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Looking for 1,3-di- and 1,2,3-triphosphindolide ions

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Summary — The reaction of 1,2-bis(lithiophenylphosphino)benzene **1** with dichloromethane leads to a *cis–trans* mixture of 1,3-diphenyl-2,3-dihydro-1*H*-1,3-benzodiphosphole **2**. The reaction of **2**, first with Et_2NLi, then with an excess of lithium leads to the parent 1,3-diphosphindolide ion **4**. Starting from **1** and tetrachloroethylene, both the expected dibenzotetraphosphafulvalene **7** and its 6-membered ring isomer **6** are obtained. One stereoisomer of each has been characterized by X-ray crystal structure analysis. Whereas **6** is strain-free, **7** displays some strain, thus explaining the competition between **6** and **7** in the reaction. Compound **7** yields the corresponding dianion **8** upon cleavage of its four P–Ph bonds by an excess of lithium in THF. An attempted synthesis of the 1,2,3-triphosphindolide ion has failed.

1,2-bis(lithiophenylphosphino)benzene / 2,3-dihydro-1*H*-1,3-benzodiphosphole / phosphafulvalene / 1*H*-1,3-benzodiphospholide ion / 1*H*-benzotriphospholide ion

Résumé — À la recherche des ions 1*H*-1,3-benzodiphospholure et 1*H*-benzotriphospholure. La réaction du 1,2-bis(lithiophénylphosphino)benzène **1** avec le dichlorométhane conduit à un mélange *cis–trans* du 1,3-diphényl-2,3-dihydro-1*H*-1,3-benzodiphosphole **2**. La réaction de **2** d'abord avec Et_2NLi, puis avec un excès de lithium donne l'ion 1*H*-1,3-benzodiphospholure parent **4**. En partant de **1** et du tétrachloroéthylène, on obtient le 2,2′-bi-1*H*-1,3-benzodiphospholylidene **7** attendu et son isomère cyclique à six chaînons **6**. Un stéréoisomère de chacun d'eux a été caractérisé par détermination de structure aux rayons X. Alors que **6** ne présente aucune contrainte, **7** montre une certaine rigidité expliquant ainsi la formation compétitive de **6** et **7**. Le composé **7** donne le dianion correspondant **8** après coupure de ses quatre liaisons P–Ph par un excès de lithium dans le THF. En revanche, l'essai de synthèse de l'ion 1*H*-benzotriphospholure a échoué.

1,2-bis(lithiophénylphosphino)benzène / 2,3-dihydro-1*H*-1,3-benzodiphosphole / phosphafulvalène / ion 1*H*-1,3-benzodiphospholure / ion 1*H*-benzotriphospholure

Introduction

Presently, the whole series of polyphospholide ions from the 1,2- and 1,3-di- to the 1,2,3,4,5-penta-phospholide has been reported in the literature [1–6]. In the benzo-annellated series, only phosphonium-substituted 1,2-P_2 derivatives have been recently described by Schmidpeter et al [7]. In the course of our systematic study of the synthesis and chemical properties of polyphospholide ions [8], we wished to investigate the effect of benzo-annellation upon the aromaticity of these highly delocalised phosphorus analogues of cyclopentadienide ion [9–11]. Hereafter, we describe our attempts to synthesize the 1*H*-1,3-benzodiphospholide and 1*H*-benzotriphospholide ions together with the related 2,2′-bi-1*H*-1,3-benzodiphospholide dianion.

