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Syntheses and structures of one diphosphane monochalcogenide, one diphosphane dioxide and three diphosphane hetero-dichalcogenides

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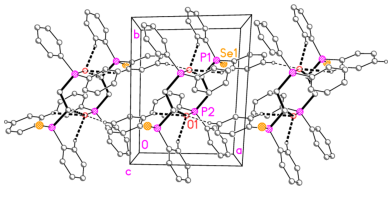
Compound **1**, $C_{13}H_{30}P_2S$ or $SP^iPr_2CH_2P^iPr_2$, bis(diisopropylphosphano)-methane monosulfide, crystallizes in the space group $P2_1/n$ with Z = 4; it is isotypic to the corresponding selenium and tellurium derivatives. Compound **2**, $C_{25}H_{22}OP_2Se$ or $SePPh_2CH_2PPh_2O$, bis(diphenylphosphano)methane monoxide monoselenide, crystallizes in $P2_12_12_1$ with Z = 4; it is isotypic to the analogous O,S derivative. Compound **3**, bis(diphenylphosphano)methane dioxide dichloromethane quarter-solvate, $C_{24}H_{22}O_2P_2{\cdot}0.25CH_2Cl_2$ or $OPPh_2CH_2PPh_2O{\cdot}0.25CH_2Cl_2$, crystallizes in $P\overline{1}$ with Z = 4; it is isotypic to the corresponding benzene and toluene quarter-solvates. The two independent molecules are very similar; each displays a pair of parallel, intramolecularly stacked, phenyl rings (intercentroid distances 3.78 and 3.62 Å, interplanar angles *ca* 10°). An analysis of bis(diphenylphosphano)methane dichalcogenides shows three classes of structure according to the central E—P···P—E torsion angles (E = chalcogenide), and also the ring orientations. Compounds **4**, $C_{26}H_{24}OP_2S$ or dppeSO, 1,2-bis(diphenylphosphano)ethane monoxide monosulfide, and **5**, $C_{26}H_{24}OP_2S$ or dppeSeO, 1,2-bis(diphenylphosphano)ethane monoxide monoselenide, crystallize with Z = 2 in the same space group $P\overline{1}$, but are not isotypic. They both show the typical *trans* disposition of the P atoms around the central C—C bond. For the oxo derivatives **2**–**5**, the packing is characterized by hydrogen bonding from three C—H moieties to the O atom; the hydrogen bonds involving methylene H atoms are very short (H···O = 2.17–2.39 Å).

1. Chemical context

We are interested in metal complexes of phosphane and diphosphane chalcogenide ligands of various types. Our first such publication reported the structures of $[(Ph_3PSe)_2Au]^+SbF_6^-$ and $[(Ph_3P)Au(SePPh_2Me)]^+SbF_6^-$ (Jones & Thöne, 1991). With hindsight regrettably, only in 2013 did we begin to number (most of) the new publications as a series; Part 1 was Taouss & Jones (2013). Much introductory material is given in Part 6 (Upmann *et al.*, 2024). Here we present the crystal structures of some of the ligands, namely, one bis(dialkylphosphano)methane monochalcogenide, $SP^iPr_2CH_2P^iPr_2$, **1**, one bis(diphenylphosphano)methane hetero-dichalcogenide, $SePPh_2CH_2PPh_2O$, **2**, bis(diphenylphosphano)methane dioxide as its dichloromethane quarter-solvate, $OPPh_2CH_2PPh_2O{\cdot}0.25CH_2Cl_2$, **3**, and two 1,2-bis(diphenylphosphano)-ethane hetero-dichalcogenides, $SPPh_2CH_2CH_2PPh_2O$, **4**, and $SePPh_2CH_2CH_2PPh_2O$, **5**. Both ligand types have been used much less often than the well-known diphosphanes (bearing no chalcogenides) or diphosphane homo-dichalcogenides.



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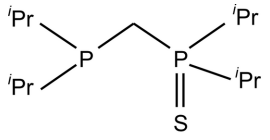
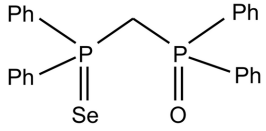
2. Structural commentary

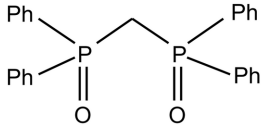
All compounds except **3** crystallized solvent-free and with $Z' = 1$. Selected molecular dimensions are given in Tables 1–5. In the text, generalized chalcogenides are represented by the symbol *E*. The abbreviations 'dppm' and 'dppe' are used for the diphosphanes bis(diphenylphosphano)methane and 1,2-bis(diphenylphosphano)ethane, respectively.



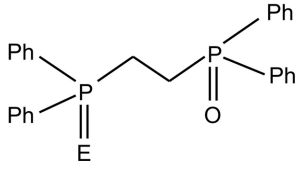
(**1**)



(**2**)



• ¼ (CH_2Cl_2) (**3**)



(**4**, E = S; **5**, E = Se)

Bond lengths and angles may be regarded as normal, *e.g.* the formal double-bond lengths P═O = 1.4830 (13)–1.4925 (16) Å (7 values), P═S = 1.9531 (4) and 1.9631 (5) Å, and P═Se = 2.1063 (6) and 2.1148 (5) Å. The largest bond angles at phosphorus are those involving the chalcogen atoms.

Compound **1**, $SP^iPr_2CH_2P^iPr_2$ (Fig. 1), is a further member of the series $EP^iPr_2CH_2P^iPr_2$, for which the structures of the selenium and tellurium analogues have been determined by Elder *et al.* (2013), with Cambridge Structural Database (CSD; Groom *et al.*, 2016) refcodes REXOUH and REXPOB, respectively. The cell parameters for the three compounds imply at first sight that the sulfur and tellurium derivatives are isotypic (confirmed by inspection of the atom coordinates), but the selenium derivative, surprisingly, is not. However, the reported cell for the latter is not reduced; the reduced form is very similar to those of the other analogues. Without deposited structure factors, this cannot be proved unambiguously, but it seems likely that all three compounds are indeed isotypic. Details of the cells are given in Table 6. The P—C—P angle in **1** is 111.94 (7)° and the S—P—C—P torsion angle is 56.97 (8)°. The largest angles at four-coordinate atom P1 are those involving the S atom.

Table 1
Selected geometric parameters (Å, °) for **1**.

S1—P1	1.9631 (5)	P2—C3	1.8609 (14)
P1—C10	1.8177 (13)	P2—C10	1.8658 (13)
P1—C1	1.8349 (13)	P2—C4	1.8662 (14)
P1—C2	1.8409 (14)		
C10—P1—C1	104.53 (6)	C2—P1—S1	113.78 (5)
C10—P1—C2	103.45 (6)	C3—P2—C10	100.56 (6)
C1—P1—C2	108.43 (6)	C3—P2—C4	104.72 (6)
C10—P1—S1	112.03 (5)	C10—P2—C4	99.17 (6)
C1—P1—S1	113.73 (5)	P1—C10—P2	111.94 (7)
C1—P1—C10—P2	−179.43 (7)	C4—P2—C10—P1	−110.46 (8)
S1—P1—C10—P2	56.97 (8)	C10—P1—C1—C11	176.18 (10)
C3—P2—C10—P1	142.57 (8)		

Table 2
Selected geometric parameters (Å, °) for **2**.

P1—C21	1.819 (2)	P2—O1	1.4920 (16)
P1—C11	1.820 (2)	P2—C41	1.806 (2)
P1—C1	1.826 (2)	P2—C31	1.816 (2)
P1—Se1	2.1063 (6)	P2—C1	1.819 (2)
C21—P1—C11	105.34 (10)	O1—P2—C31	111.86 (10)
C21—P1—C1	103.37 (10)	C41—P2—C31	108.43 (10)
C11—P1—C1	106.04 (10)	O1—P2—C1	113.85 (10)
C21—P1—Se1	113.48 (8)	C41—P2—C1	107.91 (10)
C11—P1—Se1	113.39 (8)	C31—P2—C1	101.96 (10)
C1—P1—Se1	114.26 (7)	P2—C1—P1	116.49 (11)
O1—P2—C41	112.21 (10)		
O1—P2—C1—P1	45.98 (15)	C21—P1—C11—C16	−130.2 (2)
C41—P2—C1—P1	−79.28 (14)	C1—P1—C11—C16	120.64 (19)
C31—P2—C1—P1	166.64 (12)	Se1—P1—C11—C16	−5.5 (2)
C21—P1—C1—P2	171.27 (12)	C21—P1—C11—C12	47.6 (2)
C11—P1—C1—P2	−78.17 (14)	C1—P1—C11—C12	−61.5 (2)
Se1—P1—C1—P2	47.47 (14)	Se1—P1—C11—C12	172.28 (18)

Table 3
Selected geometric parameters (Å, °) for **3**.

P1—O1	1.4838 (13)	P1′—O1′	1.4830 (13)
P1—C11	1.8047 (18)	P1′—C21′	1.8029 (18)
P1—C21	1.8070 (18)	P1′—C11′	1.8099 (17)
P1—C1	1.8097 (17)	P1′—C1′	1.8146 (17)
P2—O2	1.4879 (13)	P2′—O2′	1.4869 (13)
P2—C41	1.7966 (17)	P2′—C31′	1.7994 (19)
P2—C31	1.7986 (18)	P2′—C41′	1.8003 (18)
P2—C1	1.8165 (16)	P2′—C1′	1.8219 (17)
O1—P1—C11	112.44 (8)	O1′—P1′—C21′	113.03 (8)
O1—P1—C21	112.49 (8)	O1′—P1′—C11′	112.42 (8)
C11—P1—C21	106.50 (8)	C21′—P1′—C11′	105.02 (8)
O1—P1—C1	114.06 (8)	O1′—P1′—C1′	114.55 (8)
C11—P1—C1	104.25 (8)	C21′—P1′—C1′	106.75 (8)
C21—P1—C1	106.42 (8)	C11′—P1′—C1′	104.24 (8)
O2—P2—C41	112.05 (7)	O2′—P2′—C31′	111.16 (9)
O2—P2—C31	111.39 (8)	O2′—P2′—C41′	111.42 (8)
C41—P2—C31	107.19 (8)	C31′—P2′—C41′	107.84 (8)
O2—P2—C1	110.56 (8)	O2′—P2′—C1′	110.18 (8)
C41—P2—C1	106.38 (8)	C31′—P2′—C1′	108.60 (8)
C31—P2—C1	109.07 (8)	C41′—P2′—C1′	107.51 (8)
P1—C1—P2	119.07 (9)	P1′—C1′—P2′	121.07 (9)
O1—P1—C1—P2	−53.22 (13)	O1′—P1′—C1′—P2′	−58.61 (13)
C11—P1—C1—P2	−176.23 (10)	C21′—P1′—C1′—P2′	67.30 (12)
C21—P1—C1—P2	71.41 (12)	C11′—P1′—C1′—P2′	178.12 (10)
O2—P2—C1—P1	−145.14 (10)	O2′—P2′—C1′—P1′	−150.02 (10)
C41—P2—C1—P1	92.98 (11)	C31′—P2′—C1′—P1′	−28.05 (13)
C31—P2—C1—P1	−22.34 (13)	C41′—P2′—C1′—P1′	88.38 (12)

Table 4
Selected geometric parameters (Å, °) for **4**.

P1—C11	1.8121 (11)	P2—C41	1.8042 (11)
P1—C21	1.8151 (10)	P2—C31	1.8060 (11)
P1—C1	1.8224 (10)	P2—C2	1.8112 (10)
P1—S1	1.9531 (4)	C1—C2	1.5326 (14)
P2—O1	1.4925 (8)		
C11—P1—C21	107.88 (5)	O1—P2—C31	113.36 (5)
C11—P1—C1	104.20 (5)	C41—P2—C31	106.91 (5)
C21—P1—C1	107.06 (5)	O1—P2—C2	112.56 (5)
C11—P1—S1	113.20 (4)	C41—P2—C2	105.73 (5)
C21—P1—S1	111.65 (4)	C31—P2—C2	105.79 (5)
C1—P1—S1	112.37 (4)	C2—C1—P1	111.36 (7)
O1—P2—C41	111.94 (5)	C1—C2—P2	111.71 (7)
C11—P1—C1—C2	175.65 (7)	C31—P2—C2—C1	179.61 (7)
C21—P1—C1—C2	−70.22 (8)	C1—P1—C11—C12	58.93 (9)
S1—P1—C1—C2	52.72 (8)	C1—P1—C21—C22	−27.17 (10)
P1—C1—C2—P2	−173.52 (5)	C2—P2—C31—C32	179.29 (9)
O1—P2—C2—C1	−56.08 (9)	C2—P2—C41—C42	90.01 (10)
C41—P2—C2—C1	66.42 (8)		

Table 5
Selected geometric parameters (Å, °) for **5**.

P1—C11	1.8173 (18)	P2—C31	1.8023 (18)
P1—C21	1.8182 (17)	P2—C2	1.8026 (18)
P1—C1	1.8252 (18)	P2—C41	1.8084 (18)
P1—Se1	2.1148 (5)	C1—C2	1.529 (2)
P2—O1	1.4908 (14)		
C11—P1—C21	107.27 (8)	O1—P2—C2	114.98 (8)
C11—P1—C1	105.53 (8)	C31—P2—C2	102.48 (8)
C21—P1—C1	105.82 (8)	O1—P2—C41	111.96 (8)
C11—P1—Se1	113.50 (6)	C31—P2—C41	107.42 (8)
C21—P1—Se1	113.37 (6)	C2—P2—C41	107.36 (8)
C1—P1—Se1	110.77 (6)	C2—C1—P1	109.91 (12)
O1—P2—C31	111.97 (8)	C1—C2—P2	115.36 (12)
C11—P1—C1—C2	177.01 (12)	C41—P2—C2—C1	60.19 (15)
C21—P1—C1—C2	−69.48 (13)	C1—P1—C11—C12	48.62 (16)
Se1—P1—C1—C2	53.78 (13)	C1—P1—C21—C22	−23.50 (17)
P1—C1—C2—P2	−165.25 (9)	C2—P2—C31—C32	−61.28 (17)
O1—P2—C2—C1	−65.11 (15)	C2—P2—C41—C42	20.29 (18)
C31—P2—C2—C1	173.17 (13)		

Compound **2**, dppmSeO (Fig. 2), is isotypic to dppmSO (refcode VETCAA; Heuclin *et al.*, 2012). The P—C—P angle is 116.49 (11)°, the Se—P···P—O torsion angle is 83.35 (7)° and the intramolecular Se···O contact is 3.9944 (16) Å, too long to be considered as a significant secondary contact.

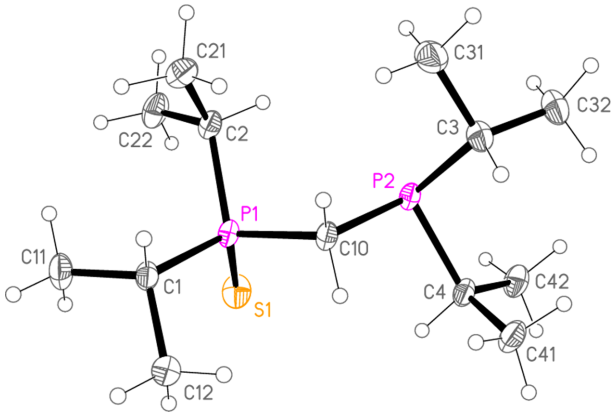


Figure 1
The molecule of compound **1** in the crystal. Ellipsoids correspond to 50% probability levels.

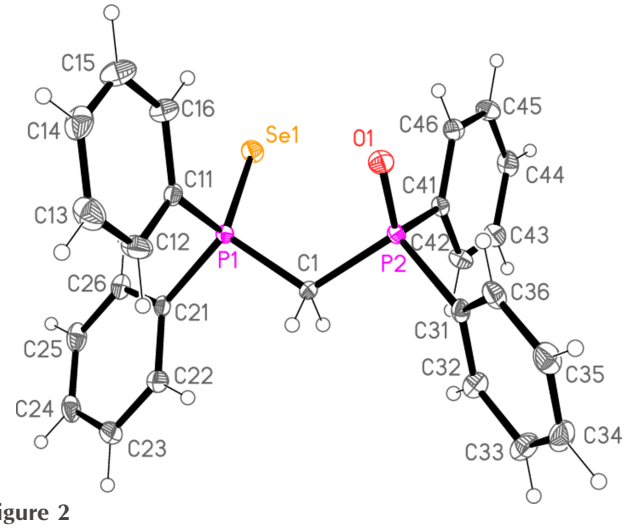


Figure 2
The molecule of compound **2** in the crystal. Ellipsoids correspond to 50% probability levels.

Compound **3**, $dppmO_2{\cdot}0.25(CH_2Cl_2)$ (Fig. 3), crystallizes with two independent molecules of the dioxide and half a molecule of dichloromethane (disordered over an inversion centre) in the asymmetric unit; this structure is isotypic to the corresponding benzene and toluene solvates (see below). The

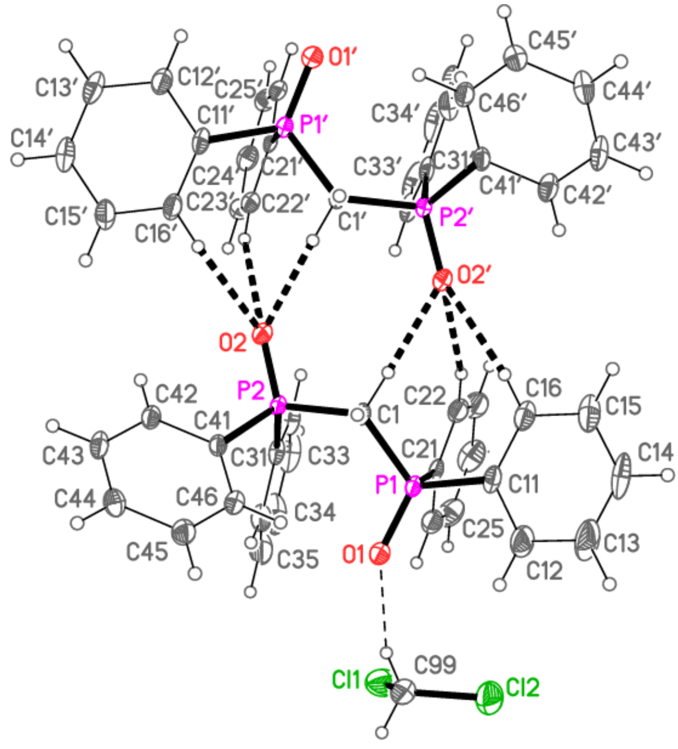


Figure 3
The asymmetric unit of compound **3** in the crystal. Dashed lines indicate hydrogen bonds (see *Supramolecular features*). Only one position of the disordered solvent molecule is shown. Ellipsoids correspond to 50% probability levels.

Table 6
Cell constants (Å and °) of compounds $E{=}P^{i}Pr_2CH_2P^{i}Pr_2$ (E = chalcogen).

E	Space group	a	b	c	β	Temperature (° C)	Reference
S	$P2_1/n$	10.7521 (5)	11.5873 (5)	14.0337 (8)	110.005 (6)	−173	This work
Se	$P2_1/c$	10.7760 (3)	11.7200 (3)	14.2610 (5)	114.7410 (11)	−100	Elder *et al.* (2013)
Se	$P2_1/n$	10.776	11.720	14.072	109.33	−100	See Note *a*
Te	$P2_1/n$	10.7440 (4)	11.9840 (2)	14.0990 (5)	107.8230 (11)	−150	Elder *et al.* (2013)

Note: (*a*) transformed from the published cell using the matrix (100/0$\bar{1}$0/$\bar{1}$0$\bar{1}$)

two independent molecules can be fitted *via* the P, O, C1 and *ipso* C atoms to an r.m.s.d. of 0.12 Å (Fig. 4); the ring orentations are very similar. The P—C—P angles are 119.07 (9) and 121.07 (9)°, and the torsion angles across the P···P vectors, Se1—P1···P2—O1 and Se1′—P1′···P2′—O1′, are −179.35 (9) and 169.49 (10)°, respectively. Similarly to **1**, the largest angles at the P atoms of **2** and **3** are those involving the chalcogen atoms. The ring pairs C21–26 and C31–36 in both molecules display intramolecular stacking, with intercentroid distances of 3.78 and 3.62 Å, and interplanar angles of 11.7 (1) and 9.1 (1)°.

