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First structural characterisation of 1,2,4-selenadiphosphole and 1,2,4-telluradiphosphole ring systems. Crystal and molecular structures of the η^1-complexes $[M(CO)_5(P_2SeC_2Bu^t_2)]$ ($M = Cr, W$) and $[W(CO)_5(P_2TeC_2Bu^t_2)]$

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Abstract

The molecular geometries of the 1,2,4-selenadiphosphole and the 1,2,4-telluradiphosphole aromatic ring systems, established by single crystal X-ray diffraction studies on their metal pentacarbonyl complexes, are presented and discussed together with theoretical data. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Selenadiphosphole; Telluradiphosphole; Complexes; Chromium; Tungsten; Carbonyls

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1. Introduction

Two very recent independent reports [1,2] describe the synthesis of the first example of a 1,2,4-selenadiphosphole, $P_2SeC_2Bu^t_2$ **1**, whose formulation was mainly based on elemental analysis and NMR spectroscopic data. Compond **1**, which can be made (i) by treatment of the 1,2,4-triphosphole $P_3C_2Bu^t_2CH(SiMe_3)_2$ with Se_8; (ii) from the reaction between Bu^tCP and the 1,2,3-selena diazole $C_2H_2N_2Se$; and (iii) by treating a DME solution of $LiSbP_2C_2Bu^t_2$ with $Se(S_2CNEt_2)_2$, [3] is a yellow oil and no structural data are available on this novel type of aromatic [4] ring system. We now describe the solid state structures of Cr and W pentacarbonyl metal complexes of **1** which establish the geometry of the 1,2,4-selenadiphosphole ring. We also report the structure of the corresponding 1,2,4-telluradiphosphole-$W(CO)_5$ complex.

2. Results and discussion

Treatment of a THF solution of **1** with $[M(CO)_5THF]$ ($M = Cr, W$) followed by column chromatography gave the yellow complexes $[M(CO)_5$-$P_2SeC_2Bu^t_2]$ $M = Cr$ **2**, and $M = W$ **3**, in 36 and 27% yield, respectively (Scheme 1).

The mode of attachment of the $[M(CO)_5]$ fragment to the 1,2,4-selenadiphosphole ring was established as being via the phosphorus P(A) atom bonded directly to selenium. The $^{31}P\{^1H\}$-NMR spectrum of both **2** and **3** shows a pattern of lines typical for an [AB] spin system with satellites due to further coupling to the ^{77}Se nu-

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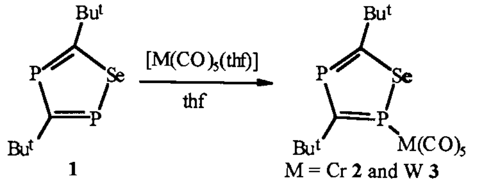


Scheme 1.

cleus (I = 1/2, 7.58%). Chemical shift and coupling constant data for **1**, **2** and **3** are listed in Section 3.

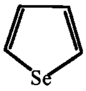
Additional evidence for the proposed structures of **3** comes from its ^{77}Se-NMR spectrum which shows a doublet of doublets pattern and the observation of tungsten satellites (^{183}W, I = 1/2, 14.40%) around the resonance of P(A). The magnitude of the $^1J_{P(A)W}$ coupling constant lies in the expected range for complexes containing an η^1-ligated [W(CO)$_5$] fragment (234–276 Hz) [5,6].

As expected, ^{1}H-NMR spectra of complexes **2** and **3** each show two resonances corresponding to the different types of Bu^t groups. Infrared spectra of compounds **2** and **3** in the ν_{CO} stretching region consist of three bands and data are comparable with those for other closely related complexes of azaphospholes [6]. Further evidence supporting the formulation of both **2** and **3** is the observation in their mass spectra of the expected parent ions at $m/z = 472$ and 604, respectively and peaks corresponding to loss of two, four and five CO groups ($m/z = 416$, 360, and 332 for **2**) and ($m/z = 548$, 492, and 464 for **3**) as well as a peak at $m/z = 280$ for **1**.