Results and discussion

In order to get the parent 1*H*-1,3-benzodiphospholide ion, we successfully transposed our preferred route to 1,3-diphospholides [2b] (eq 1). Our starting point was the dilithio derivative of 1,2-bis(phenylphosphino)-benzene **1** [12]. Ring closure of **1** with dichloromethane afforded the 2,3-dihydro-1*H*-1,3-benzodiphosphole **2** as a 2:1 mixture of *cis* and *trans* isomers. In the ^1H NMR spectra, the CH_2 group appears as the AB part of a ABX_2 spin system (X = P) for *cis*-**2** and as the AA′ part of a AA′XX′ spin system for *trans*-**2**. The anionisation of **2** to **3** is necessary in order to prevent the collapse of the 5-membered ring upon cleavage of the P–Ph bonds by lithium. The ion **4** was characterized by its ^1H, ^{13}C and ^{31}P NMR spectra together with its negative ion mass spectrum. When compared with that of lithium 4,5-diphenyl-1,3-diphospholide **5** (eq 2) [13], the spectral data of **4** shows two main differences. The ^{31}P resonance is shifted upfield: $\delta^{31}P$ +153.4 (**4**) versus +193.3 (**5**) in THF. The reverse trend is observed for the *C*H resonances: $\delta^{13}C(H)$ +168.8 (**4** in C_6D_6) versus +158 (**5** in C_4D_8O). Conversely, the couplings are closely similar in both cases: $^1J_{(HC-P)}$ = 54 (**4**) and 52.4 Hz (**5**). This probably means that the benzo-annellation does not significantly affect the delocalisation within the 1,3-diphospholide ion.

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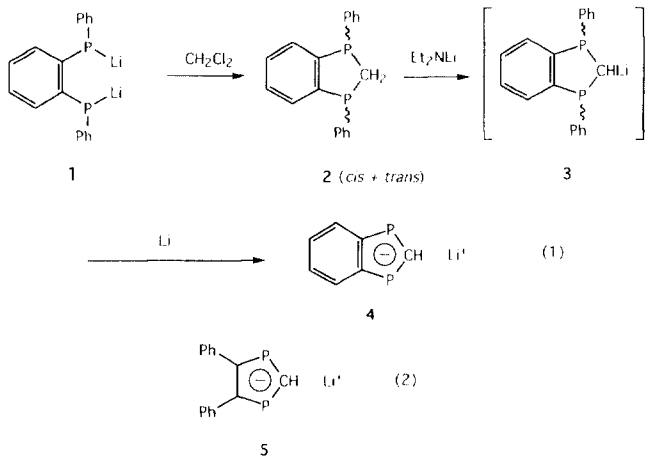
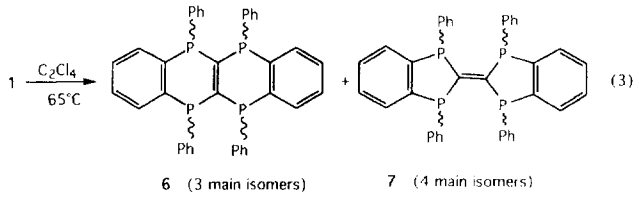


In the same vein, we were also able to duplicate the chemistry which led us to tetraphosphafulvalenes and their dianions [14, 15]. The reaction of **1** with tetrachloroethylene is slow and leads to a complicated mixture of isomeric products in 10% overall yield (eq 3).



The identification of **6** and **7** was achieved by X-ray crystal structure analysis of one isomer of each product (figs 1 and 2). In the isomer of **6**, the central 6-membered rings are boat- shaped and are *trans* to