We also managed to obtain single crystals of the diphosphane monoxide dppmO, which crystallizes in the space group $Pca2_1$ with a = 9.6579 (2), b = 39.5181 (5), c = 10.7374 (2) Å and Z = 8. However, the O atoms proved to be disordered over the two P atoms in both molecules, and the structure could not be satisfactorily refined. The parent diphosphane dppm crystallizes in the orthorhombic space group *Pbca* with a = 9.795, b = 10.198 and c = 40.417 Å at 153 K (293 K, Burrow *et al.*, 2007; 233 K, Schmidbaur *et al.*, 1988; 153 K, Di Nicola *et al.*, 2005) and Z = 8. The cell constants of both compounds are similar, and at first we thought that the sample was simply dppm, reflecting perhaps incomplete oxidation of dppm and its subsequent preferential crystallization. It is possible that other workers had been similarly misled by this 'camouflaged' cell, which would have explained why the structure of dppmO had never been reported. However, the structure is in any case unsatisfactory because of the disorder.

Table 7 presents significant bond and torsion angles for structures of dppm dichalcogenides. Clearly there is a considerable degree of conformational flexibility, but the central E—P···P—E torsion angles, which determine the basic form of the molecule, fall into three classes, with absolute values of *ca* 90° (for **2**, dppmSO, $dppmO_2{\cdot}CHCl_3$ and the $P2_1/n$ forms of $dppmS_2$ and $dppmSe_2$), *ca* 180° for **3** and the other $dppmO_2$ solvates, and *ca* 150° for the $C2/c$ forms of $dppmS_2$ and $dppmSe_2$. The pairs of torsion angles

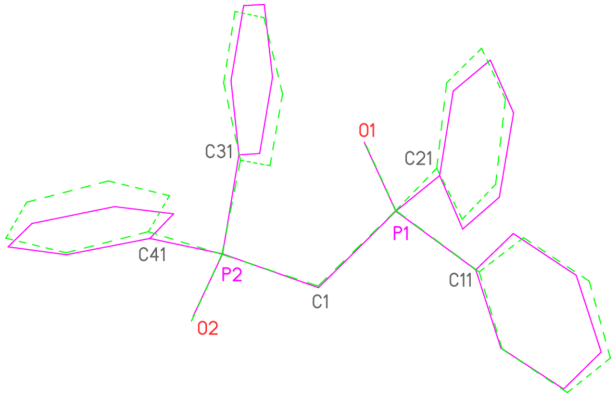


Figure 4
A least-squares fit of both independent molecules of **3**. Fitted atoms are labelled. Molecule 1 is drawn with full bonds in purple and molecule 2 with dotted bonds in green.

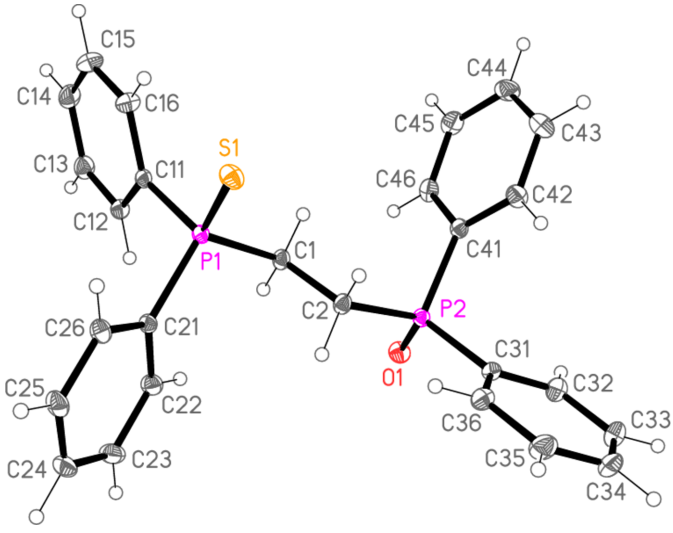


Figure 5
The molecule of compound **4** in the crystal. Ellipsoids correspond to 50% probability levels.

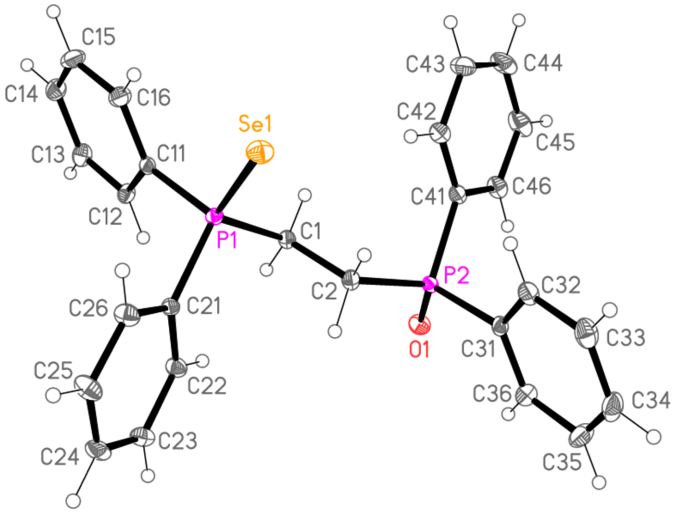


Figure 6
The molecule of compound **5** in the crystal. Ellipsoids correspond to 50% probability levels.

Table 7
Bond angles and torsion angles (°) of dppm dichalcogenides $E^1PPh_2CH_2PPh_2E^2$.

Values not given in the original publications have been calculated from deposited coordinates or by new refinements using deposited structure factors

Compound	Reference	P—C—P	E^1—P—C—P	P—C—P—E^2	E^1—P···P—E^2	P1—C1—P2—C(Ph)	P2—C1—P1—C(Ph)
dppmSO[a,b]	Heuclin *et al.* (2012)	115.72 (13)	46.3	45.8	82.2	166.5,. 79.5	170.6, −79.3
dppmSeO (**2**)[a,b]	This work	116.49 (11)	47.47 (14)	45.98 (15)	83.35 (7)	166.64 (12), −79.28 (14)	171.27 (12), −78.17 (14)
$dppmO_2{\cdot}CHCl_3$[f]	Grachova *et al.* (2016)	116.04 (10)	56.86 (13)	51.93 (13)	96.12 (8)	175.97 (10), −73.55 (12)	176.48 (10), −68.87 (14)
$dppmO_2{\cdot}0.25CH_2Cl_2$, (**3**), Mol. 1	This work	119.07 (9)	−53.22 (13)	−145.14 (10)	−179.35 (9)	−22.34 (13), 92.98 (11)	−176.23 (10), 71.41 (12)
$dppmO_2{\cdot}0.25CH_2Cl_2$, (**3**), Mol. 2	This work	121.07 (9)	−58.61 (13)	−150.02 (10)	169.49 (10)	−28.05 (13), 88.38 (12)	178.12 (10), 67.30 (12)
$dppmS_2$ ($P2_1/n$)[c]	Carmalt *et al.* (1996)	117.9 (6)	−61.3	−47.3	95.64 (5)	−168.9, 79.8	175.5, 65.3
$dppmS_2$ ($C2/c$)[d,e]	Thirumoorthi & Chivers (2012)	120.8 (2)	−85.8	−148.8		151.0, 40.3	
$dppmSe_2$ ($P2_1/n$)[c]	Carroll & Titus (1977)	117.9 (6)	60.0	48.7	95.5	170.4, −79,7	−178.0, 66.9
$dppmSe_2$ ($C2/c$)[d,e]	Jones & Ahrens (1998)	121.1 (3)	85.05 (8)	148.12 (7)		−151.98 (15), −40.84 (15)	

Notes: (*a*) E^1 = Se or S; E^2 = O. (*b*)/(*c*)/(*d*) Isotypic pairs. (*e*) The molecule displays crystallographic twofold symmetry. (*f*) The structure was re-refined from deposited coordinates and structure factors.

P—C—P—C_{phenyl}, which describe the ring orientations, also display similar values for each class. It is noteworthy that the P—C—P angles are significantly greater for the latter two classes.

Compounds **4**, dppeSO (Fig. 5), and **5**, dppeSeO (Fig. 6), crystallize in the same space group $P\overline{1}$, but are not isotypic (see below). To the best of our knowledge, these are the first 1,2-bis(diphenylphosphano)ethane hetero-dichalcogenides to be structurally characterized. They show the typical *trans* disposition of the P atoms around the central C—C bond of 1,2-bis(diphosphano)ethane derivatives, which leads to the perhaps counter-intuitive observation that the dppe derivatives are less conformationally flexible than the dppm derivatives, despite the longer bridge. A least-squares fit of the two molecules (Fig. 7) shows that they are very similar except for the orientations of the two rings at C31 and C41 (*cf* torsion angles in Table 4 and 5).

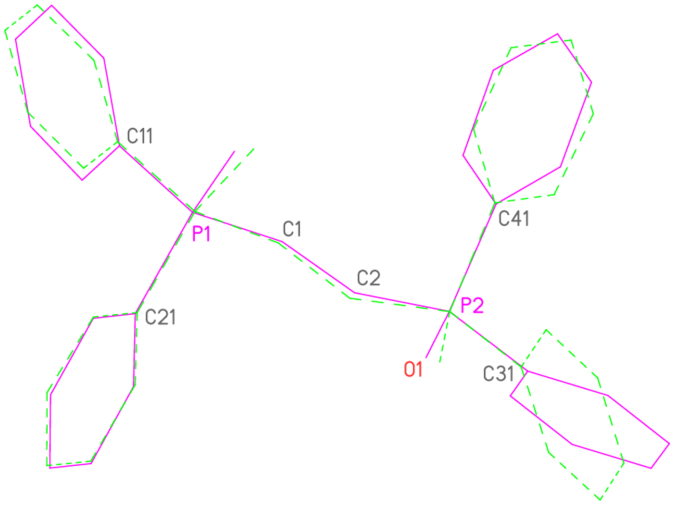


Figure 7
A least-squares fit of compound **5** (dotted lines, green) to compound **4** (full lines, purple). Fitted atoms are labelled; the r.m.s. deviation is 0.13 Å.

3. Supramolecular features

Hydrogen bonds for **1**–**5** are presented in Tables 8–12, respectively (several borderline contacts are included). The packing of compound **1** involves only one moderately short contact, from a methyl H atom to the S atom, which may correspond to a 'weak' hydrogen bond (Table 8). This connects molecules to form linear arrays propagating parallel to [101] (Fig. 8). Ignoring this interaction, an alternative description is that the molecules lie in layers parallel to (101) in a distorted hexagonal packing (Fig. 9).

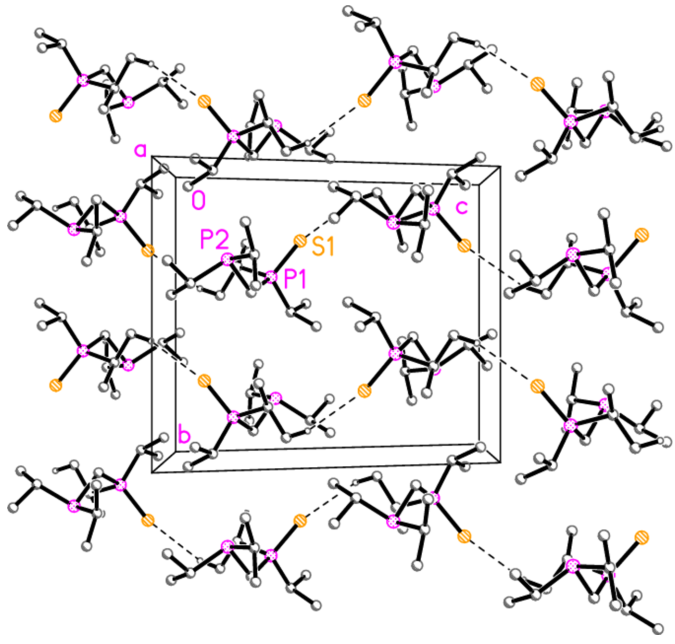


Figure 8
Packing diagram of compound **1** viewed parallel to the *a* axis, showing chains of molecules linked by short $H_{methyl}\cdots S$ contacts (dashed lines) that might qualify as 'weak' hydrogen bonds. Other H atoms are omitted. In all packing diagrams, labelled atoms correspond to the asymmetric unit and H atoms not involved in hydrogen bonding are omitted for clarity.

Table 8
Hydrogen-bond geometry (Å, °) for **1**.

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21A···S1^{i}	0.98	2.89	3.8390 (16)	162
C22—H22B···S1	0.98	2.91	3.4526 (17)	116

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Table 9
Hydrogen-bond geometry (Å, °) for **2**.

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···O1^{i}	0.99	2.37	3.342 (3)	168
C32—H32···O1^{i}	0.95	2.48	3.404 (3)	165
C42—H42···O1^{i}	0.95	2.40	3.261 (3)	151
C16—H16···Se1	0.95	2.86	3.416 (3)	118

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Table 10
Hydrogen-bond geometry (Å, °) for **3**.

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O1^{i}	0.95	2.56	3.296 (2)	134
C99—H99A···O1	0.99	2.43	3.384 (5)	162
C1′—H1A′···O2	0.99	2.22	3.211 (2)	176
C16′—H16′···O2	0.95	2.38	3.324 (2)	172
C22′—H22′···O2	0.95	2.31	3.236 (2)	163
C1—H1A···O2′	0.99	2.17	3.156 (2)	176
C16—H16···O2′	0.95	2.44	3.390 (2)	173
C22—H22···O2′	0.95	2.38	3.311 (2)	167

Symmetry code: (i) $-x+1, -y+1, -z$.

To understand the packing of compounds **2** and **3**, it is helpful to know that the methylene H atoms of dppm and its derivatives are quite acidic. This means that deprotonated forms of dppm and related ligands are easily generated and can function as multidentate ligands (see, for example, Heuclin *et al.*, 2012; Thirumoorthi & Chivers, 2012; Usón *et al.*, 1984). Furthermore, these H atoms are also excellent hydrogen-bond donors, as we pointed out earlier (Jones & Ahrens, 1998). Thus, in the packing of **2** (Fig. 10), the O atom accepts hydrogen bonds from three H atoms, a methylene H atom and

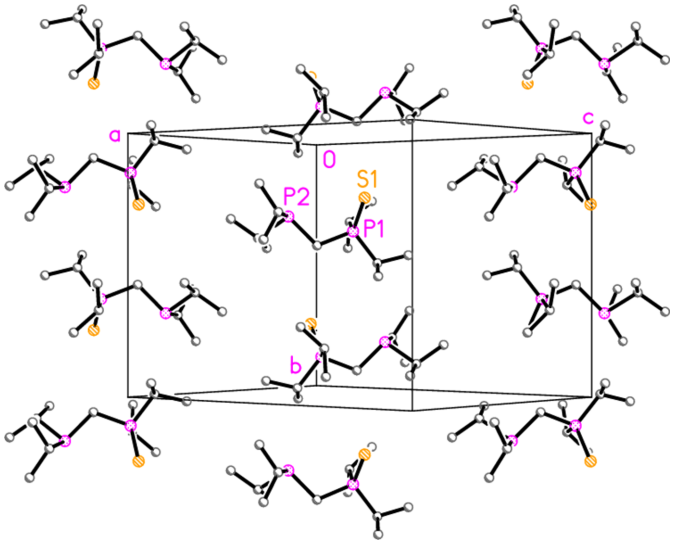


Figure 9
Alternative view of the packing of compound **1**, showing a layer of molecules parallel to (101). This layer passes through the region $x \simeq 0.25$, $z \simeq 0.25$; a further layer passes through the region $x \simeq 0.75$, $z \simeq 0.75$.

Table 11
Hydrogen-bond geometry (Å, °) for **4**.

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O1^{i}	0.99	2.38	3.3622 (13)	170
C12—H12···O1^{i}	0.95	2.44	3.2973 (13)	150
C22—H22···O1^{i}	0.95	2.39	3.2909 (13)	158
C24—H24···O1^{ii}	0.95	2.65	3.5750 (14)	165
C42—H42···S1^{iii}	0.95	2.92	3.5826 (11)	128
C44—H44···S1^{iv}	0.95	2.91	3.7405 (12)	146
C2—H2B···S1	0.99	2.93	3.4548 (11)	114
C16—H16···S1	0.95	2.80	3.3190 (12)	115
C26—H26···S1	0.95	2.85	3.3188 (11)	112

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y+2, -z+1$; (iv) $-x, -y+2, -z+1$.

Table 12
Hydrogen-bond geometry (Å, °) for **5**.

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O1^{i}	0.99	2.29	3.269 (2)	171
C12—H12···O1^{i}	0.95	2.46	3.368 (2)	159
C22—H22···O1^{i}	0.95	2.62	3.503 (2)	155
C24—H24···O1^{ii}	0.95	2.67	3.612 (2)	169
C2—H2B···Se1	0.99	2.85	3.4929 (18)	124
C16—H16···Se1	0.95	2.89	3.4346 (19)	118
C26—H26···Se1	0.95	3.07	3.5268 (19)	111
C33—H33···Se1^{iii}	0.95	2.88	3.676 (2)	142

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+2, -y+1, -z$.

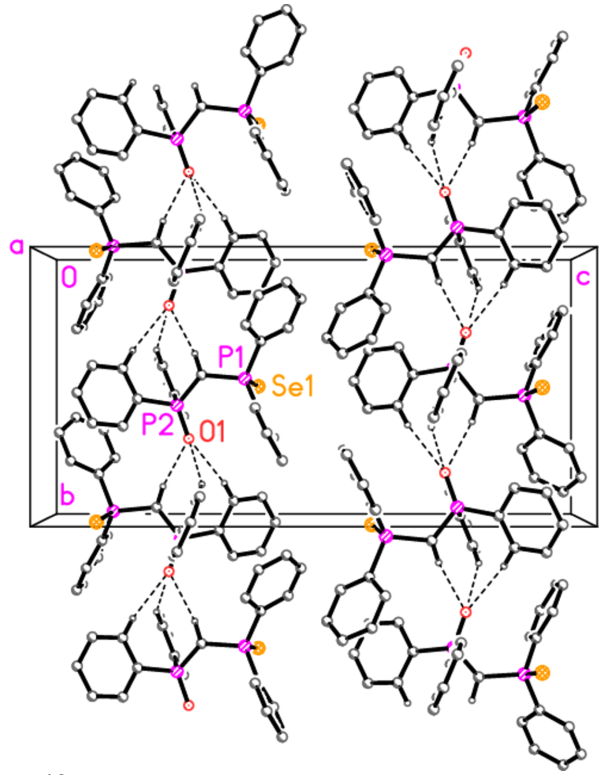


Figure 10
Packing diagram of compound **2** viewed parallel to the *a* axis in the region $x \simeq 0.75$. Dashed lines indicate hydrogen bonds.

Table 13
C—H···π contacts for compounds **2**–**5**.

Criterion: H···π < 2.9 Å. The symbol *Cgn* indicates the centre of gravity of the ring with atoms C*n*1–C*n*6.

Compd	Interaction	H···π (Å)	C—H···π (°)	Operator
2	C23—H23···*Cg*4	2.84	120.8	$-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$
3	C43—H43···*Cg*2	2.65	138.0	$-x+1, -y+2, -z$
3	C34—H34···*Cg*4	2.65	152.1	$x+1, y, z$
3	C42—H42···*Cg*1′	2.88	134.3	$-x+1, -y+1, -z+1$
3	C43—H43···*Cg*2′	2.62	166.5	$-x+1, -y+1, -z+1$
4	C25—H25···*Cg*1	2.69	149.0	$x+1, y, z$
4	C14—H14···*Cg*2	2.58	150.5	$-x+1, -y+1, -z+2$
5	C34—H34···*Cg*2	2.58	153.6	$x, y-1, z$
5	C45—H45···*Cg*3	2.86	155.4	$x-1, y, z$
5	C14—H14···*Cg*4	2.78	151.6	$x, y+1, z$

two *ortho*-H atoms from different rings (Table 9), linking neighbouring molecules *via* a 2_1 screw axis to form chains running parallel to the *b* axis. In compound **3** (Fig. 3 and Table 10), the two molecules in the asymmetric unit are linked in pairs by two similar trios of hydrogen bonds to the acceptor atoms O2 and O2′. The dimers are further linked by the hydrogen bond H12···O1($-x+1, -y+1, -z$) to form inversion-symmetric pairs.

The packing of homologous diphosphane dioxides Ph_2PO–$(CH_2)_n$–PPh_2O has been discussed in detail by Calcagno *et al.* (2000).

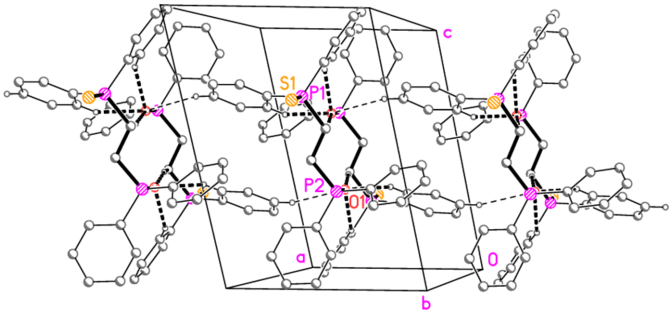


Figure 11
Packing diagram of compound **4** viewed perpendicular to the *ac* plane. Short hydrogen bonds (see text) are drawn as thick dashed lines and the longer hydrogen bond (connecting dimers horizontally) as thin dashed lines.