Suitable single crystals of **2** and **3** were grown from a hexane solution, and single crystal X-ray diffraction studies established their molecular structures (Figs. 1 and 2), and confirm the structures proposed from the solution NMR data. Both **2** and **3** exhibit a planar $P_2SeC_2Bu^t_2$ ring and some equalisation of the ring bond lengths expected for an aromatic selenadiphosphole ring system.

The ring angles at carbon are close to 120° while those at P or Se are closer to 100° and the ring geometry is essentially independent of the nature of the [M(CO)$_5$] fragment to which it is attached (see Table 1).

The Se–P and Se–C bond lengths (a) and (e) for **2** and **3** are significantly shorter than Se–P and Se–C single bonds (2.273 and 1.972 Å, respectively), [7] and are in good agreement with values obtained from theoretical calculations on ring systems **4**–**6** [4], suggesting that the ring geometry of **1** may be very similar to that in **2** and **3**.



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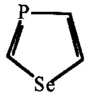

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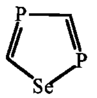

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The relative stabilities of the two possible η^1-P-bonded chromium pentacarbonyl complexes were calculated at the HF/3-21G(*) and at the B3LYP/3-21G(*)

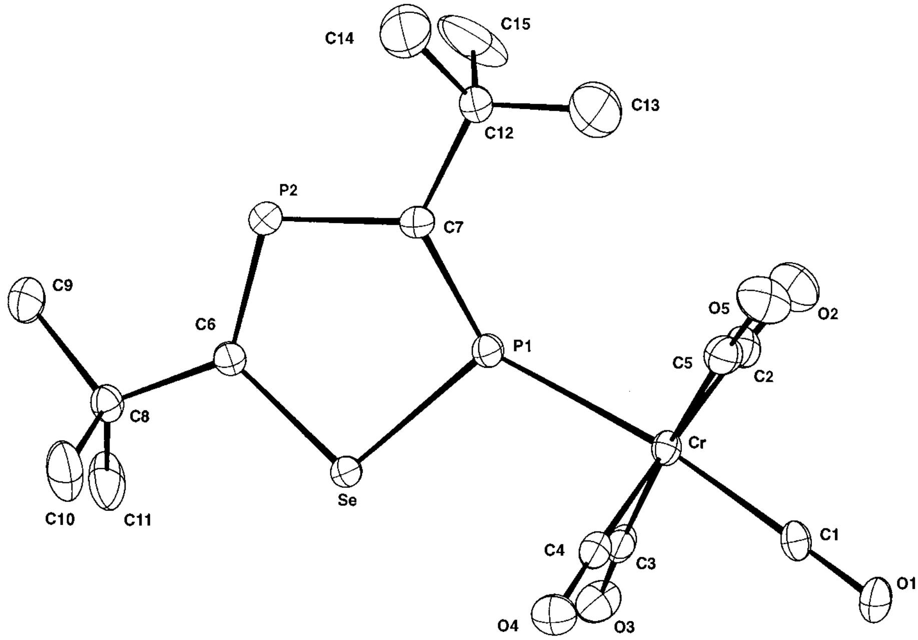


Fig. 1. Molecular structure of **2** with selected bond lengths (Å) and bond angles (°). Se–C(6) 1.873(6); Se–P(1) 2.182(2); Cr–P(1) 2.359(2); P(1)–C(7) 1.695(6); P(2)–C(6) 1.681(6); P(2)–C(7) 1.774(6); C(6)–Se–P(1) 96.2(2); C(1)–Cr–P(1) 173.7(2); C(7)–P(1)–Se 101.1(2); Se–P(1)–Cr 111.83(7); C(6)–P(2)–C(7) 103.7(3); P(1)–C(7)–P(2) 120.0(3); P(2)–C(6)–Se 118.9(3).

