
		C20–W–C22	175.7(3)
		C20–W–C23	93.9(3)
		C20–W–C24	89.7(3)
		C21–W–C22	84.7(4)
		C21–W–C23	173.8(3)
		C21–W–C24	86.9(3)
		C22–W–C23	90.4(4)
		C22–W–C24	89.9(3)
		C23–W–C24	89.4(3)

Numbers in parentheses are estimated standard deviations.

ous NH_4Cl solution) was added to the reaction mixture. The reaction mixture was extracted with chloroform, dried with $MgSO_4$, and the solvent was evaporated in vacuo. The residue was washed with hexane to give butatriene **1** as yellow crystals (52.5 mg, 47% yield). Moreover, 18.9 mg of butadiene **5b** was obtained from the washings after silica-gel column chromatographic treatment (13% yield).

When this reaction was carried out at −95°C, only butadiene **5b** was obtained as a coupling compound (22% yield). **5b**: Yellow crystals, mp 220–221°C (decomp.); ^{1}H NMR (200 MHz, $CDCl_3$) $\delta = 1.34$ (18H, s, *p*-*t*-Bu), 1.53 (36H, s, *o*-*t*-Bu), and 7.42 (4H, m, *m*-Ar); ^{13}C{^{1}H} NMR (50 MHz, $CDCl_3$) $\delta = 31.3$ (s, *p*-C(CH_3)$_3$), 32.8 (dd, $^4J_{PC} = 4.0$ Hz, $^6J_{PC} = 4.0$ Hz, *o*-C(CH_3)$_3$), 35.1 (s, *p*-*C*(CH_3)$_3$), 38.0 (brs, *o*-*C*(CH_3)$_3$), 122.1 (brs, *m*-Ar), 138.1 (dd, $^1J_{PC} = 28.3$ Hz, $^4J_{PC} = 28.3$ Hz, *ipso*-Ar), 151.2 (s, *p*-Ar), 153.5 (dd, $^2J_{PC} = 2.3$ Hz, $^5J_{PC} = 2.3$ Hz, *o*-Ar), and 160.4 (dd, $^1J_{PC} = 23.1$ Hz, $^2J_{PC} = 19.9$ Hz, P=*C*); ^{31}P{^{1}H} NMR (81 MHz, $CDCl_3$) $\delta = 261.6$; IR (KBr) 1595 cm^{-1}; UV (hexane) λ_{max} (log ε) 246 (4.38) and 362 nm (4.00); MS (70 eV, EI) *m*/*z* (rel. intensity) 738 ($M^+ + 4$; 15), 736 ($M^+ + 2$; 23), 734 (M^+; 9), 677 ($M^+ - {}^t$Bu; 10), 655 ($M^+ -$ Br; 54), 601 ($M^+ -$ Br $- {}^t$Bu + 3; 31), 599 ($M^+ -$ Br $- {}^t$Bu + 1; 27), 575 ($M^+ - 2$Br − 1; 19), 368 ($ArPCBr^+ + 1$; 23), 275 ($ArP^+ - 1$; 24), 231 ($Ar^+ - CH_3 - 1$; 100), and 57 ($^t Bu^+$; 50). Found: 734.2373; Calcd. for $C_{38}H_{58}^{79}Br_2P_2$: 734.2380.

3.3. Formation of pentacarbonyltungsten complexes of *1*

To a solution of butatriene **1** (23.0 mg, 39.9 μmol) in THF (15 ml) was added a THF (5 ml) solution of $W(CO)_5$(THF) (ca. 0.6 mmol, prepared by irradiation to a THF solution of $W(CO)_6$ at 5°C for 3 h with a medium-pressure Hg lamp [26]), and being stirred for 10 h at room temperature. The solvent was evaporated in vacuo, and the residue was passed through silica gel column (hexane/Et_3N 10:1). The solvent was evaporated to leave the tungsten complex **6**. After being washed with acetone, 24.0 mg of **6** was obtained (49%). **6**: Deep blue prisms (hexane), mp 220°C (decomp.); ^{1}H NMR (200 MHz, $CDCl_3$) $\delta = 1.32$ (18H, s, *p*-*t*-Bu), 1.67 (36H, s, *o*-*t*-Bu), and 7.42 (4H, dd, $^4J_{PH} = 2.2$ Hz, $^7J_{PH} = 1.9$ Hz, *m*-Ar); ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) $\delta = 31.0$ (s, *p*-C(CH_3)$_3$), 34.5 (s, *o*-C(CH_3)$_3$), 35.1 (s, *p*-*C*(CH_3)$_3$), 39.1 (s, *o*-*C*(CH_3)$_3$), 123.4 (s, *m*-Ar), 129.5 (s, *ipso*-Ar), 152.4 (s, *p*-Ar), 155.7 (s, *o*-Ar), 175.0 (dd, $^1J_{PC} = 30.0$ Hz, $^2J_{PC} = 26.0$ Hz, P=*C*), 195.5 (brs, CO_{eq}), and 199.8 (d, $^2J_{PC} = 18.9$ Hz, CO_{ax}); ^{31}P{^{1}H} NMR (81 MHz, $CDCl_3$) $\delta = 105.0$ (satellite, $^1J_{PW} = 169.6$ Hz, $^4J_{PW} = 109.0$ Hz); IR (KBr) 2065, 2000, 1955, and 1940 cm^{-1}; UV–vis (hexane) λ_{max} (log ε) 206 (5.05), 230 (5.05) 318 (4.05), and 609 nm (4.83); FAB-MS *m*/*z* 1225 ($M^+ + 1$) and 548 ($ArPC-CPAr^+ - 2Me + 2$). Anal. Found: C, 47.25; H, 4.62%; Calcd. for $C_{48}H_{58}O_{10}P_2W_2$: C, 47.08; H, 4.77%.

The reaction of **1** with 1 equivalent of $W(CO)_5$(THF) gave monocoordinated complex **7** together with bis-tungsten complex **6** and the starting material **1**. **7**: ^{31}P{^{1}H} NMR (81 MHz, $CDCl_3$) $\delta = 181.5$ and 105.3 (ABq, $^3J_{PP} = 315.1$ Hz).

3.4. X-ray structure determination of [ArP=C=C=PAr][W(CO)$_5$]$_2$ (6)

The complex **6** was recrystallized from hexane. $1/2 \cdot C_{48}H_{58}O_{10}P_2W_2$, $M_r = 612.31$, monoclinic, space group $P2_1/n$, $a = 11.116(6)$, $b = 12.65(1)$, $c = 18.973(5)$ Å, $\beta = 104.56(3)°$. $V = 2581(2)$ Å^3, $Z = 4$, $D_c = 1.575$ g cm^{-3}, $\mu = 45.70$ cm^{-1}. The 4792 unique reflections with $2\theta < 50.0°$ were recorded on a four-circle diffractometer using graphite-monochromated Mo-Kα radiation. Of these, 3443 with $F > 3\sigma(F)$ were judged as observed. The structure was solved using SHELXS86 [27]. The methyl carbons C(13), C(14), C(15) on C(12) are disordered (occupancy factors for the dominant: 0.51). The nonhydrogen atoms except the disordered C-atoms were refined anisotropically. Hydrogen atoms were included but not refined. $R = 0.033$, $R_w = 0.041$. Further details of the crystal structure investigation are available from the Cambridge Crystallographic Data center, 12 Union Road, CB-Cambridge CB2 1EZ (UK).

[4] Satellite signals were tentatively assigned as $^1J_{PW}$ and $^4J_{PW}$.

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