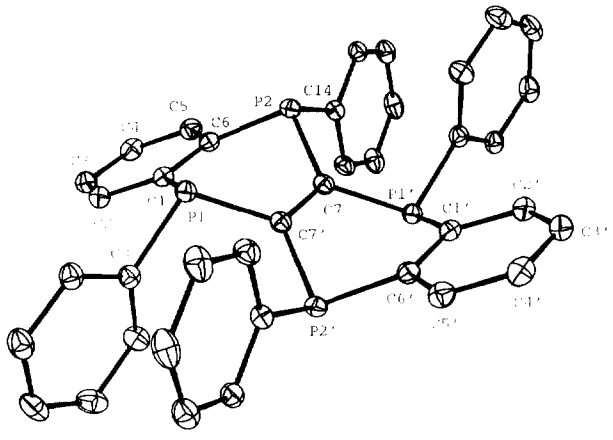


Fig 1. ORTEP drawing of one isomer (*D*) of **6**, as determined by a single crystal X-ray diffraction study. Ellipsoids are scaled to enclose 50% of the electronic density. Hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (deg): P(1)-C(1) 1.837(2), P(1)-C(7′) 1.840(2), P(1)-C(8) 1.834(2), C(1)-C(6) 1.402(3), C(6)-P(2) 1.836(2), P(2)-C(7) 1.832(2), P(2)-C(14) 1.826(2), C(7)-C(7′) 1.365(4), C(1)-P(1)-C(7′) 100.6(1), C(1)-P(1)-C(8) 100.5(1), C(7′)-P(1)-C(8) 105.64(9), P(1)-C(1)-C(6) 119.2(2), C(1)-C(6)-P(2) 119.1(2), C(6)-P(2)-C(7) 101.1(1), C(6)-P(2)-C(14) 99.9(1), C(7)-P(2)-C(14) 104.6(1), P(1′)-C(7)-P(2) 120.3(1), P(1′)-C(7)-C(7′) 119.6(2), P(2)-C(7)-C(7′) 120.1(2).

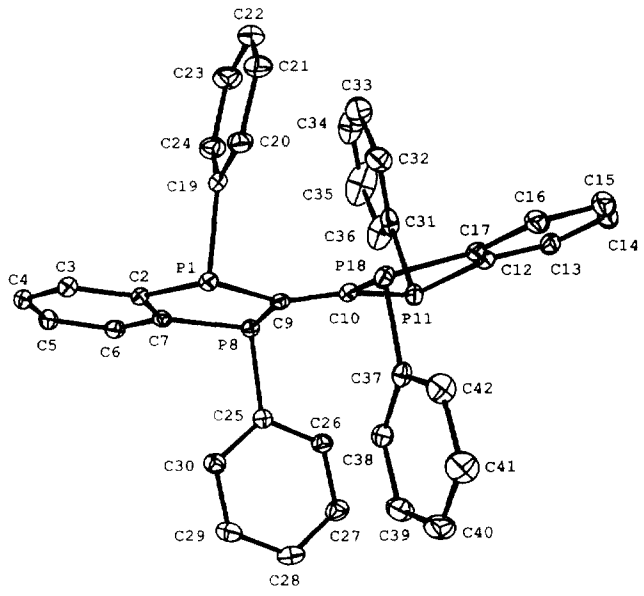
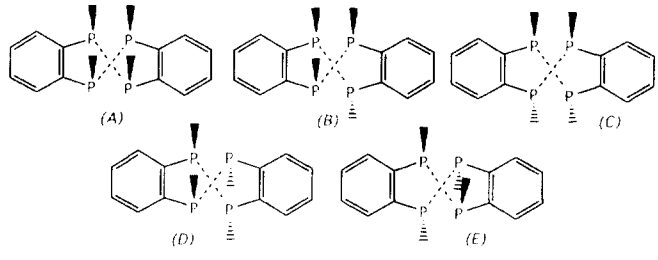


Fig 2. ORTEP drawing of one isomer (*E*) of **7**, as determined by a single crystal X-ray diffraction study. Ellipsoids are scaled to enclose 50% of the electronic density. Hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (deg): P(1)-C(2) 1.818(2), P(1)-C(9) 1.845(2), P(1)-C(19) 1.842(2), P(8)-C(7) 1.818(2), P(8)-C(9) 1.840(2), P(8)-C(25) 1.847(2), P(11)-C(10) 1.832(2), P(18)-C(10) 1.838(2), C(2)-C(7) 1.405(3), C(9)-C(10) 1.343(3), C(2)-P(1)-C(9) 94.29(9), C(2)-P(1)-C(19) 102.14(9), C(9)-P(1)-C(19) 97.26(9), C(7)-P(8)-C(9) 94.50(9), C(7)-P(8)-C(25) 102.60(9), C(9)-P(8)-C(25) 98.48(9), P(1)-C(9)-P(8) 115.1(1), P(1)-C(9)-C(10) 121.6(2), P(8)-C(9)-C(10) 123.2(2), P(11)-C(10)-P(18) 114.9(1).