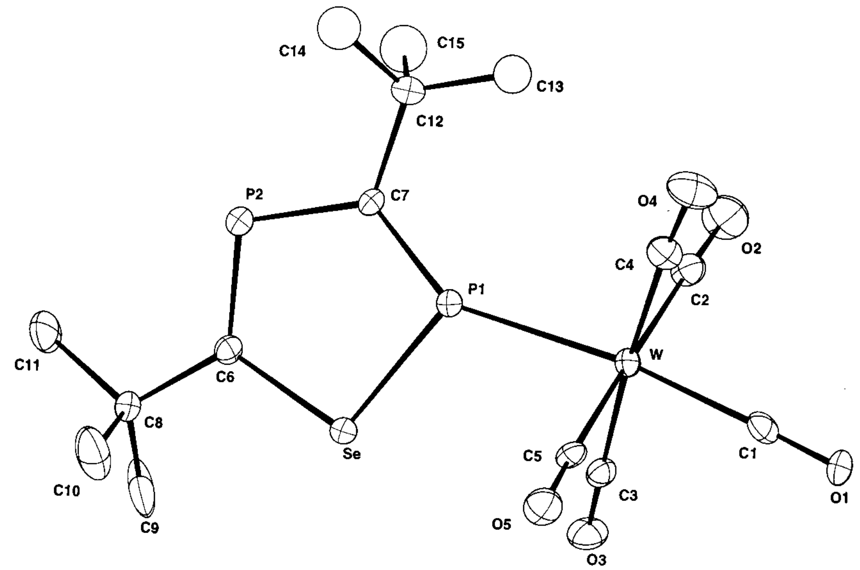


Fig. 2. Molecular structure of **3** with selected bond lengths (Å) and bond angles (°). Se–C(6) 1.868(6); Se–P(1) 2.180(2); W–P(1) 2.489(2); P(1)–C(7) 1.687(6); P(2)–C(6) 1.690(6); P(2)–C(7) 1.772(6); C(6)–Se–P(1) 95.9(2); C(7)–P(1)–Se 101.3(2); Se–P(1)–W 111.91(7); C(6)–P(2)–C(7) 103.1(3); P(1)–C(7)–P(2) 120.4(4); P(2)–C(6)–Se 119.3(3).

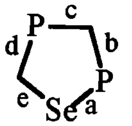
level of the theory[1], and at both levels the preferred site is the P4 position rather than that found in **2**.

However, the energy difference is quite small between the isomeric structures (1.5, 1.6 and 3.1 kcal mol^{-1}, respectively at the HF/3-21G(*), B3LYP/3-21G(*) and B3LYP/6-311 + G**//B3LYP/3-21G(*) levels). Attachment of Bu^t groups to both ring C atoms changes the relative stabilities of the two complexes at the B3LYP/3-21G* level and complex **2** is more stable by 6.5 kcal mol^{-1} in accord with the experimental data. The different relative stabilities of the H and the Bu^t substituted complexes presumably can be attributed to steric congestion. The calculated structural parameters of the ring in the different complexes (excluding the Bu^t substituents) are compared in Table 2 together with the data for the hypothetical ($P_2SeC_2H_2$) **1′** and the experimental data for **2**. It can be seen that the bond lengths do not change significantly upon complexation, likewise the position of attachment of the ring to Cr has little effect on either the geometry or on the relative energies.

The new 1,2,4-telluradiphosphole $P_2TeC_2Bu^t_2$ **7** which was made from $LiSbP_2C_2Bu^t_2$ and $Te(S_2CNEt_2)_2$ [3], was converted into its yellow $[W(CO)_5]$ adduct **8** (40%) (Scheme 2) and detailed ^{1}H-, ^{31}P- and ^{125}Te-NMR spectroscopic studies on the latter established an analogous structure to **2** and **3**.