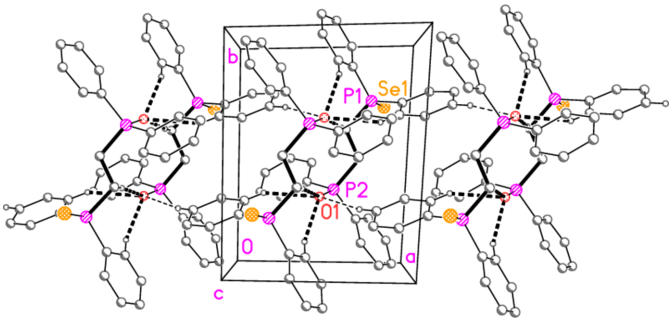


Figure 12
Packing diagram of compound **5** viewed parallel to the *c* axis. Short hydrogen bonds (see text) are drawn as thick dashed lines and the longer hydrogen bond (connecting dimers horizontally) as thin dashed lines.

In the packing of compound **4**, the O atom accepts three short hydrogen bonds from one bridge and two *ortho*-H atoms, forming inversion-symmetric pairs. These pairs are further linked by a longer hydrogen bond from a *para*-H atom to the O atom, leading to a ribbon structure propagating parallel to the *a*-axis direction (Fig. 11 and Table 11). The packing of **5** (Fig. 12 and Table 12) is exactly analogous to that of **4**, which begs the question whether **4** and **5** might indeed be isotypic despite their different cell constants and somewhat different ring orientations. This would require a non-standard triclinic cell to be found for one structure in which the coordinates were then effectively the same as the other structure, but we are not aware of any systematic procedure for finding such a cell. One indication that the two structures are indeed different is the analysis of longer contacts, such as the borderline hydrogen bonds in Tables 11 and 12. The shortest intermolecular H···S contacts for **4** are H42···S1 at 2.92 Å and H44···S1 at 2.91 Å, *via* different inversion operators, and the next longest (not in Table 11) is H33···S1 at 3.16 Å *via* translation. For compound **5**, there is only one borderline H33···Se1 contact of 2.88 Å, and the next longest (not in Table 12) are two H44···Se1 contacts of 3.45 and 3.48 Å, the former *via* inversion and the latter by translation. Finally, the projections of the two structures along the *a* axes (Figs. 13 and 14) are significantly different.

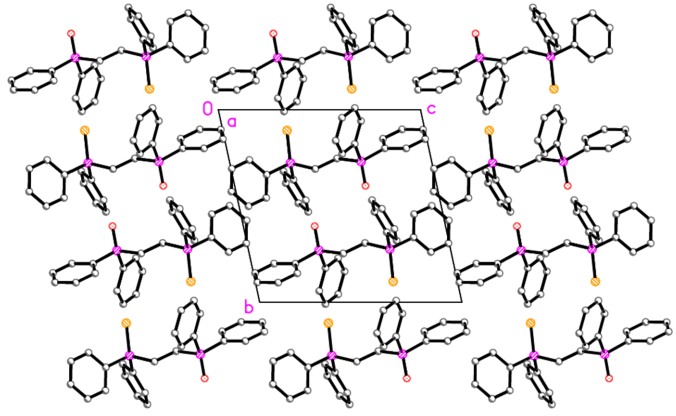


Figure 13
Projection of the structure of **4** along the *a* axis (H atoms omitted).

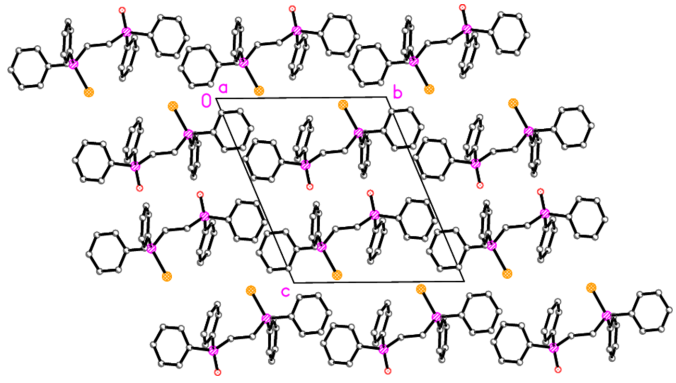


Figure 14
Projection of the structure of **5** along the *a* axis (H atoms omitted).

Table 14
Cell constants (Å, °) of dppe homo- and hetero-dichalcogenides $E^1PPh_2CH_2CH_2PPh_2E^2$.

Rounded to three and two decimal places for axis lengths and angles respectively, without s.u. values.

E^1, E^2	Reference	Refcode	*a*	*b*	*c*	α	β	γ	Space group	*Z*
O, O	Calcagno *et al.* (2000)	WITHEM	16.338	5.817	23.199	90	102.26	90	*Cc*	8
S, S[a]	Rheingold & Yap (1996)	NADXIZ	15.810	9.064	17.448	90	107.92	90	*C*2/*c*	4
Se, Se	Risto *et al.* (2007)	XILSIV	10.322	10.843	12.285	90.93	108.13	112.14	$P\overline{1}$	2
Se, Se	Nemati Kharat *et al.* (2008)	XILSIV01	15.828	9.206	16.967	90	121.65	90	*C*2/*c*	4
Se, Se[b]	Nemati Kharat *et al.* (2008)		15.828	9.206	17.820	90	107.47	90	*C*2/*c*	4
Se, Se	Upmann & Jones (2018)	XILSIV02	9.594	16.196	15.800	90	103.38	90	$P2_1/c$	4
Se, Se[c]	Upmann & Jones (2018)	DEQWOO	12.131	16.100	14.964	90	111.59	90	$P2_1/c$	4
Se, Se[d]	Brown *et al.* (1980)	PSETXY	8.958	14.822	15.946	81.20	83.05	79.38	$P\overline{1}$	6
Te, Te	Jeremias *et al.* (2014)	KUFTIQ	17.383	9.831	14.572	90	99.73	90	*C*2/*c*	4
S, O (**4**)	This work		8.419	12.074	12.144	74.90	77.43	72.77	$P\overline{1}$	2
Se, O (**4**)	This work		8.300	11.330	13.585	66.39	75.87	82.41	$P\overline{1}$	2

Notes: (*a*) one of seven reports of the same structure; six are Database Communications and the other reports cell constants only. (*b*) Cell transformed by the matrix ($\overline{1}00/0\overline{1}0/101$); the structure can be refined in this cell using the deposited data. (*c*) Dichloromethane 1/3-solvate. (*d*) *p*-Xylene solvate.

We have presented the packing of compounds **2**–**5** largely in terms of hydrogen bonding, which we subjectively judge to be the most important type of intermolecular contact. However, all these structures also show short C—H···π contacts, which are shown in Table 13 for completeness. For compound **2**, the H···π contact lies within the hydrogen-bonded chains; for all other structures, the H···π contacts connect the hydrogen-bonded units. None of the structures displays intercentroid distances less than 3.9 Å between phenyl rings, except intramolecularly for compound **3** (as discussed above).

4. Database survey

The searches employed the routine *ConQuest* (Bruno *et al.*, 2002), part of Version 2024.1.0 of the Cambridge Structural Database (CSD; Groom *et al.*, 2016). Several structures thus found, related to those of **1**–**5**, are referred to in the sections *Chemical context* and *Structural commentary*.

The mono-oxo derivative analogous to **1** does not appear in the CSD. The corresponding disulfide also does not appear, but we have determined the structure of its diiodine adduct (CSD refcode CIXRIN; Strey & Jones, 2018).

Several structures of homo-dichalcogenides dppmE_2 have been reported. The dioxide is known only in the form of various solvates: its chloroform solvate (EBIXEV; Grachova *et al.*, 2016) and three isotypic forms with two molecules of dppmE_2 and half a solvent molecule in the asymmetric unit, namely the toluene (GASMAR; Lu *et al.*, 2017), dichloromethane (**3**, see above; previously deposited by the authors as a *Database Communication*, KURDIM, Jones & Taouss, 2015) and benzene (OXPMPS; Antipin *et al.*, 1980) quarter-solvates. The solvent lies on an inversion centre and is thus disordered in the toluene and dichloromethane quarter-solvates. The disulfide is known as two different solvent-free polymorphs, both of which have been reported twice: in $P2_1/n$ (ZUKHAO: Carmalt *et al.*, 1996; ZUKHAO03: Clegg & Scott, 2017) and in *C*2/*c* with imposed twofold symmetry [ZUKHAO01 (Hursthouse *et al.*, 2003) and ZUKHAO02 (Thirumoorthi & Chivers, 2012)]. The diselenide, refcode family DPPSEM, shows the same two forms (isotypic to the disulfides); two further supposedly different polymorphs are in fact probably identical to these, but were presented in non-standard settings [as discussed in detail by us in Taouss & Jones (2013)].

Because the chloroform solvate of dppmO_2 is a deposited structure (Grachova *et al.*, 2016), we have used the deposited data to investigate the packing (Fig. 15). The compound crystallizes in $P2_1/c$ with Z = 4. Analogously to the P═O derivatives discussed above, molecules of dppmO_2 are linked by the *c*-glide operator to form ribbons parallel to the *c* axis by three C—H···O interactions to the same O atom. The shortest H···O contact, 2.28 Å, involves a methylene H atom. The other O atom accepts a short hydrogen bond, again 2.28 Å, from the chloroform H atom (also known to be a good hydrogen-bond donor). The chloroform molecules are linked, also parallel to the *c* axis, by a short Cl···Cl contact of 3.4967 (9) Å. The overall pattern is a layer structure parallel to (100).

Several structures of homo-dichalcogenides dppeE_2 have been reported and are summarized in Table 14, together with **4** and **5**. Despite the apparent scope for isotypy, none of the

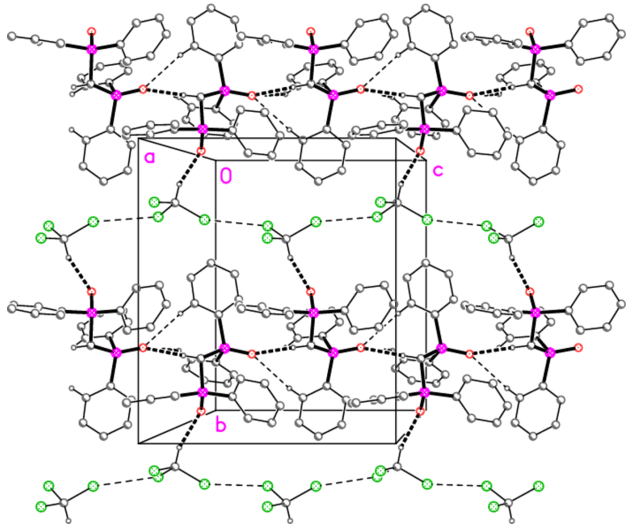


Figure 15
The packing of dppmO_2·$CHCl_3$ (Grachova *et al.*, 2016) viewed perpendicular to the *bc* plane in the region $x \simeq 0.25$. Dashed lines indicate short H···O contacts (thick), or longer H···O contacts and Cl···Cl contacts (thin).

Table 15
Experimental details.

Experiments were carried out at 100 K with Mo $K\alpha$ radiation using a Oxford Diffraction Xcalibur Eos diffractometer. Absorption was corrected for by multi-scan methods (*CrysAlis PRO*; Rigaku OD, 2013). H-atom parameters were constrained.

	1	**2**	**3**
Crystal data			
Chemical formula	$C_{13}H_{30}P_2S$	$C_{25}H_{22}OP_2Se$	$C_{24}H_{22}O_2P_2{\cdot}0.25CH_2Cl_2$
M_r	280.37	479.32	437.60
Crystal system, space group	Monoclinic, $P2_1/n$	Orthorhombic, $P2_12_12_1$	Triclinic, $P\overline{1}$
a, b, c (Å)	10.7521 (5), 11.5873 (5), 14.0337 (8)	8.6854 (2), 11.1358 (2), 22.5660 (3)	9.0964 (5), 13.2138 (6), 19.4218 (8)
α, β, γ (°)	90, 110.005 (6), 90	90, 90, 90	74.009 (4), 79.568 (4), 84.861 (4)
V (Å^3)	1642.92 (15)	2182.56 (7)	2205.09 (19)
Z	4	4	4
μ (mm^{-1})	0.37	1.88	0.28
Crystal size (mm)	0.4 × 0.1 × 0.08	0.15 × 0.08 × 0.04	0.2 × 0.2 × 0.15
Data collection			
T_{min}, T_{max}	0.797, 1.000	0.915, 1.000	0.922, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	58038, 4787, 3873	84057, 6637, 6143	116593, 11405, 9018
R_{int}	0.060	0.046	0.080
θ values (°)	θ_{max} = 30.0, θ_{min} = 2.3	θ_{max} = 30.9, θ_{min} = 2.5	θ_{max} = 28.7, θ_{min} = 2.2
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.704	0.723	0.676
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.094, 1.04	0.027, 0.055, 1.06	0.045, 0.112, 1.03
No. of reflections	4787	6637	11405
No. of parameters	153	263	550
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.31	0.38, −0.31	0.51, −0.41
Absolute structure	–	Refined as an inversion twin.	–
Absolute structure parameter	–	0.452 (6)	–

	4	**5**
Crystal data		
Chemical formula	$C_{26}H_{24}OP_2S$	$C_{26}H_{24}OP_2Se$
M_r	446.45	493.35
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
a, b, c (Å)	8.4187 (3), 12.0744 (4), 12.1438 (5)	8.3800 (4), 11.3301 (6), 13.5851 (8)
α, β, γ (°)	74.896 (3), 77.433 (4), 72.768 (3)	66.391 (4), 75.870 (5), 82.410 (4)
V (Å^3)	1125.02 (8)	1145.29 (11)
Z	2	2
μ (mm^{-1})	0.30	1.80
Crystal size (mm)	0.2 × 0.1 × 0.06	0.2 × 0.18 × 0.04
Data collection		
T_{min}, T_{max}	0.988, 1.000	0.950, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	60880, 6684, 6008	54824, 6630, 5686
R_{int}	0.030	0.048
θ values (°)	θ_{max} = 30.8, θ_{min} = 2.2	θ_{max} = 30.0, θ_{min} = 2.5
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.721	0.704
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.079, 1.04	0.034, 0.083, 1.08
No. of reflections	6684	6630
No. of parameters	271	271
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.44, −0.27	0.94, −0.53

Computer programs: *CrysAlis PRO* (Rigaku OD, 2013), *SHELXS* (Sheldrick, 2008), *SHELXL2019* (Sheldrick, 2015), *XP* (Bruker, 1998) and *publCIF* (Westrip, 2010).

structures appears isotypic at first sight (but see *Supramolecular features* for the cases of **4** and **5**). However, the second polymorph of the diselenide, refcode XILSIV01 (Nemati Kharat *et al.*, 2008), was not presented in a reduced cell. The reduced cell (and successful refinement in this cell using the deposited data) shows that the structure is isotypic to the disulfide.

A search for all structures containing the moiety EPh$_2$PCH$_2$CH$_2$PPh$_2E$ gave 42 hits. The absolute P—C—C—P torsion angles all lay between 160 and 180° (with many residues lying across inversion centres), except for the disulfides and diselenides discussed here, which have values of *ca* 155°.

5. Synthesis and crystallization

Details of the preparations of compounds **1**, **2**, **4** and **5** are taken from the PhD thesis of Daniel Upmann (Upmann, 2015), but are unfortunately not complete; some elemental

analyses could not be performed because the facility was closed for a long period, and some crystallization details have been lost.

Compound **1**: bis(diisopropylphospano)methylene (1.033 g, 4.2 mmol) was dissolved in toluene (20 ml). Sulfur (130 mg, 4.2 mmol) was added and the mixture stirred for 3 h at room temperature. After warming, the sulfur had completely dissolved and a mixture of the starting material and its mono- and disulfide formed. The toluene was removed *in vacuo* and the residue suspended in *n*-pentane (10 ml); the monosulfide was insoluble, but the starting material and the disulfide dissolved. The flask was stored at −25 °C overnight, whereby colourless crystals of the monosulfide formed. On redissolving the monosulfide, an equilibrium mixture of all three compounds re-formed.

Compound **2**: bis(diphenylphosphanomethane) monoxide, dppmO (174 mg, 0.5 mmol), was dissolved in toluene (15 ml). Selenium (36 mg, 0.5 mmol) was added and the solution refluxed for 3 h. Excess selenium was then filtered off. The solvent was removed *in vacuo*. ^{31}P NMR (81 MHz, $CDCl_3$): δ 25.89 (*dd*, J_{PP} = 19.2 Hz, J_{PSe} = 732.2 Hz, P═Se), 23.90 (*d*, J_{PP} = 19.2 Hz, P═O).

Compound **3**: the monoxide dppmO was obtained according to the method of Grushin (2001) and recrystallized as colourless needles by liquid diffusion of pentane into a solution in dichloromethane. The dioxide $dppmO_2$ was obtained by oxidizing dppmO with urea/H_2O_2 and was recrystallized as small colourless blocks by liquid diffusion of diisopropyl ether into a solution in dichloromethane.

Compound **4**: bis(1,2-diphenylphosphanoethane) monoxide, dppeO (496 mg, 1.2 mmol), was dissolved in 20 ml dichloromethane. Sulfur (38 mg, 1.2 mmol) was added and the solution refluxed for 3 h. Excess sulfur was then filtered off. The solvent was removed *in vacuo*. Elemental analysis (%) found: C 68.24, H 5.11, S 7.28; calculated: C 68.89, H 4.82, S 7.28. ^{31}P NMR (81 MHz, $CDCl_3$): δ 45.31 (*d*, J_{PP} = 57.3 Hz, P═S), 33.19 (*d*, J_{PP} = 57.3 Hz, P═O). Colourless crystals were obtained by chance from a failed synthesis of a gold(I) complex of **4**.

Compound **5**: bis(1,2-diphenylphosphanoethane) monoxide, dppeO (460 mg, 1.1 mmol), was dissolved in 20 ml dichloromethane. Selenium (88 mg, 1.1 mmol) was added and the solution refluxed for 4 h. Excess selenium was then filtered off. The solvent was removed *in vacuo*. ^{31}P NMR (81 MHz, $CDCl_3$): δ 38.01 (*dd*, J_{PP} = 59.7 Hz, J_{PSe} = 729.6 Hz, P═Se), 33.09 (*d*, J_{PP} = 59.7 Hz, P═O).

6. Refinement

Details of the measurements and refinements are given in Table 15. Structures were refined anisotropically on F^2. Methyl groups were refined as idealized rigid groups with C—H = 0.98 Å, H—C—H = 109.5° and U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were included at calculated positions and refined using a riding model with C—H = 1.00 (methine), 0.99 (methylene) or 0.95 Å (aromatic), and U_{iso}(H) = 1.2U_{eq}(C).

Exceptions and special features. Badly fitting reflections ($\Delta/\sigma > 7$) were omitted as follows: **3**, two reflections; **4**, one reflection; **5**, two reflections. The Flack parameter for **2** refined to 0.452 (6), indicating an inversion twin; the compound itself is achiral. The weighting parameters *a* and *b* (Sheldrick, 2015) for **2** oscillated slightly. The dichloromethane molecule of **3** is disordered over an inversion centre and was thus refined as a half-occupied site. Appropriate restraints were used to improve refinement stability, but the dimensions of disordered moieties should always be interpreted with caution.

