Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structures of six complexes of phosphane chalcogenides $R^1R^2R^3PE$ (R = *tert*-butyl or isopropyl, E = S or Se) with the metal halides MX_2 (M = Pd or Pt, X = Cl or Br), two halochalcogenylphosphonium derivatives $(^tBu_2{}^iPrPEBr)_2[Pd_2Br_6]$ and one hydrolysis product

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Received 3 January 2025
Accepted 28 January 2025

Edited by C. Schulzke, Universität Greifswald, Germany

Phosphane chalcogenides and their metal complexes, Part 11. Part 10: Upmann *et al.* (2024e). Dedicated to Prof. Dr. Wolf-Walther du Mont on the occasion of his 80th birthday

Keywords: crystal structure; palladium; platinum; phosphane chalcogenides; secondary interactions.

CCDC references: 2420224; 2182097; 2420223; 2182096; 2182118; 2182120; 2182108; 2182092; 2182093

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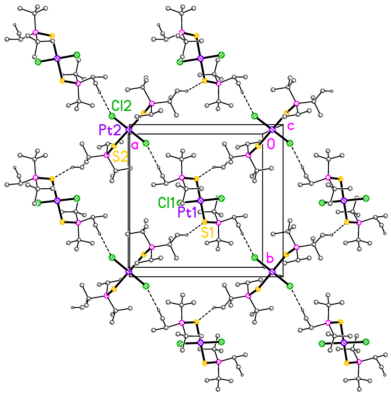
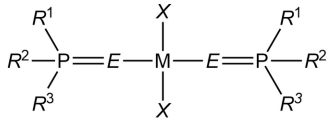


The L_2MX_2 complexes **1**–**5** (**1**: $L = {}^tBu^iPr_2PSe$, M = Pd, X = Cl; **2**: $L = {}^tBu_2{}^iPrPSe$, M = Pd, X = Cl; **3**: $L = {}^tBu_2{}^iPrPSe$, M = Pd, X = Br; **4**: $L = {}^tBu_2{}^iPrPS$, M = Pd, X = Br; **5**: $L = {}^tBu_2{}^iPrPS$, M = Pt, X = Cl) {systematic names: (*tert*-butyldiisopropylphosphine selenide-κSe)dichloridopalladium(II), $[PdCl_2(C_{10}H_{23}PSe)_2]$ (**1**), (di-*tert*-butylisopropylphosphine selenide-κSe)dichloridopalladium(II), $[PdCl_2(C_{11}H_{25}PSe)_2]$ (**2**), dibromido(di-*tert*-butylisopropylphosphine selenide-κSe)palladium(II), $[PdBr_2(C_{11}H_{25}PSe)_2]$ (**3**), dibromido(di-*tert*-butylisopropylphosphine sulfide-κS)palladium(II), $[PdBr_2(C_{11}H_{25}PS)_2]$ (**4**), dichlorido(di-*tert*-butylisopropylphosphine sulfide-κS)palladium(II), $[PdCl_2(C_{11}H_{25}PS)_2]$ (**5**)} all display a *trans* configuration with square-planar geometry at the metal atom. Compounds **2** and **3** are isotypic. The molecules of **1** and **4** display crystallographic inversion symmetry; compound **5** involves two independent molecules, each with inversion symmetry but with differing orientations of the trialkylphosphane groups. Chemically equivalent bond lengths all lie in narrow ranges, whereby the values for palladium and platinum compounds scarcely differ. Compound **6**, $(^tBu^iPr_2PS)_2Pd_2Cl_4$ {systematic name: di-μ-chlorido-bis[(*tert*-butyldiisopropylphosphine sulfide-κS)chloridopalladium(II)], $[PdCl_2(C_{10}H_{23}PS)_2]$}, is dinuclear with a central Pd_2Cl_2 ring, and displays crystallographic inversion symmetry. The bonds to the bridging are longer than those to the terminal chlorine atoms; the Pd—S bond is shorter than the M—S bonds in **4** and **5**, reflecting the weaker *trans* influence of (bridging) chlorine compared to sulfur. Compounds **7** and **8**, $2(^tBu_2{}^iPrPEBr)^+\ [Pd_2Br_6]^{2-}$ with E = S for **7** and Se for **8** {systematic names: (bromosulfanyl)di-*tert*-butylisopropylphosphanium di-μ-bromido-bis[dibromidopalladium(II)], $(C_{11}H_{25}BrPS)_2[Pd_2Br_6]$ (**7**) and (bromoselanyl)di-*tert*-butylisopropylphosphanium di-μ-bromido-bis[dibromidopalladium(II)], $(C_{11}H_{25}BrPS_2)_2[Pd_2Br_6]$, (**8**)}, were obtained by oxidizing the appropriate Pd^{II} precursors with elemental bromine; they are not isotypic. The ions are connected by very short halogen bonds Br···Br. For both compounds, two E···Br contacts further link the cations and anions to form ribbons. Compound **9** {systematic name: bis[dimethyl(sulfanylidene)phosphinito-κSe]bis(hydroxydiisopropylphosphine selenide-κSe)palladium(II), $[Pd(C_6H_{14}OP)_2(C_6H_{15}OP)_2]$, $\{(^iPr_2PSeO)_2H\}_2Pd$, is a hydrolysis product with inversion symmetry and contains an intramolecular P—O···H—O—P group with a disordered hydrogen atom. Compounds **1**–**6** and **9** show few, if any, short intermolecular contacts, although some H···M contacts are observed. A problem with atom-type assignment for structure refinement is discussed.