each other. In each ring, the two P–Ph bonds are *cis*. The angle between the benzo P_2 planes and the plane of the central C=C double bond is 124.6°. The 6-membered rings show no strain at P and at the central C=C bond. In the isomer of **7**, the P–Ph bonds are in an all *trans* disposition as in the already described tetraphosphafulvalene [14]. The two planes of the 5-membered rings form an angle of 21.8°. Some strain at P and at the central C=C bond is obvious from the angle values. At this point, it must be underlined that in compounds such as **6** and **7** with 4 phosphorus atoms linked by two different types of functions, 5 isomers are possible as shown in scheme 1.

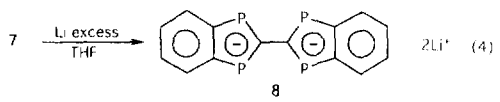


Scheme 1. The five possible isomers of compounds **6** and **7**.

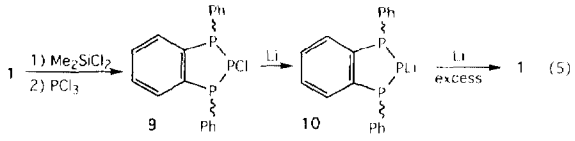
When the phosphorus phenyl substituents of **1** lie on the same side of the benzo-P_2 plane, the attack of 2 mol of **1** on C_2Cl_4 results in the formation of isomers *A* and *D*. When the phenyl substituents of **1** are on each side of

the benzo-P_2 plane, the reaction yields isomers *C* and *E*. These four isomers possess at least two symmetry elements, thus making all phosphorus atoms equivalent. On the other hand, isomer *B* is formed when the 2 mol of **1** have both conformations, hence, all phosphorus atoms are inequivalent. Similar cases have already been discussed in the literature [16]. In our case, the recorded X-ray structures correspond to isomers **6***D* and **7***E*.

As for the tetraphosphafulvalene [15], it is possible to cleave all the four P–Ph bonds of **7** by an excess of lithium in THF (eq 4).



The dianion **8** displays a ^{31}P resonance at 135 ppm versus 167.3 for the already described tetraphenyl–tetraphosphafulvalene dianion [15]. No additional chemistry was performed in view of the low yield of the synthesis of **7**. We also tried to transpose the chemistry which led us to the discovery of the first practical synthesis of 1,2,3-triphospholide ions [3] (eq 5).



According to the monitoring of the reaction mixture by ^{31}P NMR spectroscopy, all the preliminary steps go smoothly until the attempted cleavage of the two P–Ph bonds by lithium. A collapse of the 5-membered ring was observed in this last step, leading to the starting dianion **1**. This observation seems to suggest that the benzo-annellation severely reduces the electronic delocalisation within **10** by comparison with its non-benzoannellated analogue [3].

The most significant result of this study is the synthesis of the 1*H*-1,3-benzodiphospholide ion **4**. The fair yields of the various steps together with the simplicity of the overall scheme (eq 1) mean that the chemistry of this new aromatic species can now be investigated seriously.

Experimental section

All the reactions were performed under argon; the solvents were purified, dried and degased by standard techniques. ^1H, ^{13}C and ^{31}P NMR spectra were recorded on a Bruker AC 200 SY spectrometer operating at 200.13, 50.32 and 81.01 MHz, respectively. All chemical shifts are reported in ppm downfield from internal TMS (^1H and ^{13}C) and external 85% H_3PO_4 (^{31}P). Mass spectra (EI) were obtained at 70 eV with a MS engine by the direct inlet method. Elemental analyses were performed by the 'Service d'analyse du CNRS'.