This was subsequently confirmed by a single crystal X-ray analysis of **8** whose molecular structure is shown in Fig. 3. The P–W bond distances in **3** and **8** are

Table 1
Selected bond lengths (Å) and bond angles (°) for the $P_2SeC_2Bu^t_2$ ring in complexes **2** and **3**

		2	3
Bond lengths	P(1)–Se(a)	2.182(2)	2.180(2)
	P(1)–C(7)(b)	1.695(6)	1.687(6)
	P(2)–C(7)(c)	1.774(6)	1.772(6)
	P(2)–C(6)(d)	1.681(6)	1.690(6)
	C(6)–Se(e)	1.873(6)	1.868(6)
Bond angles	C(6)–Se–P(1)(α)	96.2(2)	95.9(2)
	Se–P(1)–C(7)(β)	101.1(2)	101.3(2)
	P(1)–C(7)–P(2)(γ)	120.0(3)	120.4(4)
	C(7)–P(2)–C(6)(δ)	103.7(3)	103.1(3)
	P(2)–C(6)–Se(ε)	118.9(3)	119.3(3)

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[1] The Gaussian 94 package was used for the quantum chemical calculations. Gaussian 94, Revision E.2, M.J. Frisch, G.W. Trucks, H.B. Schlegel, P.M.W. Gill, B.G. Johnson, M.A. Robb, J.R. Cheesman, T. Keith, G.A. Petersson, J.A. Montgomery, K. Raghavachari, M.A. Al-Laham, V.G. Zakrewski, J.V. Ortiz, J.B. Foresman, J. Cioslowski, B.B. Stefanov, A. Nanayakkara, M. Challacombe, C.Y. Peng, P. Ayala, W. Chen, M.W. Wong, J.L. Andres, E.S. Reoplogle, R. Gomperts, R.L. Martin, D.J. Fox, J.S. Binkley, D.J. Defrees, J. Baker, J.P. Stewart, M. Head-Gordon, C. Gonzalez, and J.A. Pople, Gaussian Inc. Pittsburg, Pennsylvania, USA, 1995. Geometry optimisation was carried out at the HF/3-21G(*) level, followed by the calculation of the second derivatives on the optimised structure. This calculation proved that a real minimum was obtained on the potential energy surface. Further optimisation was then performed at the B3LYP/3-21G(*) level. Improved relative energies were obtained at the B3LYP/6-311 + G**//B3LYP/3-21G(*) level.

Table 2
Comparison of calculated bond lengths (Å) and bond angles (°) for **1′** ($P_2SeC_2H_2$) and the hypothetical 2P- and 4P-ligated complexes $[Cr(CO)_5P_2SeC_2H_2]$ with the experimental data for **2**

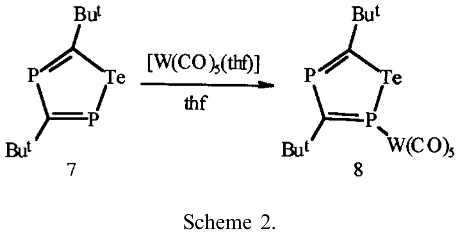
	1′	2	P(A)–Cr complex	P(B)–Cr–complex
a	2.218	2.182	2.195	2.222
b	1.709	1.695	1.692	1.708
c	1.764	1.774	1.764	1.743
d	1.704	1.681	1.706	1.692
e	1.867	1.873	1.874	1.863
P–Cr	–	2.359	2.308	2.324
α	96.0	96.2	93.9	96.8
β	97.1	101.1	100.6	97.5
γ	126.4	120.0	123.5	124.1
δ	98.4	103.7	99.1	102.1
ε	122.1	118.9	122.9	119.4

almost identical and the overall geometry of the planar 1,2,4-telluradiphosphole ring in **8** is very similar to that of the 1,2,4-selenadiphosphole ring in **2** and **3**, with the expected lengthening of the Te–C 2.067(4)Å and Te–P distances (2.378(2)Å compared with their Se analogues and a narrowing of the CTeP angle (90.9°) in **8** compared with the corresponding CSeP angle (96.2° in **2** and 95.9° in **3**)). In all three complexes **2**, **3** and **8** the sum of the angles at the P atom coordinated to the metal centre is as expected very close to 360°.

3. Experimental

All manipulations were carried out under dry nitrogen gas or in vacuo. Solvents were rigorously dried before use.

3.1. $[Cr(CO)_5\{\eta^1\text{-}P_2SeC_2Bu^t_2\}]$ **2**



Scheme 2.