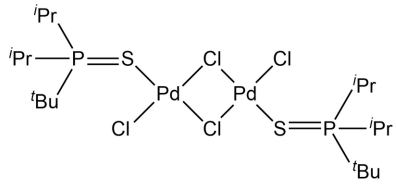
1. Chemical context

We are interested in metal complexes of trialkylphosphane chalcogenide ligands $R^1R^2R^3PE$ (R = *tert*-butyl or isopropyl, E

= S or Se; here we use the general abbreviation *L* for these ligands). In a recent series of papers in this journal (Upmann *et al.*, 2024*a*–*e*; much introductory material is given in the first of these publications) we have reported on the gold(I) complexes *L*Au*X* (*X* = Cl or Br) and their oxidation with elemental bromine or the chlorine equivalent iodobenzene dichloride, $PhICl_2$. The two main series of products were the simple gold(III) complexes *L*AuX_3 and the doubly oxidized halochalcogenylphosphonium derivatives ($R^1R^2R^3PEX$)[AuX_4], corresponding to the addition of two or four halogen atom equivalents, respectively, per metal atom.



1: M = Pd, *X* = Cl, *E* = Se, R^1 = ^{t}Bu, R^2 = R^3 = ^{i}Pr
2: M = Pd, *X* = Cl, *E* = Se, R^1 = R^2 = ^{t}Bu, R^3 = ^{i}Pr
3: M = Pd, *X* = Br, *E* = Se, R^1 = R^2 = ^{t}Bu, R^3 = ^{i}Pr
4: M = Pd, *X* = Br, *E* = S, R^1 = R^2 = ^{t}Bu, R^3 = ^{i}Pr
5: M = Pt, *X* = Cl, *E* = S, R^1 = R^2 = ^{t}Bu, R^3 = ^{i}Pr



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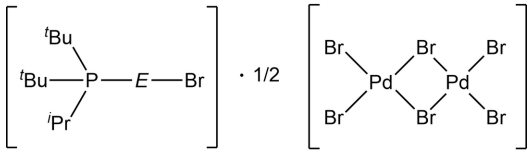
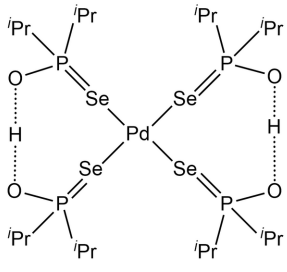


7: *E* = S **8**: *E* = Se



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We decided to extend our studies to palladium(II) or platinum(II) complexes L_2MX_2 (*M* = Pd or Pt) in the hope that these could be similarly oxidized to give *M*(IV) derivatives. Although the M^{II} precursors L_2MX_2 proved to be generally accessible, all attempts to oxidize L_2MCl_2 using iodobenzene dichloride led to immediate decomposition (with formation of a black precipitate), whereas bromine was too weak an oxidizing agent to convert M^{II} to M^{IV}, leading instead