1,3-Diphenyl-2,3-dihydro-1H-1,3-benzodiphosphole **2**

A solution of compound **1** [12] (5.4 mmol) in 8 mL of dry THF was cooled to −30 °C and treated dropwise with 9 mmol of freshly distilled CH_2Cl_2 (1 mL). After the reaction mixture was slowly warmed to room temperature, the solvent was evaporated and the residue was chromatographed on silica gel (70–230 mesh) with a degased hexane/toluene (70:30) mixture. Compound **2** is obtained as a mixture of 2 isomers *cis/trans* (2:1) with a yield of 79% (1.3 g).

^{31}P NMR (CD_2Cl_2) δ +12.8, *cis* isomer +15.2 *trans* isomer.

^1H NMR (CD_2Cl_2) δ 2.14 (Part B of a ABX_2 spin system, 1H, $^2J_{HAHB}$ = 14.7 Hz, $^2J_{HBPX}$ = 2.9 Hz, PCH_BH_AP), 2.72 (Part A of a ABX_2, 1H, $^2J_{HAHB}$ = 14.7 Hz, $^2J_{HAPX}$ = 24.8 Hz, PCH_BH_AP), 6.9–7.7 (m, 14H, phenyl), *cis* isomer; 2.33 (Part AA′ of a AA′XX′ spin system, 2H, Σ J_{HAPX} + $J_{HAPX'}$ = 15 Hz, PCH_2P), 6.9–7.7 (m, 14H, phenyl), *trans* isomer.

^{13}C NMR (CD_2Cl_2) δ 24.38 (t, $^1J_{CP}$ = 24.6 Hz, *P*CP), 128–141 (C Arom), 146.86 (pt, *P*CCP), *cis* isomer 28.11 (t, $^1J_{CP}$ = 21.5 Hz, *P*CP), 128–141 (C Arom), 147.92 (s, *P*CCP), *trans* isomer.

MS *m/z*: 306 (100).

Anal $C_{19}H_{16}P_2$: calc (%) C 74.51, H 5.35; found: C 74.92, H 5.27.

2-Lithio-1,3-diphenyl-2,3-dihydro-1H-1,3-benzodiphosphole **3**

A solution (0.7 M) of 3 equiv of LDA in hexane/THF was added dropwise to 4.2 mmol of **2** (1.3 g) in 20 mL of dry THF cooled to −80 °C. The reaction mixture was slowly warmed to room temperature. After evaporation of the solvents and iPr_2NH under vacuum, compound **3** is obtained as a mixture of 2 isomers *cis/trans* (2:1).

^{31}P NMR (THF d_8) δ −37.3 ($^2J_{HP}$ = 38 Hz), *cis* isomer +51.9 ($^2J_{HP}$ = 0 Hz) *trans* isomer.

1H-1,3-Benzodiphospholide ion **4**

Dry THF (20 mL) and 7 equiv of lithium (200 mg, 28 mmol) were added to 4.2 mmol of **3**. The mixture was stirred at room temperature for one week. The intermediate dianion $(C_6H_4P_2C)^{2-}$ (^{31}P NMR in THF δ 186 ppm) is protonated at room temperature by NH_4Cl (500 mg) for 2 h. The solution was filtered then evaporated on vacuum line.

^{31}P NMR (THF d_8) δ +155.6 ($^2J_{HP}$ = 36 Hz).

^1H NMR (C_6D_6) δ 7.2 (s broad, *CH* benzo), 8.8 (s broad, *CH* benzo) 9.77 (t, $^2J_{HP}$ = 36 Hz, PC*H*P).

^{13}C NMR (C_6D_6) δ 116.5 and 130.5 (pt, *C*H benzo), 161.5 (pt, *P*CCP), 168.8 (t, $^1J_{CP}$ = 54 Hz, *P*CHP).