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Syntheses and structures of one diphosphane monochalcogenide, one diphosphane dioxide and three diphosphane hetero-dichalcogenides

Daniel Upmann, Christina Taouss and Peter G. Jones

Computing details

{[Bis(propan-2-yl)phosphanyl]methyl}bis(propan-2-yl)-l^{5}-phosphanethione (1)

Crystal data

$C_{13}H_{30}P_2S$
M_r = 280.37
Monoclinic, $P2_1/n$
a = 10.7521 (5) Å
b = 11.5873 (5) Å
c = 14.0337 (8) Å
β = 110.005 (6)°
V = 1642.92 (15) Å^3
Z = 4
$F(000)$ = 616
D_x = 1.133 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9834 reflections
θ = 2.7–29.9°
μ = 0.37 mm^{-1}
T = 100 K
Prism, colourless
0.4 × 0.1 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2013)
T_{min} = 0.797, T_{max} = 1.000
58038 measured reflections
4787 independent reflections
3873 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 30.0°, θ_{min} = 2.3°
h = −15→15
k = −16→15
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.094
S = 1.04
4787 reflections
153 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 0.560P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.32284 (4)	0.23721 (3)	0.40082 (3)	0.02767 (10)
P1	0.24690 (3)	0.36565 (3)	0.30790 (3)	0.01642 (9)
P2	0.41977 (3)	0.30785 (3)	0.18130 (3)	0.01742 (9)
C10	0.36238 (13)	0.42201 (11)	0.25042 (10)	0.0179 (3)
H10A	0.439893	0.455401	0.303944	0.021*
H10B	0.318991	0.484574	0.202555	0.021*
C1	0.20288 (13)	0.49104 (12)	0.36964 (10)	0.0186 (3)
H1	0.164859	0.551391	0.316718	0.022*
C2	0.10171 (13)	0.32501 (12)	0.19804 (11)	0.0198 (3)
H2	0.137332	0.284353	0.149993	0.024*
C3	0.42780 (14)	0.39160 (12)	0.07041 (11)	0.0216 (3)
H3	0.485370	0.460853	0.094994	0.026*
C4	0.59539 (13)	0.29559 (12)	0.26726 (11)	0.0199 (3)
H4	0.591853	0.283797	0.336875	0.024*
C11	0.09787 (15)	0.45995 (13)	0.41637 (12)	0.0268 (3)
H11A	0.132868	0.400595	0.468436	0.040*
H11B	0.018741	0.430473	0.363392	0.040*
H11C	0.074731	0.528862	0.447202	0.040*
C12	0.32416 (14)	0.54260 (14)	0.45107 (11)	0.0256 (3)
H12A	0.297374	0.610308	0.481190	0.038*
H12B	0.389541	0.565638	0.420255	0.038*
H12C	0.363215	0.484867	0.503893	0.038*
C21	0.02498 (14)	0.42970 (13)	0.13997 (12)	0.0252 (3)
H21A	−0.036481	0.404598	0.073972	0.038*
H21B	0.087299	0.485994	0.129783	0.038*
H21C	−0.024860	0.465355	0.179083	0.038*
C22	0.00841 (15)	0.23935 (13)	0.22206 (13)	0.0282 (3)
H22A	−0.041117	0.278813	0.259519	0.042*
H22B	0.060094	0.176181	0.263386	0.042*
H22C	−0.053580	0.208141	0.158689	0.042*
C31	0.28855 (16)	0.43092 (15)	0.00533 (12)	0.0304 (3)
H31A	0.291441	0.465687	−0.057541	0.046*
H31B	0.256291	0.487963	0.042906	0.046*
H31C	0.228847	0.364269	−0.011008	0.046*
C32	0.48687 (16)	0.31567 (13)	0.00732 (12)	0.0273 (3)
H32A	0.440147	0.241580	−0.006657	0.041*
H32B	0.580907	0.302312	0.045073	0.041*
H32C	0.477355	0.354525	−0.056820	0.041*
C41	0.68237 (14)	0.40175 (13)	0.27433 (12)	0.0262 (3)
H41A	0.765729	0.392423	0.331040	0.039*
H41B	0.636158	0.470598	0.285421	0.039*
H41C	0.701015	0.410339	0.211051	0.039*
C42	0.66105 (14)	0.18707 (13)	0.24361 (12)	0.0261 (3)
H42A	0.670636	0.195306	0.176963	0.039*
H42B	0.605946	0.119585	0.243231	0.039*

H42C	0.748541	0.176945	0.295508	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0298 (2)	0.02388 (19)	0.0315 (2)	0.00855 (14)	0.01323 (16)	0.01068 (15)
P1	0.01410 (16)	0.01615 (16)	0.02109 (18)	0.00181 (12)	0.00871 (13)	0.00181 (12)
P2	0.01393 (16)	0.01826 (17)	0.02196 (18)	0.00042 (12)	0.00857 (13)	−0.00095 (13)
C10	0.0147 (6)	0.0186 (6)	0.0227 (7)	−0.0003 (5)	0.0094 (5)	−0.0014 (5)
C1	0.0164 (6)	0.0206 (6)	0.0207 (7)	0.0016 (5)	0.0089 (5)	−0.0008 (5)
C2	0.0153 (6)	0.0191 (6)	0.0261 (7)	−0.0009 (5)	0.0086 (5)	−0.0029 (5)
C3	0.0235 (7)	0.0220 (7)	0.0216 (7)	0.0023 (5)	0.0107 (6)	0.0006 (5)
C4	0.0140 (6)	0.0237 (7)	0.0233 (7)	0.0012 (5)	0.0079 (5)	0.0025 (5)
C11	0.0249 (7)	0.0290 (8)	0.0334 (8)	−0.0005 (6)	0.0189 (6)	−0.0037 (6)
C12	0.0208 (7)	0.0317 (8)	0.0244 (7)	0.0002 (6)	0.0078 (6)	−0.0045 (6)
C21	0.0199 (7)	0.0245 (7)	0.0282 (8)	0.0016 (5)	0.0042 (6)	−0.0012 (6)
C22	0.0206 (7)	0.0231 (7)	0.0442 (9)	−0.0050 (6)	0.0153 (7)	−0.0037 (6)
C31	0.0300 (8)	0.0381 (9)	0.0229 (7)	0.0108 (7)	0.0088 (6)	0.0030 (6)
C32	0.0322 (8)	0.0292 (8)	0.0258 (7)	0.0052 (6)	0.0168 (6)	0.0019 (6)
C41	0.0158 (6)	0.0302 (8)	0.0331 (8)	−0.0041 (6)	0.0091 (6)	−0.0019 (6)
C42	0.0194 (7)	0.0262 (7)	0.0345 (8)	0.0059 (6)	0.0116 (6)	0.0043 (6)

Geometric parameters (Å, º)

S1—P1	1.9631 (5)	C11—H11C	0.9800
P1—C10	1.8177 (13)	C12—H12A	0.9800
P1—C1	1.8349 (13)	C12—H12B	0.9800
P1—C2	1.8409 (14)	C12—H12C	0.9800
P2—C3	1.8609 (14)	C21—H21A	0.9800
P2—C10	1.8658 (13)	C21—H21B	0.9800
P2—C4	1.8662 (14)	C21—H21C	0.9800
C10—H10A	0.9900	C22—H22A	0.9800
C10—H10B	0.9900	C22—H22B	0.9800
C1—C11	1.5301 (19)	C22—H22C	0.9800
C1—C12	1.531 (2)	C31—H31A	0.9800
C1—H1	1.0000	C31—H31B	0.9800
C2—C22	1.5286 (19)	C31—H31C	0.9800
C2—C21	1.535 (2)	C32—H32A	0.9800
C2—H2	1.0000	C32—H32B	0.9800
C3—C32	1.5317 (19)	C32—H32C	0.9800
C3—C31	1.532 (2)	C41—H41A	0.9800
C3—H3	1.0000	C41—H41B	0.9800
C4—C41	1.5278 (19)	C41—H41C	0.9800
C4—C42	1.5330 (19)	C42—H42A	0.9800
C4—H4	1.0000	C42—H42B	0.9800
C11—H11A	0.9800	C42—H42C	0.9800
C11—H11B	0.9800		

C10—P1—C1	104.53 (6)	H11B—C11—H11C	109.5
C10—P1—C2	103.45 (6)	C1—C12—H12A	109.5
C1—P1—C2	108.43 (6)	C1—C12—H12B	109.5
C10—P1—S1	112.03 (5)	H12A—C12—H12B	109.5
C1—P1—S1	113.73 (5)	C1—C12—H12C	109.5
C2—P1—S1	113.78 (5)	H12A—C12—H12C	109.5
C3—P2—C10	100.56 (6)	H12B—C12—H12C	109.5
C3—P2—C4	104.72 (6)	C2—C21—H21A	109.5
C10—P2—C4	99.17 (6)	C2—C21—H21B	109.5
P1—C10—P2	111.94 (7)	H21A—C21—H21B	109.5
P1—C10—H10A	109.2	C2—C21—H21C	109.5
P2—C10—H10A	109.2	H21A—C21—H21C	109.5
P1—C10—H10B	109.2	H21B—C21—H21C	109.5
P2—C10—H10B	109.2	C2—C22—H22A	109.5
H10A—C10—H10B	107.9	C2—C22—H22B	109.5
C11—C1—C12	109.70 (12)	H22A—C22—H22B	109.5
C11—C1—P1	111.31 (10)	C2—C22—H22C	109.5
C12—C1—P1	111.72 (9)	H22A—C22—H22C	109.5
C11—C1—H1	108.0	H22B—C22—H22C	109.5
C12—C1—H1	108.0	C3—C31—H31A	109.5
P1—C1—H1	108.0	C3—C31—H31B	109.5
C22—C2—C21	110.86 (11)	H31A—C31—H31B	109.5
C22—C2—P1	114.45 (11)	C3—C31—H31C	109.5
C21—C2—P1	112.97 (10)	H31A—C31—H31C	109.5
C22—C2—H2	105.9	H31B—C31—H31C	109.5
C21—C2—H2	105.9	C3—C32—H32A	109.5
P1—C2—H2	105.9	C3—C32—H32B	109.5
C32—C3—C31	109.88 (12)	H32A—C32—H32B	109.5
C32—C3—P2	109.32 (10)	C3—C32—H32C	109.5
C31—C3—P2	109.76 (10)	H32A—C32—H32C	109.5
C32—C3—H3	109.3	H32B—C32—H32C	109.5
C31—C3—H3	109.3	C4—C41—H41A	109.5
P2—C3—H3	109.3	C4—C41—H41B	109.5
C41—C4—C42	110.95 (11)	H41A—C41—H41B	109.5
C41—C4—P2	116.25 (10)	C4—C41—H41C	109.5
C42—C4—P2	111.19 (10)	H41A—C41—H41C	109.5
C41—C4—H4	105.9	H41B—C41—H41C	109.5
C42—C4—H4	105.9	C4—C42—H42A	109.5
P2—C4—H4	105.9	C4—C42—H42B	109.5
C1—C11—H11A	109.5	H42A—C42—H42B	109.5
C1—C11—H11B	109.5	C4—C42—H42C	109.5
H11A—C11—H11B	109.5	H42A—C42—H42C	109.5
C1—C11—H11C	109.5	H42B—C42—H42C	109.5
H11A—C11—H11C	109.5		

C1—P1—C10—P2	−179.43 (7)	S1—P1—C2—C22	38.66 (11)
C2—P1—C10—P2	−65.98 (8)	C10—P1—C2—C21	−71.40 (11)
S1—P1—C10—P2	56.97 (8)	C1—P1—C2—C21	39.18 (12)

C3—P2—C10—P1	142.57 (8)	S1—P1—C2—C21	166.80 (8)
C4—P2—C10—P1	−110.46 (8)	C10—P2—C3—C32	174.50 (10)
C10—P1—C1—C11	176.18 (10)	C4—P2—C3—C32	72.00 (11)
C2—P1—C1—C11	66.32 (11)	C10—P2—C3—C31	−64.91 (11)
S1—P1—C1—C11	−61.32 (11)	C4—P2—C3—C31	−167.41 (10)
C10—P1—C1—C12	−60.81 (11)	C3—P2—C4—C41	38.33 (12)
C2—P1—C1—C12	−170.67 (10)	C10—P2—C4—C41	−65.22 (11)
S1—P1—C1—C12	61.68 (11)	C3—P2—C4—C42	−89.89 (11)
C10—P1—C2—C22	160.46 (10)	C10—P2—C4—C42	166.56 (10)
C1—P1—C2—C22	−88.96 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C21—H21*A*···S1^{i}	0.98	2.89	3.8390 (16)	162
C22—H22*B*···S1	0.98	2.91	3.4526 (17)	116

Symmetry code: (i) *x*−1/2, −*y*+1/2, *z*−1/2.

[(Diphenylphosphoryl)methyl]diphenyl-λ^{5}-phosphanethione (2)

Crystal data

$C_{25}H_{22}OP_2Se$
M_r = 479.32
Orthorhombic, $P2_12_12_1$
a = 8.6854 (2) Å
b = 11.1358 (2) Å
c = 22.5660 (3) Å
V = 2182.56 (7) Å^3
Z = 4
F(000) = 976
D_x = 1.459 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 19548 reflections
θ = 2.3–30.9°
μ = 1.88 mm^{-1}
T = 100 K
Needle, colourless
0.15 × 0.08 × 0.04 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2013)
T_{min} = 0.915, T_{max} = 1.000
84057 measured reflections
6637 independent reflections
6143 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 30.9°, θ_{min} = 2.5°
h = −12→12
k = −16→15
l = −32→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.055
S = 1.06
6637 reflections
263 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0242P)^2 + 0.8533P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Absolute structure: Refined as an inversion twin.

Absolute structure parameter: 0.452 (6)

Special details

Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.54735 (6)	0.46910 (5)	0.37309 (2)	0.01061 (11)
P2	0.49499 (6)	0.56866 (5)	0.24645 (2)	0.00988 (10)
Se1	0.31488 (2)	0.49805 (2)	0.39550 (2)	0.01649 (6)
O1	0.5144 (2)	0.69396 (14)	0.26865 (7)	0.0157 (3)
C1	0.5830 (2)	0.45543 (19)	0.29365 (9)	0.0111 (4)
H1A	0.695649	0.457516	0.287111	0.013*
H1B	0.545935	0.375615	0.280601	0.013*
C11	0.6754 (3)	0.58705 (18)	0.39935 (9)	0.0129 (4)
C12	0.8355 (3)	0.5764 (2)	0.39489 (12)	0.0223 (5)
H12	0.879313	0.507283	0.376851	0.027*
C13	0.9305 (3)	0.6663 (2)	0.41672 (13)	0.0266 (6)
H13	1.039053	0.659077	0.413179	0.032*
C14	0.8669 (3)	0.7670 (2)	0.44376 (12)	0.0241 (6)
H14	0.932055	0.828234	0.458988	0.029*
C15	0.7094 (3)	0.7778 (2)	0.44840 (13)	0.0262 (6)
H15	0.666152	0.846742	0.466816	0.031*
C16	0.6129 (3)	0.6883 (2)	0.42625 (11)	0.0194 (5)
H16	0.504405	0.696531	0.429544	0.023*
C21	0.6255 (3)	0.33022 (19)	0.40308 (10)	0.0121 (4)
C22	0.7518 (3)	0.2727 (2)	0.37722 (11)	0.0174 (5)
H22	0.799799	0.306651	0.343300	0.021*
C23	0.8073 (3)	0.1659 (2)	0.40100 (11)	0.0212 (5)
H23	0.892667	0.126631	0.383175	0.025*
C24	0.7380 (3)	0.1170 (2)	0.45074 (12)	0.0214 (5)
H24	0.776783	0.044606	0.467242	0.026*
C25	0.6121 (3)	0.1733 (2)	0.47661 (11)	0.0207 (5)
H25	0.564678	0.139141	0.510588	0.025*
C26	0.5556 (3)	0.2794 (2)	0.45284 (11)	0.0171 (4)
H26	0.469183	0.317550	0.470466	0.020*
C31	0.5946 (2)	0.5451 (2)	0.17669 (10)	0.0128 (4)
C32	0.6090 (3)	0.4322 (2)	0.14964 (10)	0.0164 (4)
H32	0.560691	0.363846	0.166594	0.020*
C33	0.6952 (3)	0.4211 (2)	0.09748 (11)	0.0219 (5)
H33	0.705321	0.344902	0.078911	0.026*
C34	0.7657 (3)	0.5209 (3)	0.07292 (10)	0.0238 (6)
H34	0.823508	0.512927	0.037385	0.029*
C35	0.7526 (3)	0.6323 (2)	0.09983 (12)	0.0228 (5)
H35	0.801278	0.700434	0.082757	0.027*
C36	0.6678 (3)	0.6445 (2)	0.15202 (11)	0.0172 (5)

H36	0.659944	0.720625	0.170739	0.021*
C41	0.2950 (2)	0.52914 (18)	0.23656 (9)	0.0113 (4)
C42	0.2498 (3)	0.4187 (2)	0.21289 (11)	0.0154 (4)
H42	0.324646	0.359389	0.203708	0.019*
C43	0.0944 (3)	0.3962 (2)	0.20284 (11)	0.0167 (5)
H43	0.063203	0.321485	0.186404	0.020*
C44	−0.0150 (2)	0.4821 (2)	0.21670 (10)	0.0190 (5)
H44	−0.120774	0.466675	0.209117	0.023*
C45	0.0291 (3)	0.5908 (2)	0.24165 (11)	0.0201 (5)
H45	−0.046525	0.648845	0.251920	0.024*
C46	0.1843 (3)	0.6147 (2)	0.25157 (10)	0.0158 (4)
H46	0.214725	0.689106	0.268520	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0114 (2)	0.0111 (3)	0.0093 (2)	0.00121 (18)	0.00010 (19)	0.00048 (18)
P2	0.0095 (2)	0.0096 (2)	0.0105 (2)	−0.00020 (19)	−0.0004 (2)	0.00069 (19)
Se1	0.01285 (9)	0.02132 (11)	0.01531 (9)	0.00316 (10)	0.00342 (8)	0.00226 (10)
O1	0.0176 (8)	0.0114 (7)	0.0181 (8)	−0.0020 (6)	−0.0010 (7)	−0.0006 (6)
C1	0.0117 (9)	0.0127 (9)	0.0088 (9)	0.0017 (7)	0.0005 (7)	−0.0006 (8)
C11	0.0173 (10)	0.0115 (9)	0.0100 (9)	−0.0001 (8)	−0.0023 (9)	0.0003 (8)
C12	0.0197 (11)	0.0164 (10)	0.0309 (12)	0.0035 (9)	−0.0023 (11)	−0.0054 (11)
C13	0.0179 (12)	0.0249 (13)	0.0372 (16)	−0.0009 (10)	−0.0070 (11)	−0.0012 (11)
C14	0.0312 (14)	0.0181 (12)	0.0231 (13)	−0.0069 (11)	−0.0072 (11)	−0.0020 (10)
C15	0.0315 (16)	0.0173 (12)	0.0298 (14)	−0.0020 (11)	0.0028 (12)	−0.0084 (10)
C16	0.0206 (12)	0.0165 (11)	0.0211 (12)	−0.0005 (10)	0.0023 (10)	−0.0040 (10)
C21	0.0151 (10)	0.0109 (10)	0.0102 (10)	−0.0006 (8)	−0.0034 (8)	−0.0001 (8)
C22	0.0207 (11)	0.0176 (11)	0.0140 (11)	0.0033 (9)	−0.0002 (9)	0.0006 (9)
C23	0.0246 (12)	0.0180 (11)	0.0210 (11)	0.0070 (10)	−0.0054 (11)	−0.0022 (9)
C24	0.0322 (13)	0.0122 (11)	0.0199 (12)	0.0009 (10)	−0.0120 (10)	0.0013 (9)
C25	0.0279 (13)	0.0183 (12)	0.0159 (11)	−0.0060 (10)	−0.0057 (10)	0.0048 (9)
C26	0.0192 (11)	0.0186 (11)	0.0134 (11)	−0.0028 (9)	−0.0026 (9)	0.0005 (9)
C31	0.0085 (9)	0.0193 (10)	0.0107 (9)	0.0002 (8)	−0.0007 (7)	0.0024 (8)
C32	0.0126 (10)	0.0204 (11)	0.0164 (10)	−0.0043 (9)	0.0013 (9)	−0.0019 (9)
C33	0.0183 (11)	0.0300 (12)	0.0174 (11)	−0.0013 (10)	0.0003 (10)	−0.0077 (10)
C34	0.0183 (10)	0.0387 (16)	0.0143 (10)	0.0028 (11)	0.0046 (8)	0.0028 (11)
C35	0.0198 (11)	0.0275 (13)	0.0210 (12)	0.0029 (10)	0.0060 (11)	0.0115 (11)
C36	0.0149 (11)	0.0185 (11)	0.0183 (11)	0.0023 (9)	0.0001 (9)	0.0068 (9)
C41	0.0100 (9)	0.0129 (10)	0.0110 (9)	−0.0002 (7)	−0.0003 (7)	0.0022 (7)
C42	0.0123 (10)	0.0127 (10)	0.0213 (11)	0.0011 (9)	0.0001 (9)	0.0007 (9)
C43	0.0145 (10)	0.0162 (11)	0.0195 (12)	−0.0028 (8)	−0.0013 (9)	0.0005 (9)
C44	0.0092 (9)	0.0293 (14)	0.0184 (10)	−0.0002 (9)	−0.0009 (8)	0.0024 (10)
C45	0.0125 (11)	0.0258 (12)	0.0219 (12)	0.0081 (9)	0.0007 (9)	−0.0018 (10)
C46	0.0156 (10)	0.0162 (10)	0.0157 (10)	0.0018 (9)	−0.0021 (10)	−0.0015 (8)

Geometric parameters (Å, º)