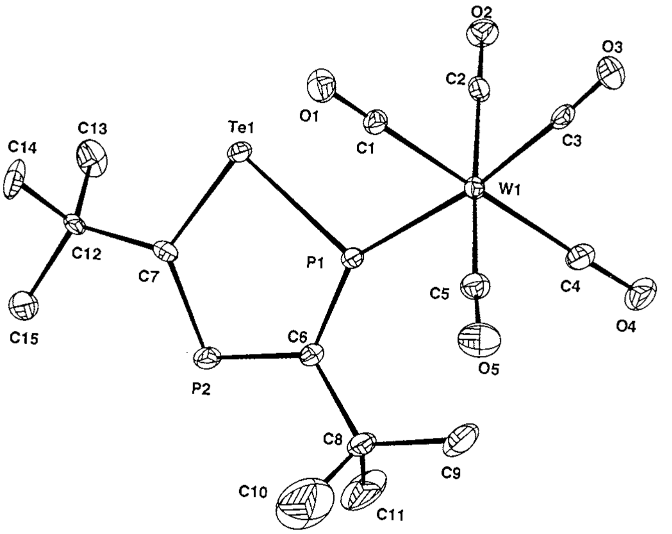


Fig. 3. Molecular structure of **8** with selected bond lengths (Å) and bond angles (°). Te(1)–C(7) 2.067(7); Te(1)–P(1) 2.378(2); W(1)–P(1) 2.506(2); P(1)–C(6) 1.702(7); P(2)–C(6) 1.785(7); P(2)–C(7) 1.695(7); C(7)–Te(1)–P(1) 90.9(2); C(6)–P(1)–Te(1) 101.7(3); Te(1)–P(1)–W(1) 111.69(7); C(6)–P(2)–C(7) 106.3(3); P(1)–C(6)–P(2) 122.3(4); P(2)–C(7)–Te(1) 118.8(4).

A solution of $[Cr(CO)_5(THF)]$ (0.38 mmol) was added to a solution of $P_2SeC_2Bu^t_2$ (113.4 mg, 0.40 mmol) in THF (15 ml). The resulting mixture was stirred for 24 h at room temperature and the solvent removed in vacuo. The residue was chromatographed (silica gel/p.e. 60–80°C) to give **2** as a yellow solid (70 mg, 36%) (m.p. = 144–145°C (dec.)). Recrystallisation from hexane afforded single crystals for the X-ray diffraction study (Found C, 37.6; H, 3.7; $C_{15}H_{18}CrO_5P_2Se$ requires C, 38.2; H, 3.8%).

NMR data: $^{31}P\{^1H\}$ δ 271.9 (d, P(B), $^2J_{P(B)P(A)}$ 62.3, $^2J_{P(B)Se}$ 54.6); δ 282.8 (d, P(A), $^1J_{P(A)Se}$ 480.2, $^2J_{P(A)P(B)}$ 62.3 Hz). ^1H: δ 1.26 (s, 9H, $C(CH_3)_3$); δ 1.52 (s, 9H, $C(CH_3)_3$).

Infrared data: ν_{CO} (cm^{-1}): 2071 (s), 1985 (w), 1960 (vs).

Mass spectrum (EI): m/z: 472 $[M]^+$ $[Cr(CO)_5\{P_2C_2Bu^t_2Se\}]^+$ (13%), 416 $[Cr(CO)_3\{P_2C_2Bu^t_2Se\}]^+$, 360 $[Cr(CO)\{P_2C_2Bu^t_2Se\}]^+$, 332 $[Cr\{P_2C_2Bu^t_2Se\}]^+$, 280 $[P_2C_2Bu^t_2Se]^+$, 57 $[Bu^t]^+$.