Mass spectrum negative ion *m/z*: 151 (10).

4,5-Diphenyl-1H-1,3-diphosphole **5**

It was obtained as described in [13].

^{31}P NMR (THF d_8) δ +193.3 ($^2J_{PH}$ = 37 Hz).

^1H NMR (THF d_8) δ 8.24 (t, $^2J_{HP}$ = 37 Hz, PC*H*P).

^{13}C NMR (THF d_8) δ 158 (t, $^1J_{CP}$ = 52.4 Hz, *P*CHP), 158.5 (Part X of an ABX spin system, A and B were the phosphorus atoms, $\nu_A - \nu_B$ = 4 Hz, $^1J_{AX}$ = −30 Hz, $^2J_{XB}$ = −4.5 Hz, $^2J_{AB}$ = +22 Hz, $P_AC_XCP_B$).

5,6,11,12-Tetraphenyl-5,6,11,12-tetrahydro-1,4-benzodiphosphinino[2,3-b][1,4]benzodiphosphinine **6** *and 1,1′,3,3′-tetraphenyl-2,2′,3,3′-tetrahydro-2,2′-bi-1H-1,3-benzodiphospholylidene* **7**

A mixture of 2.3 mmol of **1** and 30 mL of dry THF was warmed to 50 °C and treated dropwise with pure tetrachloroethylene (1 mL) in order to keep a slight ebullition. The solvent was evaporated and the residue was

chromatographed on silica gel (70–230 mesh) with degased hexane/toluene (60:40) mixture. A mixture of different isomers of **6** and **7** was obtained with a yield of 21% (0.3 g). The white isomer **6**(*D*) crystallizes from a mixture of hexane/toluene (60:40), and was separated by filtration. The mother liquor was concentrated and cooled, a new quantity of isomer **6**(*D*) and the yellow isomer **7**(*E*) crystallize together. These isomers were afterwards separated by hand.

6(*D*): Mp >250 °C.

^{31}P NMR ($CDCl_3$) δ −2.5.

^{1}H NMR ($CDCl_3$) δ 6.5–7.5 (m, 28H arom).

^{13}C NMR ($CDCl_3$) δ 125–129.5 (m, *C*H arom), 136.9–137.5 (m, *C*q arom).

Mass spectrum *m*/*z*: 608 (M^+, 80%), 531 (M − C_6H_5, 60%).

6(*A*, *C*, *E*):

^{31}P NMR ($CDCl_3$) δ +0.5, −6, −9.7.

6(*B*):

^{31}P NMR ($CDCl_3$) δ +6.6 (ddd, J_{PP} = 15, 22 and 122 Hz), +1.75 (d, J_{PP} = 15 Hz), −3.0 (dd, J_{PP} = 22 and 5 Hz), −21.1 (dd, J_{PP} = 5 and 122 Hz).

7(*E*): Mp 238 °C.

^{31}P NMR ($CDCl_3$) δ +27.9.

^{1}H NMR ($CDCl_3$) δ 7–7.7 (m, 28H arom).

^{13}C NMR ($CDCl_3$) δ 126–138 (m, *C* arom), 146.78 (s, P*C*CP), 160.63 (pt, P_2C=CP_2).

Mass spectrum *m*/*z*: 608 (M^+, 60%), 531 (M–C_6H_5, 30%).

7(*A*, *C*, *D*):

^{31}P NMR ($CDCl_3$) δ +28.8, +18.7, +16.5.

7(*B*):

^{31}P NMR ($CDCl_3$) δ +28.85 (ddd, J_{PP} = 9, 24 and 90 Hz), +25.3 (ddd, J_{PP} = 21, 24 and 67 Hz), +20.7 (ddd, J_{PP} = 9, 37 and 67 Hz), +15.6 (ddd, J_{PP} = 21, 37 and 90 Hz).