P1—C21	1.819 (2)	C24—C25	1.389 (4)
P1—C11	1.820 (2)	C24—H24	0.9500
P1—C1	1.826 (2)	C25—C26	1.387 (3)
P1—Se1	2.1063 (6)	C25—H25	0.9500
P2—O1	1.4920 (16)	C26—H26	0.9500
P2—C41	1.806 (2)	C31—C36	1.392 (3)
P2—C31	1.816 (2)	C31—C32	1.403 (3)
P2—C1	1.819 (2)	C32—C33	1.401 (3)
C1—H1A	0.9900	C32—H32	0.9500
C1—H1B	0.9900	C33—C34	1.385 (4)
C11—C16	1.391 (3)	C33—H33	0.9500
C11—C12	1.399 (3)	C34—C35	1.387 (4)
C12—C13	1.388 (4)	C34—H34	0.9500
C12—H12	0.9500	C35—C36	1.396 (3)
C13—C14	1.391 (4)	C35—H35	0.9500
C13—H13	0.9500	C36—H36	0.9500
C14—C15	1.378 (4)	C41—C46	1.396 (3)
C14—H14	0.9500	C41—C42	1.397 (3)
C15—C16	1.395 (4)	C42—C43	1.391 (3)
C15—H15	0.9500	C42—H42	0.9500
C16—H16	0.9500	C43—C44	1.384 (3)
C21—C26	1.396 (3)	C43—H43	0.9500
C21—C22	1.398 (3)	C44—C45	1.389 (3)
C22—C23	1.391 (3)	C44—H44	0.9500
C22—H22	0.9500	C45—C46	1.392 (3)
C23—C24	1.385 (4)	C45—H45	0.9500
C23—H23	0.9500	C46—H46	0.9500
C21—P1—C11	105.34 (10)	C23—C24—C25	120.4 (2)
C21—P1—C1	103.37 (10)	C23—C24—H24	119.8
C11—P1—C1	106.04 (10)	C25—C24—H24	119.8
C21—P1—Se1	113.48 (8)	C26—C25—C24	120.0 (2)
C11—P1—Se1	113.39 (8)	C26—C25—H25	120.0
C1—P1—Se1	114.26 (7)	C24—C25—H25	120.0
O1—P2—C41	112.21 (10)	C25—C26—C21	120.2 (2)
O1—P2—C31	111.86 (10)	C25—C26—H26	119.9
C41—P2—C31	108.43 (10)	C21—C26—H26	119.9
O1—P2—C1	113.85 (10)	C36—C31—C32	119.8 (2)
C41—P2—C1	107.91 (10)	C36—C31—P2	116.71 (18)
C31—P2—C1	101.96 (10)	C32—C31—P2	123.32 (17)
P2—C1—P1	116.49 (11)	C33—C32—C31	119.5 (2)
P2—C1—H1A	108.2	C33—C32—H32	120.2
P1—C1—H1A	108.2	C31—C32—H32	120.2
P2—C1—H1B	108.2	C34—C33—C32	120.1 (2)
P1—C1—H1B	108.2	C34—C33—H33	119.9
H1A—C1—H1B	107.3	C32—C33—H33	119.9

C16—C11—C12	119.2 (2)	C33—C34—C35	120.4 (2)
C16—C11—P1	119.29 (18)	C33—C34—H34	119.8
C12—C11—P1	121.49 (17)	C35—C34—H34	119.8
C13—C12—C11	120.3 (2)	C34—C35—C36	120.0 (2)
C13—C12—H12	119.9	C34—C35—H35	120.0
C11—C12—H12	119.9	C36—C35—H35	120.0
C12—C13—C14	120.1 (2)	C31—C36—C35	120.1 (2)
C12—C13—H13	119.9	C31—C36—H36	120.0
C14—C13—H13	119.9	C35—C36—H36	120.0
C15—C14—C13	119.9 (2)	C46—C41—C42	120.0 (2)
C15—C14—H14	120.1	C46—C41—P2	117.81 (16)
C13—C14—H14	120.1	C42—C41—P2	122.16 (17)
C14—C15—C16	120.5 (3)	C43—C42—C41	119.6 (2)
C14—C15—H15	119.8	C43—C42—H42	120.2
C16—C15—H15	119.8	C41—C42—H42	120.2
C11—C16—C15	120.1 (2)	C44—C43—C42	120.3 (2)
C11—C16—H16	119.9	C44—C43—H43	119.9
C15—C16—H16	119.9	C42—C43—H43	119.9
C26—C21—C22	119.4 (2)	C43—C44—C45	120.3 (2)
C26—C21—P1	118.80 (18)	C43—C44—H44	119.8
C22—C21—P1	121.79 (17)	C45—C44—H44	119.8
C23—C22—C21	120.2 (2)	C44—C45—C46	119.9 (2)
C23—C22—H22	119.9	C44—C45—H45	120.0
C21—C22—H22	119.9	C46—C45—H45	120.0
C24—C23—C22	119.9 (2)	C45—C46—C41	119.9 (2)
C24—C23—H23	120.1	C45—C46—H46	120.1
C22—C23—H23	120.1	C41—C46—H46	120.1

O1—P2—C1—P1	45.98 (15)	C24—C25—C26—C21	−0.3 (4)
C41—P2—C1—P1	−79.28 (14)	C22—C21—C26—C25	0.5 (3)
C31—P2—C1—P1	166.64 (12)	P1—C21—C26—C25	179.75 (18)
C21—P1—C1—P2	171.27 (12)	O1—P2—C31—C36	−2.9 (2)
C11—P1—C1—P2	−78.17 (14)	C41—P2—C31—C36	121.37 (18)
Se1—P1—C1—P2	47.47 (14)	C1—P2—C31—C36	−124.93 (18)
C21—P1—C11—C16	−130.2 (2)	O1—P2—C31—C32	172.74 (18)
C1—P1—C11—C16	120.64 (19)	C41—P2—C31—C32	−63.0 (2)
Se1—P1—C11—C16	−5.5 (2)	C1—P2—C31—C32	50.7 (2)
C21—P1—C11—C12	47.6 (2)	C36—C31—C32—C33	−0.9 (3)
C1—P1—C11—C12	−61.5 (2)	P2—C31—C32—C33	−176.45 (18)
Se1—P1—C11—C12	172.28 (18)	C31—C32—C33—C34	0.1 (4)
C16—C11—C12—C13	−0.5 (4)	C32—C33—C34—C35	0.4 (4)
P1—C11—C12—C13	−178.3 (2)	C33—C34—C35—C36	0.0 (4)
C11—C12—C13—C14	0.7 (4)	C32—C31—C36—C35	1.3 (3)
C12—C13—C14—C15	−0.5 (4)	P2—C31—C36—C35	177.13 (19)
C13—C14—C15—C16	0.1 (4)	C34—C35—C36—C31	−0.8 (4)
C12—C11—C16—C15	0.0 (4)	O1—P2—C41—C46	−1.7 (2)
P1—C11—C16—C15	177.9 (2)	C31—P2—C41—C46	−125.73 (18)
C14—C15—C16—C11	0.1 (4)	C1—P2—C41—C46	124.55 (17)

C11—P1—C21—C26	101.52 (19)	O1—P2—C41—C42	176.19 (17)
C1—P1—C21—C26	−147.39 (18)	C31—P2—C41—C42	52.1 (2)
Se1—P1—C21—C26	−23.1 (2)	C1—P2—C41—C42	−57.6 (2)
C11—P1—C21—C22	−79.3 (2)	C46—C41—C42—C43	1.8 (3)
C1—P1—C21—C22	31.8 (2)	P2—C41—C42—C43	−175.98 (19)
Se1—P1—C21—C22	156.09 (17)	C41—C42—C43—C44	−0.6 (4)
C26—C21—C22—C23	−0.1 (3)	C42—C43—C44—C45	−1.0 (4)
P1—C21—C22—C23	−179.31 (19)	C43—C44—C45—C46	1.4 (4)
C21—C22—C23—C24	−0.5 (4)	C44—C45—C46—C41	−0.2 (4)
C22—C23—C24—C25	0.7 (4)	C42—C41—C46—C45	−1.5 (3)
C23—C24—C25—C26	−0.3 (4)	P2—C41—C46—C45	176.46 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O1^{i}	0.99	2.37	3.342 (3)	168
C32—H32···O1^{i}	0.95	2.48	3.404 (3)	165
C42—H42···O1^{i}	0.95	2.40	3.261 (3)	151
C16—H16···Se1	0.95	2.86	3.416 (3)	118

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+1/2.

{[(Diphenylphosphoroso)methyl](phenyl)phosphoryl}benzene dichloromethane quarter-solvate (3)

Crystal data

$C_{24}H_{22}O_2P_2{\cdot}0.25CH_2Cl_2$
M_r = 437.60
Triclinic, $P\overline{1}$
a = 9.0964 (5) Å
b = 13.2138 (6) Å
c = 19.4218 (8) Å
α = 74.009 (4)°
β = 79.568 (4)°
γ = 84.861 (4)°
V = 2205.09 (19) Å^3
Z = 4
F(000) = 914
D_x = 1.318 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 14670 reflections
θ = 2.6–29.3°
μ = 0.28 mm^{-1}
T = 100 K
Block, colourless
0.2 × 0.2 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2013)
T_{min} = 0.922, T_{max} = 1.000
116593 measured reflections
11405 independent reflections
9018 reflections with $I > 2\sigma(I)$
R_{int} = 0.080
θ_{max} = 28.7°, θ_{min} = 2.2°
h = −12→12
k = −17→17
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.112
S = 1.03
11405 reflections
550 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 1.1605P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.51$ e Å^{-3}
	$\Delta\rho_{min} = -0.41$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
P1	0.39953 (5)	0.63934 (3)	0.10168 (2)	0.01689 (10)	
P2	0.41181 (5)	0.56713 (3)	0.26799 (2)	0.01441 (9)	
O1	0.38827 (16)	0.53093 (10)	0.09554 (7)	0.0246 (3)	
O2	0.42236 (15)	0.62400 (9)	0.32312 (6)	0.0213 (3)	
C1	0.48544 (19)	0.64518 (13)	0.17750 (8)	0.0170 (3)	
H1A	0.480436	0.719764	0.178862	0.020*	
H1B	0.592638	0.624226	0.166932	0.020*	
C11	0.5136 (2)	0.71993 (14)	0.02338 (9)	0.0208 (4)	
C12	0.5432 (3)	0.68464 (17)	−0.03904 (11)	0.0390 (6)	
H12	0.505631	0.619891	−0.039152	0.047*	
C13	0.6281 (3)	0.74464 (19)	−0.10127 (12)	0.0484 (7)	
H13	0.647548	0.720920	−0.144115	0.058*	
C14	0.6839 (2)	0.83749 (18)	−0.10153 (11)	0.0377 (5)	
H14	0.741595	0.877909	−0.144450	0.045*	
C15	0.6564 (2)	0.87265 (18)	−0.03942 (11)	0.0344 (5)	
H15	0.696103	0.936843	−0.039630	0.041*	
C16	0.5707 (2)	0.81427 (15)	0.02333 (10)	0.0259 (4)	
H16	0.551324	0.838593	0.065929	0.031*	
C21	0.21928 (19)	0.70815 (13)	0.11122 (8)	0.0179 (3)	
C22	0.1990 (2)	0.80478 (15)	0.12796 (9)	0.0234 (4)	
H22	0.280891	0.836210	0.137032	0.028*	
C23	0.0579 (2)	0.85503 (17)	0.13132 (11)	0.0318 (5)	
H23	0.043422	0.920749	0.143134	0.038*	
C24	−0.0619 (2)	0.80999 (19)	0.11755 (11)	0.0360 (5)	
H24	−0.157714	0.845156	0.119334	0.043*	
C25	−0.0415 (2)	0.71402 (18)	0.10127 (11)	0.0333 (5)	
H25	−0.123532	0.682856	0.092061	0.040*	
C26	0.0985 (2)	0.66287 (15)	0.09831 (10)	0.0247 (4)	
H26	0.111792	0.596511	0.087407	0.030*	
C31	0.22162 (19)	0.53730 (14)	0.27029 (9)	0.0199 (4)	
C32	0.1078 (2)	0.60369 (16)	0.29370 (10)	0.0286 (4)	
H32	0.130884	0.662975	0.307843	0.034*	
C33	−0.0412 (2)	0.5821 (2)	0.29617 (12)	0.0413 (6)	
H33	−0.119664	0.627322	0.311752	0.050*	
C34	−0.0748 (3)	0.4959 (2)	0.27616 (13)	0.0462 (6)	
H34	−0.176348	0.481733	0.278270	0.055*	
C35	0.0372 (2)	0.43007 (18)	0.25319 (12)	0.0386 (5)	
H35	0.013076	0.370700	0.239430	0.046*	
C36	0.1856 (2)	0.45027 (15)	0.25008 (10)	0.0258 (4)	

H36	0.263013	0.404686	0.234128	0.031*
C41	0.51949 (18)	0.44452 (13)	0.28214 (9)	0.0155 (3)
C42	0.54407 (19)	0.39531 (13)	0.35297 (9)	0.0183 (3)
H42	0.501223	0.425325	0.391596	0.022*
C43	0.6313 (2)	0.30246 (14)	0.36661 (10)	0.0223 (4)
H43	0.647600	0.268767	0.414721	0.027*
C44	0.6946 (2)	0.25873 (14)	0.31032 (10)	0.0239 (4)
H44	0.755408	0.195727	0.319816	0.029*
C45	0.6694 (2)	0.30698 (14)	0.23994 (10)	0.0236 (4)
H45	0.712167	0.276555	0.201509	0.028*
C46	0.5817 (2)	0.39947 (14)	0.22581 (9)	0.0201 (4)
H46	0.564102	0.432140	0.177770	0.024*
P1′	0.60121 (5)	0.84416 (3)	0.39530 (2)	0.01636 (10)
P2′	0.53896 (5)	0.92954 (3)	0.23368 (2)	0.01771 (10)
O1′	0.67976 (14)	0.93660 (10)	0.39745 (6)	0.0219 (3)
O2′	0.48429 (16)	0.88193 (10)	0.18250 (7)	0.0274 (3)
C1′	0.6103 (2)	0.82679 (13)	0.30512 (9)	0.0176 (3)
H1A′	0.556820	0.762202	0.310800	0.021*
H1B′	0.716701	0.812207	0.286858	0.021*
C11′	0.67860 (19)	0.72107 (14)	0.44606 (9)	0.0184 (3)
C12′	0.7620 (2)	0.72617 (15)	0.49859 (9)	0.0228 (4)
H12′	0.776945	0.792425	0.505930	0.027*
C13′	0.8233 (2)	0.63424 (16)	0.54031 (10)	0.0267 (4)
H13′	0.879653	0.637944	0.576141	0.032*
C14′	0.8023 (2)	0.53815 (15)	0.52970 (10)	0.0257 (4)
H14′	0.845585	0.475824	0.557733	0.031*
C15′	0.7181 (2)	0.53183 (15)	0.47828 (10)	0.0258 (4)
H15′	0.703262	0.465282	0.471371	0.031*
C16′	0.6554 (2)	0.62335 (14)	0.43676 (9)	0.0223 (4)
H16′	0.596664	0.618963	0.402005	0.027*
C21′	0.40595 (19)	0.84674 (13)	0.43455 (8)	0.0167 (3)
C22′	0.3105 (2)	0.77037 (14)	0.43304 (9)	0.0189 (3)
H22′	0.347154	0.716620	0.409650	0.023*
C23′	0.1623 (2)	0.77340 (14)	0.46574 (10)	0.0218 (4)
H23′	0.096786	0.722458	0.463933	0.026*
C24′	0.1092 (2)	0.85068 (15)	0.50118 (10)	0.0237 (4)
H24′	0.007545	0.852160	0.523721	0.028*
C25′	0.2035 (2)	0.92543 (14)	0.50385 (10)	0.0234 (4)
H25′	0.167063	0.977614	0.528697	0.028*
C26′	0.3518 (2)	0.92398 (14)	0.47003 (9)	0.0200 (4)
H26′	0.416317	0.975961	0.471168	0.024*
C31′	0.3915 (2)	1.00463 (14)	0.27502 (9)	0.0212 (4)
C32′	0.2460 (2)	0.96946 (17)	0.29050 (10)	0.0298 (4)
H32′	0.226501	0.905907	0.280558	0.036*
C33′	0.1297 (2)	1.0279 (2)	0.32052 (11)	0.0410 (6)
H33′	0.031148	1.002901	0.332382	0.049*
C34′	0.1563 (3)	1.1215 (2)	0.33319 (11)	0.0427 (6)
H34′	0.075587	1.161974	0.352218	0.051*

C35′	0.3000 (3)	1.15679 (17)	0.31834 (11)	0.0365 (5)	
H35′	0.318267	1.221001	0.327739	0.044*	
C36′	0.4179 (2)	1.09814 (14)	0.28957 (10)	0.0264 (4)	
H36′	0.516864	1.122072	0.279833	0.032*	
C41′	0.6890 (2)	1.01627 (13)	0.18825 (9)	0.0180 (3)	
C42′	0.6966 (2)	1.05825 (14)	0.11335 (10)	0.0240 (4)	
H42′	0.624224	1.040922	0.089283	0.029*	
C43′	0.8088 (2)	1.12466 (16)	0.07426 (10)	0.0288 (4)	
H43′	0.813686	1.152719	0.023336	0.035*	
C44′	0.9142 (2)	1.15053 (14)	0.10912 (10)	0.0252 (4)	
H44′	0.991232	1.196474	0.082111	0.030*	
C45′	0.9075 (2)	1.10947 (14)	0.18340 (10)	0.0213 (4)	
H45′	0.980028	1.127565	0.207087	0.026*	
C46′	0.7957 (2)	1.04216 (13)	0.22335 (9)	0.0195 (3)	
H46′	0.791704	1.013871	0.274205	0.023*	
C99	0.1355 (5)	0.4374 (3)	0.0229 (2)	0.0331 (9)	0.5
H99A	0.200423	0.453122	0.054064	0.040*	0.5
H99B	0.165315	0.366176	0.016500	0.040*	0.5
Cl1	−0.05305 (17)	0.43888 (11)	0.06594 (7)	0.0482 (3)	0.5
Cl2	0.16130 (17)	0.53056 (10)	−0.06237 (6)	0.0462 (3)	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0240 (2)	0.0155 (2)	0.01006 (19)	0.00079 (17)	−0.00394 (16)	−0.00130 (15)
P2	0.0183 (2)	0.01387 (19)	0.00999 (19)	−0.00017 (15)	−0.00324 (15)	−0.00092 (15)
O1	0.0395 (8)	0.0184 (6)	0.0161 (6)	0.0015 (5)	−0.0067 (5)	−0.0043 (5)
O2	0.0314 (7)	0.0191 (6)	0.0138 (6)	0.0002 (5)	−0.0051 (5)	−0.0045 (5)
C1	0.0216 (8)	0.0163 (8)	0.0114 (7)	−0.0019 (6)	−0.0038 (6)	0.0003 (6)
C11	0.0239 (9)	0.0217 (9)	0.0127 (8)	0.0047 (7)	−0.0032 (7)	0.0004 (7)
C12	0.0654 (16)	0.0276 (10)	0.0173 (9)	0.0051 (10)	0.0053 (10)	−0.0048 (8)
C13	0.0699 (18)	0.0432 (14)	0.0179 (10)	0.0117 (12)	0.0129 (10)	−0.0017 (9)
C14	0.0266 (11)	0.0502 (13)	0.0180 (9)	0.0070 (9)	0.0039 (8)	0.0136 (9)
C15	0.0277 (10)	0.0419 (12)	0.0251 (10)	−0.0101 (9)	−0.0077 (8)	0.0100 (9)
C16	0.0255 (10)	0.0329 (10)	0.0158 (8)	−0.0058 (8)	−0.0040 (7)	0.0014 (7)
C21	0.0225 (9)	0.0197 (8)	0.0086 (7)	0.0002 (7)	−0.0031 (6)	0.0008 (6)
C22	0.0282 (10)	0.0248 (9)	0.0167 (8)	0.0012 (7)	−0.0058 (7)	−0.0042 (7)
C23	0.0388 (12)	0.0303 (10)	0.0271 (10)	0.0103 (9)	−0.0074 (9)	−0.0115 (8)
C24	0.0263 (10)	0.0505 (13)	0.0293 (11)	0.0110 (9)	−0.0071 (8)	−0.0097 (10)
C25	0.0243 (10)	0.0501 (13)	0.0266 (10)	−0.0044 (9)	−0.0060 (8)	−0.0101 (9)
C26	0.0280 (10)	0.0272 (9)	0.0185 (9)	−0.0058 (8)	−0.0045 (7)	−0.0033 (7)
C31	0.0192 (8)	0.0227 (9)	0.0129 (8)	−0.0006 (7)	−0.0034 (6)	0.0036 (6)
C32	0.0265 (10)	0.0320 (10)	0.0181 (9)	0.0054 (8)	0.0003 (7)	0.0038 (8)
C33	0.0214 (10)	0.0540 (14)	0.0311 (11)	0.0098 (10)	0.0035 (8)	0.0089 (10)
C34	0.0240 (11)	0.0537 (15)	0.0460 (14)	−0.0121 (10)	−0.0092 (10)	0.0168 (11)
C35	0.0331 (12)	0.0373 (12)	0.0410 (12)	−0.0142 (9)	−0.0172 (10)	0.0080 (10)
C36	0.0249 (10)	0.0258 (9)	0.0235 (9)	−0.0065 (7)	−0.0080 (7)	0.0031 (7)
C41	0.0168 (8)	0.0149 (7)	0.0135 (7)	−0.0023 (6)	−0.0041 (6)	−0.0002 (6)