Crystal Data: **2** $C_{15}H_{18}CrO_5P_2Se$, M = 471.2, monoclinic, space group $P2_1/n$ (non standard no. 14), $a = 6.249(2)$, $b = 29.965(10)$, $c = 10.670(3)$ Å, $\beta = 92.36(2)°$, $U = 1996.3(11)$ Å^3, $Z = 4$, $D_{calc} = 1.57$ g cm^{-3}, $F(000) = 944$. Monochomated Mo–K$_\alpha$ radiation $\lambda = 0.71073$ Å, T = 173(2) K. Data were collected on an Enraf-Nonius CAD 4 diffractometer using a crystal of 0.20 × 0.15 × 0.15 mm. A total of 3515 unique reflections were measured for $2 < \theta < 25°$ of which 2491 had $I > 2\sigma(I)$. The structure was solved by direct methods using SHELXS-86 and refined on F^2 with all non-H atoms anisotropic [8,9]. H atoms were included in riding mode with $U_{iso} = 1.5$ $U_{eq}(C)$. The C(8) Bu^t group was disordered over two equally populated orientations. Final residuals were $R_1 = 0.053$ for $I > 2\sigma(I)$ and $wR_2 = 0.150$ (for all data).

3.2. *[W(CO)$_5${η^1-P$_2$C$_2$SeBu$^t_2$}]* **3**

A solution of [W(CO)$_5$(THF)] (0.12 mmol) was added to a solution of $P_2C_2Bu^t_2Se$ (100 mg, 0.35 mmol) and the resulting mixture stirred for 48 h at room temperature. Removal of the solvent produced an oily-orange residue which was purified by chromatography (silica gel/hexane) to give **3** as a yellow solid (60 mg, 27%) (m.p. = 140–142°C). Recrystallisation from hexane afforded single crystals for the X-ray diffraction study (Found C, 29.9; H, 3.0; $C_{15}H_{18}WO_5P_2Se$ requires C, 29.8; H, 3.0%).

NMR data: $^{31}P\{^1H\}$ δ 221.8 (d, P(A), $^1J_{P(A)Se}$ 473.2, $^1J_{P(A)W}$ 241.1, $^2J_{P(A)P(B)}$ 64.0); δ 266.4 (d, P(B), $^2J_{P(B)P(A)}$ 64.0, $^2J_{P(B)Se}$ 55.2 Hz). $^{77}Se\{^1H\}$ δ 935.1 (rel Me$_2$Se) (dd, $^1J_{SeP(A)}$ 472.4, $^2J_{SeP(B)}$ 54.9 Hz). ^{1}H (300.1 MHz, benzene-d$_6$, 25°C): δ 1.02 ppm (d, 9H, C(CH_3)$_3$, $^4J_{HP}$ 1.4); δ 1.28 (t, 9H, C(CH_3)$_3$, $^4J_{HP(A)} = {}^4J_{HP(B)}$ 1.1 Hz).

Infrared data: ν_{CO} (cm^{-1}): 2078 (w), 1981 (s), 1957 (vs).

Mass spectrum (EI): m/z: 604 [M]$^+$ [W(CO)$_5${P$_2$C$_2$Bu$^t_2$Se}]$^+$ (59%), 548 [W(CO)$_3${P$_2$C$_2$Bu$^t_2$Se}]$^+$, 492 [W(CO){P$_2$C$_2$Bu$^t_2$Se}]$^+$, 464 [W{P$_2$C$_2$Bu$^t_2$Se}]$^+$, 280 [P$_2$C$_2$Bu$^t_2$Se]$^+$, 57 [Bu^t]$^+$.

Crystal Data **3**: $C_{15}H_{18}WO_5P_2Se$, M = 603.0, monoclinic, space group $P2_1/n$ (non standard no. 14), $a = 6.375(2)$, $b = 30.204(6)$, $c = 10.685(4)$ Å, $\beta = 93.07(3)°$, $U = 2054.4(11)$ Å^3, $Z = 4$, $D_{calc.} = 1.95$ g cm^{-3}, $F(000) = 1144$. Monochomated Mo–K$_\alpha$ radiation $\lambda = 0.71073$ Å, T = 173(2) K. Data were collected on an Enraf-Nonius CAD 4 diffractometer using a crystal of 0.15 × 0.15 × 0.10 mm. A total of 3625 unique reflections were measured for $2 < \theta < 25°$ of which 2996 had $I > 2\sigma(I)$. The structure was solved by direct methods using SHELXS-86 and refined on F^2 with all non-H atoms anisotropic. H atoms were included in riding mode with $U_{iso} = 1.5\ U_{eq}(C)$. The C(12) Bu^t group was disordered 52:48% over two orientations. Final residuals were $R_1 = 0.032$ [for $I > 2\sigma(I)$] and $wR_2 = 0.073$ (for all data).