2,2′-Bi-1H-1,3-benzodiphospholide ion ***8***

A mixture of isomer **7**(*E*) (0.2 g) and 5 mL of dry THF was stirred at ambient temperature with 25 mg of lithium. The reaction was complete after 1 h.

^{31}P NMR (THF) δ +135.

2-Chloro-1,3-diphenyl-2,3-dihydro-1H-benzotriphosphole ***9***

A mixture of 2.3 mmol of **1** in 10 mL of dry THF was cooled to −30 °C and treated dropwise with 1 equiv of pure Me_2SiCl_2 (270 μL), the mixture was stirred 15 min (^{31}P NMR δ −47.8 and −50.4 *cis* and *trans* isomers), then 1 equiv of pure PCl_3 (200 μL) was added at the same temperature. The reaction mixture was slowly warmed to room temperature. After evaporation of the solvent under vacuum, the residue was used without purification.

^{31}P NMR ($CDCl_3$) δ +84.3 (part A of a AB_2 spin system, $^{1}J_{PP}$ = 310 Hz, *P*Cl), +49.4 (part B of a AB_2 spin system, $^{1}J_{PP}$ = 310 Hz, *P*Ph); *cis* isomer 60%: +98.7 (part A of a AXY spin system, $^{1}J_{PP}$ = 281 and 288 Hz, *P*Cl), +41.0 (part X of AXY spin system, $^{1}J_{PP}$ = 288 Hz, $^{2}J_{PP}$ = 0 Hz, *P*Ph), +22.3 (part Y of a AXY spin system, $^{1}J_{PP}$ = 281 Hz, $^{2}J_{PP}$ = 0 Hz, *P*Ph).

Attempted synthesis of the 1H-benzotriphospholide ion

The quantity of the crude product **9** was dissolved in 5 mL of dry THF; 50 mg of lithium was added and the mixture was stirred at ambient temperature. After 15 min the brown solution showed the formation of **10** [^{31}P NMR in THF δ −172 (t, $^{1}J_{PP}$ = 374 Hz, P^-), +33 (d, *P*Ph)]. After 1 h, only the resonance of compound **1** was observed (^{31}P NMR in THF δ −40).

X-ray structure determinations

All data sets were collected on an Enraf Nonius CAD4 diffractometer using Mo Kα (λ = 0.71073 Å) and a graphite monochromator. The crystal structures were solved by direct methods using SIR92 and refined with the Enraf Nonius MOLEN package using reflections having $F^2 < 3.0\sigma(F^2)$. The hydrogen atoms were included as fixed contributions in the final stages of least-squares refinement while using anisotropic temperature factors for all other atoms. Crystal data are assembled in table I.

Table I. Crystallographic data.

Compound	**6**	**7**
Formula	$C_{38}H_{28}P_4$	$C_{38}H_{28}P_4$
Space group	*Pbca* (61)	*P* − 1 (2)
Data collection temperature (K)	123	123
a (Å)	8.001(1)	9.783(1)
b (Å)	16.530(2)	10.107(1)
c (Å)	22.507(2)	16.847(2)
α (°)	–	94.92(1)
β (°)	–	91.85(1)
γ (°)	–	113.07(1)
V (Å^3)	2976.57(99)	1522.98(65)
Z	4	2
d_{calc} (g/cm^3)	1.358	1.327
μ (cm^{-1})	2.7	2.7
Maximum 2θ	60.0	60.0
No of reflections measured	4869	9331
Reflections included	2625	5593
Parameters refined	246	379
Unweighted agreement factor	0.039	0.046
Weighted agreement factor	0.054	0.070
GOF	1.00	1.22
Convergence, largest shift/error	0.01	0.00

Supplementary material data have been deposited with the British Library, Document Supply Center at Boston Spa, Wetherby, West Yorkshire, LS23 7BQ, UK as supplementary publication No. SUP 90479 and is available on request from the Document Supply Center.

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