C42	0.0205 (8)	0.0188 (8)	0.0138 (8)	−0.0018 (7)	−0.0030 (6)	−0.0009 (6)
C43	0.0261 (9)	0.0212 (9)	0.0164 (8)	0.0004 (7)	−0.0074 (7)	0.0023 (7)
C44	0.0262 (9)	0.0181 (8)	0.0259 (9)	0.0053 (7)	−0.0089 (7)	−0.0022 (7)
C45	0.0284 (10)	0.0215 (9)	0.0205 (9)	0.0031 (7)	−0.0050 (7)	−0.0054 (7)
C46	0.0253 (9)	0.0192 (8)	0.0151 (8)	0.0001 (7)	−0.0056 (7)	−0.0021 (6)
P1′	0.0208 (2)	0.0160 (2)	0.0116 (2)	−0.00220 (16)	−0.00512 (16)	−0.00049 (16)
P2′	0.0255 (2)	0.0148 (2)	0.0124 (2)	−0.00359 (17)	−0.00752 (17)	0.00082 (16)
O1′	0.0275 (7)	0.0210 (6)	0.0174 (6)	−0.0059 (5)	−0.0063 (5)	−0.0024 (5)
O2′	0.0437 (8)	0.0223 (7)	0.0178 (6)	−0.0095 (6)	−0.0152 (6)	0.0010 (5)
C1′	0.0240 (9)	0.0154 (8)	0.0119 (7)	−0.0019 (6)	−0.0040 (6)	0.0000 (6)
C11′	0.0168 (8)	0.0231 (9)	0.0113 (7)	−0.0012 (6)	−0.0016 (6)	0.0019 (6)
C12′	0.0218 (9)	0.0271 (9)	0.0171 (8)	−0.0054 (7)	−0.0060 (7)	0.0010 (7)
C13′	0.0200 (9)	0.0387 (11)	0.0166 (9)	−0.0025 (8)	−0.0072 (7)	0.0039 (8)
C14′	0.0230 (9)	0.0287 (10)	0.0167 (8)	0.0050 (7)	−0.0016 (7)	0.0051 (7)
C15′	0.0334 (10)	0.0211 (9)	0.0184 (9)	0.0053 (8)	−0.0039 (7)	0.0001 (7)
C16′	0.0287 (10)	0.0217 (9)	0.0145 (8)	0.0023 (7)	−0.0064 (7)	−0.0006 (7)
C21′	0.0226 (8)	0.0168 (8)	0.0095 (7)	0.0015 (6)	−0.0066 (6)	0.0004 (6)
C22′	0.0248 (9)	0.0189 (8)	0.0127 (8)	−0.0011 (7)	−0.0029 (6)	−0.0034 (6)
C23′	0.0233 (9)	0.0231 (9)	0.0192 (8)	−0.0044 (7)	−0.0038 (7)	−0.0045 (7)
C24′	0.0207 (9)	0.0295 (10)	0.0192 (9)	0.0024 (7)	−0.0029 (7)	−0.0050 (7)
C25′	0.0301 (10)	0.0219 (9)	0.0202 (9)	0.0071 (7)	−0.0082 (7)	−0.0087 (7)
C26′	0.0270 (9)	0.0178 (8)	0.0168 (8)	0.0014 (7)	−0.0115 (7)	−0.0028 (7)
C31′	0.0231 (9)	0.0224 (9)	0.0140 (8)	0.0001 (7)	−0.0078 (7)	0.0048 (7)
C32′	0.0264 (10)	0.0395 (11)	0.0184 (9)	−0.0063 (8)	−0.0097 (7)	0.0060 (8)
C33′	0.0213 (10)	0.0693 (17)	0.0196 (10)	0.0022 (10)	−0.0062 (8)	0.0097 (10)
C34′	0.0383 (13)	0.0550 (15)	0.0204 (10)	0.0204 (11)	−0.0018 (9)	0.0049 (10)
C35′	0.0481 (13)	0.0285 (10)	0.0232 (10)	0.0119 (9)	−0.0008 (9)	0.0017 (8)
C36′	0.0304 (10)	0.0212 (9)	0.0217 (9)	0.0010 (8)	−0.0014 (8)	0.0019 (7)
C41′	0.0238 (9)	0.0144 (8)	0.0133 (8)	0.0003 (6)	−0.0038 (6)	0.0005 (6)
C42′	0.0262 (9)	0.0253 (9)	0.0177 (9)	0.0008 (7)	−0.0079 (7)	0.0014 (7)
C43′	0.0305 (10)	0.0318 (10)	0.0156 (8)	−0.0007 (8)	−0.0038 (7)	0.0077 (7)
C44′	0.0219 (9)	0.0221 (9)	0.0236 (9)	−0.0007 (7)	−0.0003 (7)	0.0051 (7)
C45′	0.0215 (9)	0.0193 (8)	0.0216 (9)	−0.0009 (7)	−0.0033 (7)	−0.0030 (7)
C46′	0.0252 (9)	0.0175 (8)	0.0141 (8)	0.0012 (7)	−0.0039 (7)	−0.0018 (6)
C99	0.046 (3)	0.029 (2)	0.027 (2)	0.0013 (18)	−0.0116 (18)	−0.0075 (17)
Cl1	0.0529 (8)	0.0508 (7)	0.0435 (7)	−0.0234 (6)	0.0085 (6)	−0.0204 (6)
Cl2	0.0578 (8)	0.0452 (7)	0.0319 (6)	−0.0176 (6)	−0.0042 (5)	−0.0015 (5)

Geometric parameters (Å, º)

P1—O1	1.4838 (13)	P1′—C11′	1.8099 (17)
P1—C11	1.8047 (18)	P1′—C1′	1.8146 (17)
P1—C21	1.8070 (18)	P2′—O2′	1.4869 (13)
P1—C1	1.8097 (17)	P2′—C31′	1.7994 (19)
P2—O2	1.4879 (13)	P2′—C41′	1.8003 (18)
P2—C41	1.7966 (17)	P2′—C1′	1.8219 (17)
P2—C31	1.7986 (18)	C1′—H1A′	0.9900
P2—C1	1.8165 (16)	C1′—H1B′	0.9900

C1—H1A	0.9900	C11′—C16′	1.391 (3)
C1—H1B	0.9900	C11′—C12′	1.395 (2)
C11—C12	1.389 (3)	C12′—C13′	1.394 (3)
C11—C16	1.393 (3)	C12′—H12′	0.9500
C12—C13	1.389 (3)	C13′—C14′	1.376 (3)
C12—H12	0.9500	C13′—H13′	0.9500
C13—C14	1.368 (4)	C14′—C15′	1.387 (3)
C13—H13	0.9500	C14′—H14′	0.9500
C14—C15	1.383 (3)	C15′—C16′	1.394 (2)
C14—H14	0.9500	C15′—H15′	0.9500
C15—C16	1.390 (3)	C16′—H16′	0.9500
C15—H15	0.9500	C21′—C26′	1.391 (2)
C16—H16	0.9500	C21′—C22′	1.399 (2)
C21—C26	1.389 (3)	C22′—C23′	1.384 (2)
C21—C22	1.391 (3)	C22′—H22′	0.9500
C22—C23	1.390 (3)	C23′—C24′	1.389 (3)
C22—H22	0.9500	C23′—H23′	0.9500
C23—C24	1.386 (3)	C24′—C25′	1.382 (3)
C23—H23	0.9500	C24′—H24′	0.9500
C24—C25	1.378 (3)	C25′—C26′	1.390 (3)
C24—H24	0.9500	C25′—H25′	0.9500
C25—C26	1.386 (3)	C26′—H26′	0.9500
C25—H25	0.9500	C31′—C36′	1.391 (3)
C26—H26	0.9500	C31′—C32′	1.396 (3)
C31—C32	1.391 (3)	C32′—C33′	1.390 (3)
C31—C36	1.395 (3)	C32′—H32′	0.9500
C32—C33	1.399 (3)	C33′—C34′	1.376 (4)
C32—H32	0.9500	C33′—H33′	0.9500
C33—C34	1.376 (4)	C34′—C35′	1.381 (3)
C33—H33	0.9500	C34′—H34′	0.9500
C34—C35	1.372 (4)	C35′—C36′	1.391 (3)
C34—H34	0.9500	C35′—H35′	0.9500
C35—C36	1.387 (3)	C36′—H36′	0.9500
C35—H35	0.9500	C41′—C46′	1.397 (2)
C36—H36	0.9500	C41′—C42′	1.398 (2)
C41—C46	1.395 (2)	C42′—C43′	1.378 (3)
C41—C42	1.399 (2)	C42′—H42′	0.9500
C42—C43	1.388 (2)	C43′—C44′	1.384 (3)
C42—H42	0.9500	C43′—H43′	0.9500
C43—C44	1.386 (3)	C44′—C45′	1.386 (2)
C43—H43	0.9500	C44′—H44′	0.9500
C44—C45	1.391 (3)	C45′—C46′	1.387 (2)
C44—H44	0.9500	C45′—H45′	0.9500
C45—C46	1.387 (2)	C46′—H46′	0.9500
C45—H45	0.9500	C99—Cl2	1.761 (4)
C46—H46	0.9500	C99—Cl1	1.768 (5)
P1′—O1′	1.4830 (13)	C99—H99A	0.9900
P1′—C21′	1.8029 (18)	C99—H99B	0.9900

O1—P1—C11	112.44 (8)	O1′—P1′—C1′	114.55 (8)
O1—P1—C21	112.49 (8)	C21′—P1′—C1′	106.75 (8)
C11—P1—C21	106.50 (8)	C11′—P1′—C1′	104.24 (8)
O1—P1—C1	114.06 (8)	O2′—P2′—C31′	111.16 (9)
C11—P1—C1	104.25 (8)	O2′—P2′—C41′	111.42 (8)
C21—P1—C1	106.42 (8)	C31′—P2′—C41′	107.84 (8)
O2—P2—C41	112.05 (7)	O2′—P2′—C1′	110.18 (8)
O2—P2—C31	111.39 (8)	C31′—P2′—C1′	108.60 (8)
C41—P2—C31	107.19 (8)	C41′—P2′—C1′	107.51 (8)
O2—P2—C1	110.56 (8)	P1′—C1′—P2′	121.07 (9)
C41—P2—C1	106.38 (8)	P1′—C1′—H1A′	107.1
C31—P2—C1	109.07 (8)	P2′—C1′—H1A′	107.1
P1—C1—P2	119.07 (9)	P1′—C1′—H1B′	107.1
P1—C1—H1A	107.5	P2′—C1′—H1B′	107.1
P2—C1—H1A	107.5	H1A′—C1′—H1B′	106.8
P1—C1—H1B	107.5	C16′—C11′—C12′	119.30 (16)
P2—C1—H1B	107.5	C16′—C11′—P1′	123.58 (13)
H1A—C1—H1B	107.0	C12′—C11′—P1′	117.09 (14)
C12—C11—C16	119.81 (18)	C13′—C12′—C11′	120.12 (18)
C12—C11—P1	117.21 (15)	C13′—C12′—H12′	119.9
C16—C11—P1	122.98 (14)	C11′—C12′—H12′	119.9
C13—C12—C11	119.6 (2)	C14′—C13′—C12′	120.14 (17)
C13—C12—H12	120.2	C14′—C13′—H13′	119.9
C11—C12—H12	120.2	C12′—C13′—H13′	119.9
C14—C13—C12	120.7 (2)	C13′—C14′—C15′	120.32 (17)
C14—C13—H13	119.6	C13′—C14′—H14′	119.8
C12—C13—H13	119.6	C15′—C14′—H14′	119.8
C13—C14—C15	120.12 (19)	C14′—C15′—C16′	119.86 (18)
C13—C14—H14	119.9	C14′—C15′—H15′	120.1
C15—C14—H14	119.9	C16′—C15′—H15′	120.1
C14—C15—C16	120.1 (2)	C11′—C16′—C15′	120.22 (17)
C14—C15—H15	119.9	C11′—C16′—H16′	119.9
C16—C15—H15	119.9	C15′—C16′—H16′	119.9
C15—C16—C11	119.63 (19)	C26′—C21′—C22′	119.66 (16)
C15—C16—H16	120.2	C26′—C21′—P1′	118.67 (13)
C11—C16—H16	120.2	C22′—C21′—P1′	121.60 (13)
C26—C21—C22	119.66 (17)	C23′—C22′—C21′	119.74 (16)
C26—C21—P1	117.38 (14)	C23′—C22′—H22′	120.1
C22—C21—P1	122.91 (14)	C21′—C22′—H22′	120.1
C23—C22—C21	119.53 (18)	C22′—C23′—C24′	120.21 (17)
C23—C22—H22	120.2	C22′—C23′—H23′	119.9
C21—C22—H22	120.2	C24′—C23′—H23′	119.9
C24—C23—C22	120.51 (19)	C25′—C24′—C23′	120.37 (17)
C24—C23—H23	119.7	C25′—C24′—H24′	119.8
C22—C23—H23	119.7	C23′—C24′—H24′	119.8
C25—C24—C23	119.81 (19)	C24′—C25′—C26′	119.74 (17)
C25—C24—H24	120.1	C24′—C25′—H25′	120.1

C23—C24—H24	120.1	C26′—C25′—H25′	120.1
C24—C25—C26	120.1 (2)	C25′—C26′—C21′	120.27 (17)
C24—C25—H25	119.9	C25′—C26′—H26′	119.9
C26—C25—H25	119.9	C21′—C26′—H26′	119.9
C25—C26—C21	120.34 (18)	C36′—C31′—C32′	119.36 (18)
C25—C26—H26	119.8	C36′—C31′—P2′	121.86 (14)
C21—C26—H26	119.8	C32′—C31′—P2′	118.74 (15)
C32—C31—C36	119.62 (18)	C33′—C32′—C31′	119.7 (2)
C32—C31—P2	118.08 (15)	C33′—C32′—H32′	120.1
C36—C31—P2	122.30 (14)	C31′—C32′—H32′	120.1
C31—C32—C33	119.2 (2)	C34′—C33′—C32′	120.5 (2)
C31—C32—H32	120.4	C34′—C33′—H33′	119.7
C33—C32—H32	120.4	C32′—C33′—H33′	119.7
C34—C33—C32	120.4 (2)	C33′—C34′—C35′	120.2 (2)
C34—C33—H33	119.8	C33′—C34′—H34′	119.9
C32—C33—H33	119.8	C35′—C34′—H34′	119.9
C35—C34—C33	120.5 (2)	C34′—C35′—C36′	119.9 (2)
C35—C34—H34	119.7	C34′—C35′—H35′	120.1
C33—C34—H34	119.7	C36′—C35′—H35′	120.1
C34—C35—C36	120.0 (2)	C31′—C36′—C35′	120.3 (2)
C34—C35—H35	120.0	C31′—C36′—H36′	119.9
C36—C35—H35	120.0	C35′—C36′—H36′	119.9
C35—C36—C31	120.2 (2)	C46′—C41′—C42′	119.57 (16)
C35—C36—H36	119.9	C46′—C41′—P2′	123.88 (13)
C31—C36—H36	119.9	C42′—C41′—P2′	116.55 (14)
C46—C41—C42	119.76 (15)	C43′—C42′—C41′	120.33 (18)
C46—C41—P2	122.90 (12)	C43′—C42′—H42′	119.8
C42—C41—P2	117.31 (13)	C41′—C42′—H42′	119.8
C43—C42—C41	119.79 (16)	C42′—C43′—C44′	120.07 (17)
C43—C42—H42	120.1	C42′—C43′—H43′	120.0
C41—C42—H42	120.1	C44′—C43′—H43′	120.0
C44—C43—C42	120.27 (16)	C43′—C44′—C45′	120.10 (17)
C44—C43—H43	119.9	C43′—C44′—H44′	120.0
C42—C43—H43	119.9	C45′—C44′—H44′	120.0
C43—C44—C45	120.08 (16)	C44′—C45′—C46′	120.44 (17)
C43—C44—H44	120.0	C44′—C45′—H45′	119.8
C45—C44—H44	120.0	C46′—C45′—H45′	119.8
C46—C45—C44	120.08 (17)	C45′—C46′—C41′	119.50 (16)
C46—C45—H45	120.0	C45′—C46′—H46′	120.2
C44—C45—H45	120.0	C41′—C46′—H46′	120.2
C45—C46—C41	120.00 (16)	Cl2—C99—Cl1	111.2 (2)
C45—C46—H46	120.0	Cl2—C99—H99A	109.4
C41—C46—H46	120.0	Cl1—C99—H99A	109.4
O1′—P1′—C21′	113.03 (8)	Cl2—C99—H99B	109.4
O1′—P1′—C11′	112.42 (8)	Cl1—C99—H99B	109.4
C21′—P1′—C11′	105.02 (8)	H99A—C99—H99B	108.0
O1—P1—C1—P2	−53.22 (13)	O1′—P1′—C1′—P2′	−58.61 (13)

C11—P1—C1—P2	−176.23 (10)	C21′—P1′—C1′—P2′	67.30 (12)
C21—P1—C1—P2	71.41 (12)	C11′—P1′—C1′—P2′	178.12 (10)
O2—P2—C1—P1	−145.14 (10)	O2′—P2′—C1′—P1′	−150.02 (10)
C41—P2—C1—P1	92.98 (11)	C31′—P2′—C1′—P1′	−28.05 (13)
C31—P2—C1—P1	−22.34 (13)	C41′—P2′—C1′—P1′	88.38 (12)
O1—P1—C11—C12	16.47 (19)	O1′—P1′—C11′—C16′	−161.45 (14)
C21—P1—C11—C12	−107.18 (17)	C21′—P1′—C11′—C16′	75.27 (16)
C1—P1—C11—C12	140.53 (16)	C1′—P1′—C11′—C16′	−36.81 (17)
O1—P1—C11—C16	−163.89 (15)	O1′—P1′—C11′—C12′	20.52 (16)
C21—P1—C11—C16	72.46 (17)	C21′—P1′—C11′—C12′	−102.77 (14)
C1—P1—C11—C16	−39.83 (17)	C1′—P1′—C11′—C12′	145.16 (14)
C16—C11—C12—C13	−0.8 (3)	C16′—C11′—C12′—C13′	1.1 (3)
P1—C11—C12—C13	178.81 (19)	P1′—C11′—C12′—C13′	179.21 (14)
C11—C12—C13—C14	0.6 (4)	C11′—C12′—C13′—C14′	0.2 (3)
C12—C13—C14—C15	0.1 (4)	C12′—C13′—C14′—C15′	−1.0 (3)
C13—C14—C15—C16	−0.6 (3)	C13′—C14′—C15′—C16′	0.5 (3)
C14—C15—C16—C11	0.4 (3)	C12′—C11′—C16′—C15′	−1.6 (3)
C12—C11—C16—C15	0.4 (3)	P1′—C11′—C16′—C15′	−179.61 (14)
P1—C11—C16—C15	−179.27 (15)	C14′—C15′—C16′—C11′	0.9 (3)
O1—P1—C21—C26	−11.70 (16)	O1′—P1′—C21′—C26′	−7.63 (15)
C11—P1—C21—C26	111.91 (14)	C11′—P1′—C21′—C26′	115.27 (13)
C1—P1—C21—C26	−137.30 (13)	C1′—P1′—C21′—C26′	−134.45 (13)
O1—P1—C21—C22	170.77 (13)	O1′—P1′—C21′—C22′	175.42 (13)
C11—P1—C21—C22	−65.61 (16)	C11′—P1′—C21′—C22′	−61.68 (15)
C1—P1—C21—C22	45.18 (16)	C1′—P1′—C21′—C22′	48.60 (15)
C26—C21—C22—C23	−0.2 (3)	C26′—C21′—C22′—C23′	1.1 (2)
P1—C21—C22—C23	177.25 (14)	P1′—C21′—C22′—C23′	178.04 (13)
C21—C22—C23—C24	−0.5 (3)	C21′—C22′—C23′—C24′	−1.3 (3)
C22—C23—C24—C25	0.8 (3)	C22′—C23′—C24′—C25′	0.3 (3)
C23—C24—C25—C26	−0.3 (3)	C23′—C24′—C25′—C26′	0.8 (3)
C24—C25—C26—C21	−0.4 (3)	C24′—C25′—C26′—C21′	−1.0 (3)
C22—C21—C26—C25	0.7 (3)	C22′—C21′—C26′—C25′	0.0 (2)
P1—C21—C26—C25	−176.90 (14)	P1′—C21′—C26′—C25′	−177.02 (13)
O2—P2—C31—C32	28.14 (16)	O2′—P2′—C31′—C36′	−143.00 (14)
C41—P2—C31—C32	151.04 (13)	C41′—P2′—C31′—C36′	−20.59 (17)
C1—P2—C31—C32	−94.17 (15)	C1′—P2′—C31′—C36′	95.63 (15)
O2—P2—C31—C36	−151.37 (14)	O2′—P2′—C31′—C32′	34.79 (16)
C41—P2—C31—C36	−28.47 (16)	C41′—P2′—C31′—C32′	157.19 (14)
C1—P2—C31—C36	86.32 (16)	C1′—P2′—C31′—C32′	−86.58 (15)
C36—C31—C32—C33	−0.3 (3)	C36′—C31′—C32′—C33′	−0.3 (3)
P2—C31—C32—C33	−179.85 (14)	P2′—C31′—C32′—C33′	−178.17 (14)
C31—C32—C33—C34	0.4 (3)	C31′—C32′—C33′—C34′	1.8 (3)
C32—C33—C34—C35	−0.3 (3)	C32′—C33′—C34′—C35′	−2.1 (3)
C33—C34—C35—C36	0.1 (3)	C33′—C34′—C35′—C36′	0.8 (3)
C34—C35—C36—C31	0.1 (3)	C32′—C31′—C36′—C35′	−0.9 (3)
C32—C31—C36—C35	0.1 (3)	P2′—C31′—C36′—C35′	176.87 (14)
P2—C31—C36—C35	179.59 (15)	C34′—C35′—C36′—C31′	0.7 (3)
O2—P2—C41—C46	−151.50 (14)	O2′—P2′—C41′—C46′	−156.35 (14)