3.3. *[W(CO)$_5${η^1-P$_2$C$_2$TeBu$^t_2$}]* **8**

[W(CO)$_5${η^1-P$_2$C$_2$TeBu$^t_2$}] **8** was made in a similar fashion to the above, (40%), m.p.125–130°. Found C, 26.5; H, 1.8; $C_{15}H_{18}WO_5P_2Te$ requires C, 27.7; H, 2.8%.

NMR data: $^{31}P\{^1H\}$ δ 216.0 (d, P(A), $^1J_{P(A)Te}$ 1174, $^1J_{P(A)W}$ 231, $^2J_{P(A)P(B)}$ 66.1); δ 298.4 (d, P(B), $^2J_{P(B)P(A)}$ 66.1, $^2J_{P(B)Te}$ 132 Hz). $^{125}Te\{^1H\}$: δ 1373 (rel Me$_2$Te) (dd, $^1J_{TeP(A)}$ 1175, $^2J_{TeP(B)}$ 133 Hz). ^{1}H: δ 1.64 ppm (d, 9H, C(CH_3)$_3$, $^4J_{HP}$ 0.9); δ 1.30 (d, 9H, C(CH_3)$_3$, $^4J_{HP}$ 1.69 Hz). NMR data for comparison for **7** [3]: $^{31}P\{^1H\}$ δ 302 (d, P(A), $^1J_{P(A)Te}$ 1028, $^2J_{P(A)P(B)}$ 50.8); δ 299 (d, P(B), $^2J_{P(B)P(A)}$ 50.8, $^2J_{P(B)Te}$ 151 Hz). $^{125}Te\{^1H\}$: δ 1383 (rel Me$_2$Te) (dd, $^1J_{TeP(A)}$ 1035, $^2J_{TeP(B)}$ 133 Hz). ^{1}H: δ 1.66 ppm (d, 9H, C(CH_3)$_3$, $^4J_{HP}$ 2.4); δ 1.48 (d, 9H, C(CH_3)$_3$, $^4J_{HP}$ 0.61 Hz).

Infrared data for **8**: ν_{CO} (cm^{-1}): 2074.9 (w), 1954.5 (vs), br with shoulder.

Mass spectrum for **8** (EI/CI): m/z: 596 [W(CO)$_3${P$_2$C$_2$Bu$^t_2$Te}]$^+$, 540 [W(CO){P$_2$C$_2$Bu$^t_2$Se}]$^+$, 512 [W{P$_2$C$_2$Bu$^t_2$Se}]$^+$.

Crystal Data: **8** $C_{15}H_{18}WO_5P_2Te$, M = 651.7, monoclinic, space group $P2_{1/n}$, $a = 6.4530(10)$, $b = 29.924(6)$, $c = 10.867(2)$ Å, $\beta = 93.82(3)°$, $U = 2093.8(7)$ Å^3, $Z = 4$, $D_{calc.} = 2.067$ g cm^{-3}, $F(000) = 1216$. Monochomated Mo–K$_\alpha$ radiation $\lambda = 0.71069$ Å, T = 150(2) K. Data were collected using a FAST area detector diffractometer following previously described procedures, [10] using a crystal of 0.15 × 0.20 × 0.20 mm. A total of 3095 independent reflections were measured for $2 < \theta < 25°$. The structure was solved by heavy atom methods and refined on F^2 with all non-H atoms anisotropic. Empirical absorption corrections were carried out by the DIFABS method [11]. The C(8) Bu^t group was disordered over two sites and Fig. 3 shows the most occupied site. Final residuals were $R_1 = 0.0355$ [for $I > 2\sigma(I)$] and $wR_2 = 0.0811$ (for all data).

Acknowledgements

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