C31—P2—C41—C46	86.01 (16)	C31′—P2′—C41′—C46′	81.40 (16)
C1—P2—C41—C46	−30.57 (17)	C1′—P2′—C41′—C46′	−35.54 (17)
O2—P2—C41—C42	26.54 (16)	O2′—P2′—C41′—C42′	23.16 (16)
C31—P2—C41—C42	−95.95 (14)	C31′—P2′—C41′—C42′	−99.09 (15)
C1—P2—C41—C42	147.47 (13)	C1′—P2′—C41′—C42′	143.97 (14)
C46—C41—C42—C43	0.5 (3)	C46′—C41′—C42′—C43′	0.1 (3)
P2—C41—C42—C43	−177.55 (14)	P2′—C41′—C42′—C43′	−179.45 (15)
C41—C42—C43—C44	0.3 (3)	C41′—C42′—C43′—C44′	−0.3 (3)
C42—C43—C44—C45	−0.9 (3)	C42′—C43′—C44′—C45′	0.2 (3)
C43—C44—C45—C46	0.6 (3)	C43′—C44′—C45′—C46′	0.1 (3)
C44—C45—C46—C41	0.3 (3)	C44′—C45′—C46′—C41′	−0.3 (3)
C42—C41—C46—C45	−0.9 (3)	C42′—C41′—C46′—C45′	0.2 (3)
P2—C41—C46—C45	177.10 (14)	P2′—C41′—C46′—C45′	179.71 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1^{i}	0.95	2.56	3.296 (2)	134
C99—H99*A*···O1	0.99	2.43	3.384 (5)	162
C1′—H1*A*′···O2	0.99	2.22	3.211 (2)	176
C16′—H16′···O2	0.95	2.38	3.324 (2)	172
C22′—H22′···O2	0.95	2.31	3.236 (2)	163
C1—H1*A*···O2′	0.99	2.17	3.156 (2)	176
C16—H16···O2′	0.95	2.44	3.390 (2)	173
C22—H22···O2′	0.95	2.38	3.311 (2)	167

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

[2-(Diphenylphosphoroso)ethyl]diphenyl-λ^{5}-phosphanethione (4)

Crystal data

$C_{26}H_{24}OP_2S$
M_r = 446.45
Triclinic, $P\overline{1}$
a = 8.4187 (3) Å
b = 12.0744 (4) Å
c = 12.1438 (5) Å
α = 74.896 (3)°
β = 77.433 (4)°
γ = 72.768 (3)°
V = 1125.02 (8) Å^3

Z = 2
F(000) = 468
D_x = 1.318 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 20835 reflections
θ = 2.8–30.5°
μ = 0.30 mm^{-1}
T = 100 K
Block, colourless
0.2 × 0.1 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2013)
T_{min} = 0.988, T_{max} = 1.000

60880 measured reflections
6684 independent reflections
6008 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 30.8°, θ_{min} = 2.2°
h = −12→11
k = −17→17
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.079$
$S = 1.04$
6684 reflections
271 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 0.5599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.53537 (3)	0.72313 (2)	0.70012 (2)	0.01013 (6)
P2	0.45779 (3)	0.73083 (2)	0.34883 (2)	0.01076 (6)
S1	0.51600 (3)	0.89193 (2)	0.68210 (2)	0.01622 (7)
O1	0.46452 (10)	0.60488 (7)	0.35308 (7)	0.01594 (15)
C1	0.46161 (13)	0.69233 (9)	0.58356 (9)	0.01220 (18)
H1A	0.485659	0.605457	0.591639	0.015*
H1B	0.338258	0.725345	0.589186	0.015*
C2	0.54703 (13)	0.74639 (9)	0.46519 (9)	0.01260 (18)
H2A	0.668814	0.706721	0.456194	0.015*
H2B	0.534187	0.831580	0.460708	0.015*
C11	0.40853 (12)	0.66269 (9)	0.82980 (9)	0.01177 (18)
C12	0.41051 (13)	0.54245 (10)	0.85705 (9)	0.01430 (19)
H12	0.483890	0.490470	0.810208	0.017*
C13	0.30447 (14)	0.49937 (10)	0.95312 (10)	0.0179 (2)
H13	0.304905	0.417842	0.971494	0.022*
C14	0.19808 (15)	0.57476 (12)	1.02225 (10)	0.0213 (2)
H14	0.125609	0.544856	1.087566	0.026*
C15	0.19738 (15)	0.69387 (12)	0.99610 (10)	0.0230 (2)
H15	0.125127	0.745119	1.044032	0.028*
C16	0.30218 (14)	0.73853 (10)	0.89987 (10)	0.0180 (2)
H16	0.301378	0.820104	0.881964	0.022*
C21	0.75163 (12)	0.63604 (9)	0.70267 (8)	0.01128 (18)
C22	0.81413 (13)	0.52977 (10)	0.66373 (9)	0.0153 (2)
H22	0.741714	0.498512	0.637256	0.018*
C23	0.98362 (14)	0.46991 (11)	0.66402 (10)	0.0188 (2)
H23	1.026469	0.398033	0.636989	0.023*
C24	1.09049 (14)	0.51446 (11)	0.70349 (10)	0.0178 (2)
H24	1.205935	0.473604	0.702781	0.021*
C25	1.02728 (14)	0.61906 (10)	0.74396 (10)	0.0169 (2)
H25	1.099250	0.648961	0.772406	0.020*
C26	0.85942 (13)	0.67998 (10)	0.74295 (9)	0.01426 (19)
H26	0.817404	0.751992	0.769783	0.017*
C31	0.57092 (13)	0.79946 (9)	0.21728 (9)	0.01321 (19)
C32	0.52837 (15)	0.79902 (11)	0.11214 (10)	0.0187 (2)

H32	0.439938	0.765272	0.111762	0.022*
C33	0.61469 (16)	0.84759 (12)	0.00858 (10)	0.0232 (2)
H33	0.584980	0.847436	−0.062497	0.028*
C34	0.74481 (16)	0.89653 (11)	0.00888 (10)	0.0236 (2)
H34	0.804408	0.929275	−0.062082	0.028*
C35	0.78766 (15)	0.89765 (11)	0.11226 (11)	0.0217 (2)
H35	0.876257	0.931479	0.112090	0.026*
C36	0.70122 (14)	0.84932 (10)	0.21654 (10)	0.0167 (2)
H36	0.730961	0.850271	0.287342	0.020*
C41	0.24456 (13)	0.82198 (9)	0.36187 (9)	0.01222 (18)
C42	0.20183 (14)	0.94132 (10)	0.30658 (10)	0.0163 (2)
H42	0.285932	0.976697	0.257419	0.020*
C43	0.03608 (15)	1.00851 (11)	0.32345 (11)	0.0208 (2)
H43	0.006928	1.089245	0.284528	0.025*
C44	−0.08672 (14)	0.95792 (11)	0.39697 (11)	0.0208 (2)
H44	−0.199383	1.004272	0.409211	0.025*
C45	−0.04463 (14)	0.83951 (11)	0.45256 (10)	0.0194 (2)
H45	−0.128620	0.805117	0.503229	0.023*
C46	0.11964 (14)	0.77101 (10)	0.43449 (9)	0.0158 (2)
H46	0.147216	0.689561	0.471430	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.00876 (11)	0.01170 (12)	0.01037 (12)	−0.00238 (9)	−0.00256 (9)	−0.00239 (9)
P2	0.01126 (12)	0.01155 (12)	0.00990 (12)	−0.00285 (9)	−0.00256 (9)	−0.00227 (9)
S1	0.01702 (13)	0.01228 (12)	0.02032 (13)	−0.00283 (9)	−0.00600 (10)	−0.00350 (9)
O1	0.0186 (4)	0.0123 (3)	0.0179 (4)	−0.0036 (3)	−0.0039 (3)	−0.0043 (3)
C1	0.0115 (4)	0.0155 (5)	0.0106 (4)	−0.0045 (4)	−0.0030 (3)	−0.0020 (4)
C2	0.0114 (4)	0.0164 (5)	0.0108 (4)	−0.0047 (4)	−0.0022 (3)	−0.0023 (4)
C11	0.0091 (4)	0.0162 (5)	0.0100 (4)	−0.0026 (4)	−0.0027 (3)	−0.0026 (4)
C12	0.0131 (5)	0.0165 (5)	0.0128 (5)	−0.0030 (4)	−0.0024 (4)	−0.0027 (4)
C13	0.0172 (5)	0.0198 (5)	0.0153 (5)	−0.0062 (4)	−0.0025 (4)	0.0006 (4)
C14	0.0165 (5)	0.0301 (6)	0.0136 (5)	−0.0062 (5)	0.0011 (4)	−0.0008 (4)
C15	0.0195 (5)	0.0281 (6)	0.0176 (5)	−0.0018 (5)	0.0040 (4)	−0.0087 (5)
C16	0.0168 (5)	0.0192 (5)	0.0170 (5)	−0.0025 (4)	0.0003 (4)	−0.0068 (4)
C21	0.0093 (4)	0.0138 (4)	0.0102 (4)	−0.0033 (3)	−0.0017 (3)	−0.0009 (3)
C22	0.0127 (5)	0.0182 (5)	0.0166 (5)	−0.0033 (4)	−0.0032 (4)	−0.0062 (4)
C23	0.0141 (5)	0.0204 (5)	0.0214 (5)	−0.0002 (4)	−0.0017 (4)	−0.0094 (4)
C24	0.0097 (4)	0.0225 (5)	0.0189 (5)	−0.0020 (4)	−0.0021 (4)	−0.0028 (4)
C25	0.0121 (5)	0.0209 (5)	0.0190 (5)	−0.0062 (4)	−0.0056 (4)	−0.0011 (4)
C26	0.0134 (5)	0.0148 (5)	0.0157 (5)	−0.0045 (4)	−0.0043 (4)	−0.0024 (4)
C31	0.0134 (5)	0.0130 (4)	0.0114 (4)	−0.0008 (4)	−0.0010 (4)	−0.0032 (4)
C32	0.0203 (5)	0.0215 (5)	0.0143 (5)	−0.0042 (4)	−0.0035 (4)	−0.0043 (4)
C33	0.0257 (6)	0.0283 (6)	0.0120 (5)	−0.0023 (5)	−0.0024 (4)	−0.0036 (4)
C34	0.0231 (6)	0.0246 (6)	0.0151 (5)	−0.0034 (5)	0.0051 (4)	0.0001 (4)
C35	0.0184 (5)	0.0234 (6)	0.0210 (6)	−0.0074 (4)	0.0029 (4)	−0.0030 (4)
C36	0.0159 (5)	0.0186 (5)	0.0148 (5)	−0.0043 (4)	−0.0007 (4)	−0.0038 (4)

C41	0.0116 (4)	0.0147 (5)	0.0116 (4)	−0.0037 (4)	−0.0034 (3)	−0.0030 (4)
C42	0.0152 (5)	0.0156 (5)	0.0176 (5)	−0.0045 (4)	−0.0031 (4)	−0.0013 (4)
C43	0.0186 (5)	0.0164 (5)	0.0261 (6)	−0.0011 (4)	−0.0067 (4)	−0.0032 (4)
C44	0.0124 (5)	0.0267 (6)	0.0237 (6)	−0.0010 (4)	−0.0044 (4)	−0.0093 (5)
C45	0.0134 (5)	0.0290 (6)	0.0172 (5)	−0.0087 (4)	−0.0015 (4)	−0.0041 (4)
C46	0.0155 (5)	0.0189 (5)	0.0142 (5)	−0.0075 (4)	−0.0037 (4)	−0.0005 (4)

Geometric parameters (Å, º)

P1—C11	1.8121 (11)	C23—H23	0.9500
P1—C21	1.8151 (10)	C24—C25	1.3897 (16)
P1—C1	1.8224 (10)	C24—H24	0.9500
P1—S1	1.9531 (4)	C25—C26	1.3875 (15)
P2—O1	1.4925 (8)	C25—H25	0.9500
P2—C41	1.8042 (11)	C26—H26	0.9500
P2—C31	1.8060 (11)	C31—C36	1.3964 (15)
P2—C2	1.8112 (10)	C31—C32	1.4008 (15)
C1—C2	1.5326 (14)	C32—C33	1.3871 (16)
C1—H1A	0.9900	C32—H32	0.9500
C1—H1B	0.9900	C33—C34	1.3913 (19)
C2—H2A	0.9900	C33—H33	0.9500
C2—H2B	0.9900	C34—C35	1.3840 (18)
C11—C16	1.3975 (15)	C34—H34	0.9500
C11—C12	1.3984 (15)	C35—C36	1.3926 (16)
C12—C13	1.3908 (15)	C35—H35	0.9500
C12—H12	0.9500	C36—H36	0.9500
C13—C14	1.3869 (17)	C41—C42	1.3965 (15)
C13—H13	0.9500	C41—C46	1.3998 (14)
C14—C15	1.3881 (18)	C42—C43	1.3924 (16)
C14—H14	0.9500	C42—H42	0.9500
C15—C16	1.3927 (16)	C43—C44	1.3883 (17)
C15—H15	0.9500	C43—H43	0.9500
C16—H16	0.9500	C44—C45	1.3879 (18)
C21—C22	1.3982 (15)	C44—H44	0.9500
C21—C26	1.3992 (14)	C45—C46	1.3891 (16)
C22—C23	1.3957 (15)	C45—H45	0.9500
C22—H22	0.9500	C46—H46	0.9500
C23—C24	1.3919 (16)		

C11—P1—C21	107.88 (5)	C24—C23—C22	120.67 (10)
C11—P1—C1	104.20 (5)	C24—C23—H23	119.7
C21—P1—C1	107.06 (5)	C22—C23—H23	119.7
C11—P1—S1	113.20 (4)	C25—C24—C23	119.59 (10)
C21—P1—S1	111.65 (4)	C25—C24—H24	120.2
C1—P1—S1	112.37 (4)	C23—C24—H24	120.2
O1—P2—C41	111.94 (5)	C26—C25—C24	120.23 (10)
O1—P2—C31	113.36 (5)	C26—C25—H25	119.9
C41—P2—C31	106.91 (5)	C24—C25—H25	119.9

O1—P2—C2	112.56 (5)	C25—C26—C21	120.43 (10)
C41—P2—C2	105.73 (5)	C25—C26—H26	119.8
C31—P2—C2	105.79 (5)	C21—C26—H26	119.8
C2—C1—P1	111.36 (7)	C36—C31—C32	119.22 (10)
C2—C1—H1A	109.4	C36—C31—P2	122.67 (8)
P1—C1—H1A	109.4	C32—C31—P2	118.09 (8)
C2—C1—H1B	109.4	C33—C32—C31	120.35 (11)
P1—C1—H1B	109.4	C33—C32—H32	119.8
H1A—C1—H1B	108.0	C31—C32—H32	119.8
C1—C2—P2	111.71 (7)	C32—C33—C34	119.94 (11)
C1—C2—H2A	109.3	C32—C33—H33	120.0
P2—C2—H2A	109.3	C34—C33—H33	120.0
C1—C2—H2B	109.3	C35—C34—C33	120.19 (11)
P2—C2—H2B	109.3	C35—C34—H34	119.9
H2A—C2—H2B	107.9	C33—C34—H34	119.9
C16—C11—C12	120.01 (10)	C34—C35—C36	120.16 (11)
C16—C11—P1	119.35 (8)	C34—C35—H35	119.9
C12—C11—P1	120.57 (8)	C36—C35—H35	119.9
C13—C12—C11	119.63 (10)	C35—C36—C31	120.15 (11)
C13—C12—H12	120.2	C35—C36—H36	119.9
C11—C12—H12	120.2	C31—C36—H36	119.9
C14—C13—C12	120.37 (11)	C42—C41—C46	119.43 (10)
C14—C13—H13	119.8	C42—C41—P2	122.63 (8)
C12—C13—H13	119.8	C46—C41—P2	117.89 (8)
C13—C14—C15	120.07 (11)	C43—C42—C41	120.06 (10)
C13—C14—H14	120.0	C43—C42—H42	120.0
C15—C14—H14	120.0	C41—C42—H42	120.0
C14—C15—C16	120.27 (11)	C44—C43—C42	120.22 (11)
C14—C15—H15	119.9	C44—C43—H43	119.9
C16—C15—H15	119.9	C42—C43—H43	119.9
C15—C16—C11	119.63 (11)	C45—C44—C43	119.90 (11)
C15—C16—H16	120.2	C45—C44—H44	120.1
C11—C16—H16	120.2	C43—C44—H44	120.0
C22—C21—C26	119.49 (9)	C44—C45—C46	120.35 (11)
C22—C21—P1	122.73 (8)	C44—C45—H45	119.8
C26—C21—P1	117.76 (8)	C46—C45—H45	119.8
C23—C22—C21	119.57 (10)	C45—C46—C41	120.02 (10)
C23—C22—H22	120.2	C45—C46—H46	120.0
C21—C22—H22	120.2	C41—C46—H46	120.0

C11—P1—C1—C2	175.65 (7)	C24—C25—C26—C21	0.84 (17)
C21—P1—C1—C2	−70.22 (8)	C22—C21—C26—C25	0.25 (16)
S1—P1—C1—C2	52.72 (8)	P1—C21—C26—C25	−178.21 (8)
P1—C1—C2—P2	−173.52 (5)	O1—P2—C31—C36	−122.61 (9)
O1—P2—C2—C1	−56.08 (9)	C41—P2—C31—C36	113.56 (9)
C41—P2—C2—C1	66.42 (8)	C2—P2—C31—C36	1.20 (10)
C31—P2—C2—C1	179.61 (7)	O1—P2—C31—C32	55.48 (10)
C21—P1—C11—C16	128.39 (9)	C41—P2—C31—C32	−68.35 (9)

C1—P1—C11—C16	−118.05 (9)	C2—P2—C31—C32	179.29 (9)
S1—P1—C11—C16	4.33 (10)	C36—C31—C32—C33	−0.02 (17)
C21—P1—C11—C12	−54.62 (9)	P2—C31—C32—C33	−178.18 (9)
C1—P1—C11—C12	58.93 (9)	C31—C32—C33—C34	0.32 (18)
S1—P1—C11—C12	−178.69 (7)	C32—C33—C34—C35	−0.45 (19)
C16—C11—C12—C13	0.93 (15)	C33—C34—C35—C36	0.28 (19)
P1—C11—C12—C13	−176.03 (8)	C34—C35—C36—C31	0.02 (18)
C11—C12—C13—C14	−0.50 (16)	C32—C31—C36—C35	−0.15 (16)
C12—C13—C14—C15	−0.26 (18)	P2—C31—C36—C35	177.92 (9)
C13—C14—C15—C16	0.59 (19)	O1—P2—C41—C42	−147.09 (9)
C14—C15—C16—C11	−0.16 (18)	C31—P2—C41—C42	−22.39 (10)
C12—C11—C16—C15	−0.61 (16)	C2—P2—C41—C42	90.01 (10)
P1—C11—C16—C15	176.40 (9)	O1—P2—C41—C46	35.65 (10)
C11—P1—C21—C22	84.45 (10)	C31—P2—C41—C46	160.35 (8)
C1—P1—C21—C22	−27.17 (10)	C2—P2—C41—C46	−87.24 (9)
S1—P1—C21—C22	−150.55 (8)	C46—C41—C42—C43	−0.27 (16)
C11—P1—C21—C26	−97.14 (9)	P2—C41—C42—C43	−177.49 (9)
C1—P1—C21—C26	151.23 (8)	C41—C42—C43—C44	1.26 (17)
S1—P1—C21—C26	27.85 (9)	C42—C43—C44—C45	−0.93 (18)
C26—C21—C22—C23	−0.92 (16)	C43—C44—C45—C46	−0.41 (18)
P1—C21—C22—C23	177.46 (9)	C44—C45—C46—C41	1.40 (17)
C21—C22—C23—C24	0.52 (17)	C42—C41—C46—C45	−1.06 (16)
C22—C23—C24—C25	0.57 (18)	P2—C41—C46—C45	176.29 (8)
C23—C24—C25—C26	−1.24 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O1^{i}	0.99	2.38	3.3622 (13)	170
C12—H12···O1^{i}	0.95	2.44	3.2973 (13)	150
C22—H22···O1^{i}	0.95	2.39	3.2909 (13)	158
C24—H24···O1^{ii}	0.95	2.65	3.5750 (14)	165
C42—H42···S1^{iii}	0.95	2.92	3.5826 (11)	128
C44—H44···S1^{iv}	0.95	2.91	3.7405 (12)	146
C2—H2*B*···S1	0.99	2.93	3.4548 (11)	114
C16—H16···S1	0.95	2.80	3.3190 (12)	115
C26—H26···S1	0.95	2.85	3.3188 (11)	112

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+2, −*z*+1; (iv) −*x*, −*y*+2, −*z*+1.

[2-(Diphenylphosphoroso)ethyl]diphenyl-l^{5}-phosphaneselone (5)

Crystal data

$C_{26}H_{24}OP_2Se$
M_r = 493.35
Triclinic, $P\overline{1}$
a = 8.3800 (4) Å
b = 11.3301 (6) Å
c = 13.5851 (8) Å
α = 66.391 (4)°
β = 75.870 (5)°
γ = 82.410 (4)°
V = 1145.29 (11) Å^3
Z = 2
F(000) = 504
D_x = 1.431 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 13571 reflections
θ = 2.5–30.8°
μ = 1.80 mm^{-1}
T = 100 K
Lath, colourless
0.2 × 0.18 × 0.04 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2013)
T_{min} = 0.950, T_{max} = 1.000
54824 measured reflections
6630 independent reflections
5686 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 30.0°, θ_{min} = 2.5°
h = −11→11
k = −15→15
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.083
S = 1.08
6630 reflections
271 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 0.9965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.94 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
P1	0.76816 (5)	0.75026 (4)	0.19228 (4)	0.00954 (9)
P2	0.57184 (5)	0.35922 (4)	0.36310 (4)	0.00967 (9)
Se1	0.84848 (2)	0.72853 (2)	0.04009 (2)	0.01788 (6)
O1	0.49460 (16)	0.33110 (13)	0.48092 (11)	0.0157 (3)
C1	0.6283 (2)	0.62387 (16)	0.28864 (14)	0.0107 (3)
H1A	0.600636	0.630295	0.361499	0.013*
H1B	0.524898	0.634949	0.262345	0.013*
C2	0.7092 (2)	0.49148 (17)	0.29894 (15)	0.0125 (3)
H2A	0.795860	0.473415	0.341782	0.015*
H2B	0.763872	0.495093	0.224490	0.015*
C11	0.6542 (2)	0.90136 (16)	0.18185 (14)	0.0105 (3)
C12	0.5712 (2)	0.92195 (17)	0.27658 (15)	0.0135 (3)
H12	0.578189	0.858513	0.347097	0.016*
C13	0.4785 (2)	1.03566 (18)	0.26710 (16)	0.0165 (4)
H13	0.421164	1.049493	0.331370	0.020*
C14	0.4691 (2)	1.12950 (18)	0.16377 (17)	0.0182 (4)
H14	0.404788	1.206730	0.157679	0.022*
C15	0.5537 (3)	1.10998 (19)	0.07013 (16)	0.0200 (4)
H15	0.548629	1.174551	−0.000242	0.024*
C16	0.6465 (2)	0.99593 (18)	0.07850 (15)	0.0162 (4)
H16	0.704205	0.982756	0.014012	0.019*

C21	0.9351 (2)	0.73873 (16)	0.26008 (14)	0.0107 (3)
C22	0.9149 (2)	0.68306 (18)	0.37419 (15)	0.0141 (3)
H22	0.811558	0.650678	0.418887	0.017*
C23	1.0470 (2)	0.67514 (19)	0.42236 (16)	0.0166 (4)
H23	1.033871	0.635394	0.499964	0.020*
C24	1.1977 (2)	0.72474 (19)	0.35809 (16)	0.0169 (4)
H24	1.287251	0.718952	0.391489	0.020*
C25	1.2167 (2)	0.7831 (2)	0.24410 (17)	0.0183 (4)
H25	1.318385	0.819613	0.199789	0.022*
C26	1.0867 (2)	0.78796 (19)	0.19538 (16)	0.0157 (4)
H26	1.101261	0.824955	0.117617	0.019*
C31	0.7034 (2)	0.22687 (17)	0.34513 (15)	0.0124 (3)
C32	0.7795 (2)	0.22597 (18)	0.24191 (16)	0.0157 (4)
H32	0.758148	0.294953	0.177361	0.019*
C33	0.8869 (2)	0.1238 (2)	0.23355 (18)	0.0203 (4)
H33	0.938556	0.122967	0.163312	0.024*
C34	0.9182 (3)	0.0231 (2)	0.3281 (2)	0.0231 (4)
H34	0.992515	−0.045875	0.322223	0.028*
C35	0.8423 (3)	0.02272 (19)	0.43045 (19)	0.0235 (4)
H35	0.863528	−0.046900	0.494685	0.028*
C36	0.7346 (2)	0.12430 (18)	0.43981 (16)	0.0179 (4)
H36	0.682358	0.123924	0.510355	0.021*
C41	0.4195 (2)	0.39234 (17)	0.28035 (15)	0.0123 (3)
C42	0.4407 (2)	0.47780 (18)	0.17059 (16)	0.0164 (4)
H42	0.540315	0.522151	0.134808	0.020*
C43	0.3156 (3)	0.4979 (2)	0.11377 (17)	0.0225 (4)
H43	0.329307	0.557233	0.039579	0.027*
C44	0.1706 (3)	0.4313 (2)	0.16513 (19)	0.0238 (4)
H44	0.085895	0.444960	0.125877	0.029*
C45	0.1500 (2)	0.3449 (2)	0.27401 (19)	0.0226 (4)
H45	0.051963	0.298208	0.308825	0.027*
C46	0.2733 (2)	0.32704 (19)	0.33175 (16)	0.0167 (4)
H46	0.257695	0.269952	0.406722	0.020*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0080 (2)	0.0105 (2)	0.0099 (2)	−0.00006 (15)	−0.00226 (15)	−0.00350 (16)
P2	0.0090 (2)	0.01027 (19)	0.0106 (2)	−0.00031 (15)	−0.00231 (16)	−0.00481 (16)
Se1	0.01927 (11)	0.02073 (10)	0.01308 (9)	0.00119 (7)	−0.00260 (7)	−0.00695 (7)
O1	0.0162 (6)	0.0187 (7)	0.0122 (6)	−0.0011 (5)	−0.0016 (5)	−0.0067 (5)
C1	0.0082 (7)	0.0107 (7)	0.0137 (8)	0.0002 (6)	−0.0021 (6)	−0.0056 (6)
C2	0.0092 (8)	0.0120 (8)	0.0177 (8)	0.0009 (6)	−0.0031 (6)	−0.0074 (7)
C11	0.0088 (7)	0.0094 (7)	0.0138 (8)	−0.0005 (6)	−0.0036 (6)	−0.0041 (6)
C12	0.0145 (8)	0.0112 (8)	0.0133 (8)	−0.0022 (6)	−0.0020 (7)	−0.0033 (6)
C13	0.0158 (9)	0.0149 (8)	0.0182 (9)	−0.0008 (7)	−0.0008 (7)	−0.0072 (7)
C14	0.0188 (9)	0.0133 (8)	0.0241 (10)	0.0031 (7)	−0.0081 (8)	−0.0078 (7)
C15	0.0278 (11)	0.0145 (9)	0.0165 (9)	0.0038 (8)	−0.0101 (8)	−0.0029 (7)

C16	0.0194 (9)	0.0145 (8)	0.0143 (8)	0.0013 (7)	−0.0049 (7)	−0.0049 (7)
C21	0.0091 (8)	0.0103 (7)	0.0134 (8)	0.0010 (6)	−0.0037 (6)	−0.0051 (6)
C22	0.0118 (8)	0.0161 (8)	0.0143 (8)	−0.0017 (7)	−0.0030 (7)	−0.0053 (7)
C23	0.0167 (9)	0.0193 (9)	0.0151 (8)	0.0018 (7)	−0.0068 (7)	−0.0066 (7)
C24	0.0106 (8)	0.0215 (9)	0.0232 (10)	0.0049 (7)	−0.0083 (7)	−0.0123 (8)
C25	0.0078 (8)	0.0278 (10)	0.0211 (9)	−0.0025 (7)	−0.0015 (7)	−0.0115 (8)
C26	0.0105 (8)	0.0205 (9)	0.0150 (8)	−0.0013 (7)	−0.0017 (7)	−0.0061 (7)
C31	0.0100 (8)	0.0108 (8)	0.0182 (9)	−0.0008 (6)	−0.0043 (7)	−0.0066 (7)
C32	0.0151 (9)	0.0168 (9)	0.0179 (9)	−0.0013 (7)	−0.0039 (7)	−0.0088 (7)
C33	0.0169 (9)	0.0235 (10)	0.0246 (10)	−0.0015 (8)	−0.0003 (8)	−0.0156 (8)
C34	0.0162 (9)	0.0169 (9)	0.0413 (13)	0.0037 (7)	−0.0084 (9)	−0.0163 (9)
C35	0.0239 (10)	0.0139 (9)	0.0313 (11)	0.0039 (8)	−0.0118 (9)	−0.0052 (8)
C36	0.0203 (9)	0.0145 (9)	0.0188 (9)	0.0002 (7)	−0.0064 (7)	−0.0054 (7)
C41	0.0102 (8)	0.0134 (8)	0.0160 (8)	0.0016 (6)	−0.0037 (6)	−0.0083 (7)
C42	0.0176 (9)	0.0169 (9)	0.0162 (9)	−0.0013 (7)	−0.0048 (7)	−0.0069 (7)
C43	0.0279 (11)	0.0234 (10)	0.0194 (9)	0.0056 (8)	−0.0129 (8)	−0.0088 (8)
C44	0.0171 (10)	0.0337 (11)	0.0313 (11)	0.0098 (8)	−0.0146 (8)	−0.0212 (10)
C45	0.0109 (9)	0.0329 (11)	0.0320 (11)	0.0002 (8)	−0.0052 (8)	−0.0205 (9)
C46	0.0132 (9)	0.0196 (9)	0.0199 (9)	−0.0005 (7)	−0.0028 (7)	−0.0107 (8)

Geometric parameters (Å, º)

P1—C11	1.8173 (18)	C23—H23	0.9500
P1—C21	1.8182 (17)	C24—C25	1.396 (3)
P1—C1	1.8252 (18)	C24—H24	0.9500
P1—Se1	2.1148 (5)	C25—C26	1.391 (3)
P2—O1	1.4908 (14)	C25—H25	0.9500
P2—C31	1.8023 (18)	C26—H26	0.9500
P2—C2	1.8026 (18)	C31—C32	1.395 (3)
P2—C41	1.8084 (18)	C31—C36	1.400 (3)
C1—C2	1.529 (2)	C32—C33	1.394 (3)
C1—H1A	0.9900	C32—H32	0.9500
C1—H1B	0.9900	C33—C34	1.390 (3)
C2—H2A	0.9900	C33—H33	0.9500
C2—H2B	0.9900	C34—C35	1.379 (3)
C11—C16	1.394 (2)	C34—H34	0.9500
C11—C12	1.398 (3)	C35—C36	1.394 (3)
C12—C13	1.390 (3)	C35—H35	0.9500
C12—H12	0.9500	C36—H36	0.9500
C13—C14	1.394 (3)	C41—C46	1.395 (3)
C13—H13	0.9500	C41—C42	1.397 (3)
C14—C15	1.383 (3)	C42—C43	1.392 (3)
C14—H14	0.9500	C42—H42	0.9500
C15—C16	1.395 (3)	C43—C44	1.392 (3)
C15—H15	0.9500	C43—H43	0.9500
C16—H16	0.9500	C44—C45	1.392 (3)
C21—C22	1.395 (2)	C44—H44	0.9500
C21—C26	1.396 (2)	C45—C46	1.392 (3)

C22—C23	1.394 (2)	C45—H45	0.9500
C22—H22	0.9500	C46—H46	0.9500
C23—C24	1.389 (3)		

C11—P1—C21	107.27 (8)	C24—C23—C22	120.69 (17)
C11—P1—C1	105.53 (8)	C24—C23—H23	119.7
C21—P1—C1	105.82 (8)	C22—C23—H23	119.7
C11—P1—Se1	113.50 (6)	C23—C24—C25	119.54 (17)
C21—P1—Se1	113.37 (6)	C23—C24—H24	120.2
C1—P1—Se1	110.77 (6)	C25—C24—H24	120.2
O1—P2—C31	111.97 (8)	C26—C25—C24	119.99 (18)
O1—P2—C2	114.98 (8)	C26—C25—H25	120.0
C31—P2—C2	102.48 (8)	C24—C25—H25	120.0
O1—P2—C41	111.96 (8)	C25—C26—C21	120.38 (17)
C31—P2—C41	107.42 (8)	C25—C26—H26	119.8
C2—P2—C41	107.36 (8)	C21—C26—H26	119.8
C2—C1—P1	109.91 (12)	C32—C31—C36	119.61 (17)
C2—C1—H1A	109.7	C32—C31—P2	122.76 (14)
P1—C1—H1A	109.7	C36—C31—P2	117.57 (14)
C2—C1—H1B	109.7	C33—C32—C31	119.97 (18)
P1—C1—H1B	109.7	C33—C32—H32	120.0
H1A—C1—H1B	108.2	C31—C32—H32	120.0
C1—C2—P2	115.36 (12)	C34—C33—C32	119.91 (19)
C1—C2—H2A	108.4	C34—C33—H33	120.0
P2—C2—H2A	108.4	C32—C33—H33	120.0
C1—C2—H2B	108.4	C35—C34—C33	120.48 (18)
P2—C2—H2B	108.4	C35—C34—H34	119.8
H2A—C2—H2B	107.5	C33—C34—H34	119.8
C16—C11—C12	119.94 (16)	C34—C35—C36	120.11 (19)
C16—C11—P1	119.59 (14)	C34—C35—H35	119.9
C12—C11—P1	120.45 (13)	C36—C35—H35	119.9
C13—C12—C11	119.70 (17)	C35—C36—C31	119.93 (19)
C13—C12—H12	120.2	C35—C36—H36	120.0
C11—C12—H12	120.2	C31—C36—H36	120.0
C12—C13—C14	120.33 (18)	C46—C41—C42	119.53 (17)
C12—C13—H13	119.8	C46—C41—P2	116.43 (14)
C14—C13—H13	119.8	C42—C41—P2	124.04 (14)
C15—C14—C13	119.89 (18)	C43—C42—C41	119.89 (18)
C15—C14—H14	120.1	C43—C42—H42	120.1
C13—C14—H14	120.1	C41—C42—H42	120.1
C14—C15—C16	120.34 (18)	C42—C43—C44	120.33 (19)
C14—C15—H15	119.8	C42—C43—H43	119.8
C16—C15—H15	119.8	C44—C43—H43	119.8
C11—C16—C15	119.78 (18)	C45—C44—C43	119.94 (18)
C11—C16—H16	120.1	C45—C44—H44	120.0
C15—C16—H16	120.1	C43—C44—H44	120.0
C22—C21—C26	119.59 (16)	C44—C45—C46	119.80 (19)
C22—C21—P1	121.85 (13)	C44—C45—H45	120.1

C26—C21—P1	118.57 (13)	C46—C45—H45	120.1
C23—C22—C21	119.76 (17)	C45—C46—C41	120.49 (19)
C23—C22—H22	120.1	C45—C46—H46	119.8
C21—C22—H22	120.1	C41—C46—H46	119.8
C11—P1—C1—C2	177.01 (12)	C24—C25—C26—C21	2.3 (3)
C21—P1—C1—C2	−69.48 (13)	C22—C21—C26—C25	−0.8 (3)
Se1—P1—C1—C2	53.78 (13)	P1—C21—C26—C25	178.84 (15)
P1—C1—C2—P2	−165.25 (9)	O1—P2—C31—C32	174.97 (14)
O1—P2—C2—C1	−65.11 (15)	C2—P2—C31—C32	−61.28 (17)
C31—P2—C2—C1	173.17 (13)	C41—P2—C31—C32	51.65 (17)
C41—P2—C2—C1	60.19 (15)	O1—P2—C31—C36	−7.95 (17)
C21—P1—C11—C16	117.68 (15)	C2—P2—C31—C36	115.80 (15)
C1—P1—C11—C16	−129.82 (15)	C41—P2—C31—C36	−131.26 (15)
Se1—P1—C11—C16	−8.34 (16)	C36—C31—C32—C33	−0.5 (3)
C21—P1—C11—C12	−63.88 (16)	P2—C31—C32—C33	176.53 (14)
C1—P1—C11—C12	48.62 (16)	C31—C32—C33—C34	−0.2 (3)
Se1—P1—C11—C12	170.10 (12)	C32—C33—C34—C35	0.8 (3)
C16—C11—C12—C13	1.4 (3)	C33—C34—C35—C36	−0.7 (3)
P1—C11—C12—C13	−177.06 (14)	C34—C35—C36—C31	0.0 (3)
C11—C12—C13—C14	−0.6 (3)	C32—C31—C36—C35	0.6 (3)
C12—C13—C14—C15	−0.6 (3)	P2—C31—C36—C35	−176.61 (15)
C13—C14—C15—C16	1.0 (3)	O1—P2—C41—C46	−32.57 (17)
C12—C11—C16—C15	−1.0 (3)	C31—P2—C41—C46	90.75 (15)
P1—C11—C16—C15	177.44 (15)	C2—P2—C41—C46	−159.66 (14)
C14—C15—C16—C11	−0.2 (3)	O1—P2—C41—C42	147.38 (15)
C11—P1—C21—C22	88.80 (16)	C31—P2—C41—C42	−89.30 (17)
C1—P1—C21—C22	−23.50 (17)	C2—P2—C41—C42	20.29 (18)
Se1—P1—C21—C22	−145.10 (13)	C46—C41—C42—C43	0.5 (3)
C11—P1—C21—C26	−90.82 (16)	P2—C41—C42—C43	−179.46 (15)
C1—P1—C21—C26	156.88 (14)	C41—C42—C43—C44	−1.2 (3)
Se1—P1—C21—C26	35.29 (16)	C42—C43—C44—C45	0.4 (3)
C26—C21—C22—C23	−1.1 (3)	C43—C44—C45—C46	1.2 (3)
P1—C21—C22—C23	179.30 (14)	C44—C45—C46—C41	−1.9 (3)
C21—C22—C23—C24	1.5 (3)	C42—C41—C46—C45	1.1 (3)
C22—C23—C24—C25	0.0 (3)	P2—C41—C46—C45	−178.99 (15)
C23—C24—C25—C26	−1.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O1^{i}	0.99	2.29	3.269 (2)	171
C12—H12···O1^{i}	0.95	2.46	3.368 (2)	159
C22—H22···O1^{i}	0.95	2.62	3.503 (2)	155
C24—H24···O1^{ii}	0.95	2.67	3.612 (2)	169
C2—H2*B*···Se1	0.99	2.85	3.4929 (18)	124
C16—H16···Se1	0.95	2.89	3.4346 (19)	118

C26—H26···Se1	0.95	3.07	3.5268 (19)	111
C33—H33···Se1^{iii}	0.95	2.88	3.676 (2)	142

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*+1, −*z*.