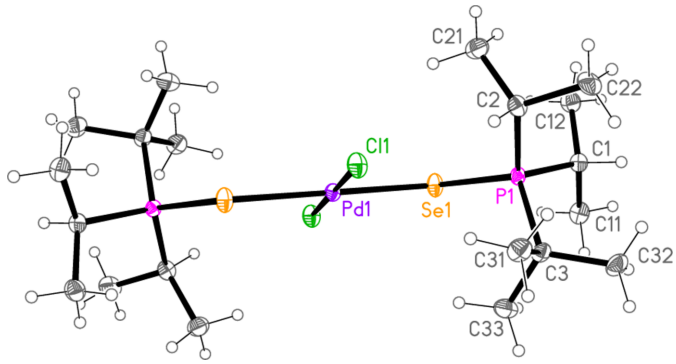

Figure 1
The molecule of compound **1** in the crystal. Only the asymmetric unit is labelled.

to only two isolable complexes (^{t}Bu$_2$^{i}PrP*E*Br)$_2$(Pd$_2$Br$_6$), whereby the ratio of ligands *L* to palladium atoms thus changes from 2 to 1. The investigations were therefore not continued. Here we present the structures of four complexes L_2PdX_2 (**1**–**4**), one complex L_2PtCl_2 (**5**), one dinuclear complex (^{t}Bu^{i}Pr$_2$PS)$_2$Pd$_2$Cl$_4$ (**6**), the two bromochalcogenylphosphonium derivatives (^{t}Bu$_2$^{i}PrP*E*Br)$_2$(Pd$_2$Br$_6$) (**7**, *E* = S; **8**, *E* = Se) and one hydrolysis product {(^{i}Pr$_2$PSeO)$_2$H}$_2$Pd (**9**).

2. Structural commentary

All compounds crystallized solvent-free. Selected molecular dimensions are given in Tables 1–9. The structures are shown in Figs. 1–9, with ellipsoids at the 50% level. The dashed bonds in Figs. 7 and 8 correspond to short interionic contacts that are discussed in *Supramolecular features*. For simplicity we write the P—*E* bonds in the text as single bonds, although they are often written as double bonds P═*E* in older literature (and indeed in the scheme). Primes (') are used to denote generalized or previously defined symmetry operators.

The simple L_2MX_2 complexes **1**–**5** (Figs. 1–5) all show the expected square-planar geometry at the metal atom. Despite the planarity, two of the four Se—Pd—*X* angles in **2** and in **3** differ by *ca*. 10° from the ideal 90°. The largest deviation from planarity for the metal atom and its four immediate neighbours *E* and *X* is observed for **3**, with a mean deviation of

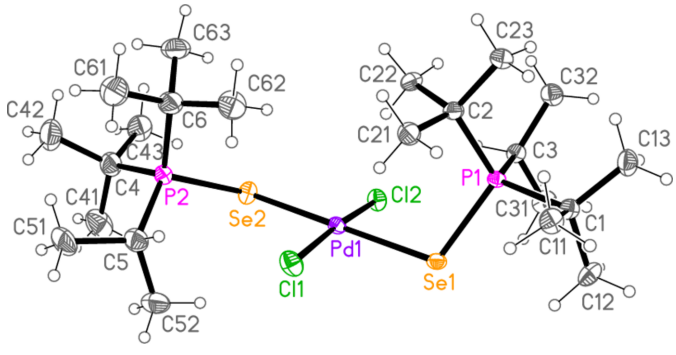

Figure 2
The molecule of compound **2** in the crystal.

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

Pd1—Cl1	2.3099 (8)	Se1—P1	2.1881 (9)
Pd1—Se1	2.4322 (4)		
Cl1—Pd1—Se1^{i}	81.37 (2)	C2—P1—Se1	114.92 (11)
Cl1—Pd1—Se1	98.63 (2)	C1—P1—Se1	102.53 (11)
Se1^{i}—Pd1—Se1	180.0	C3—P1—Se1	114.48 (12)
P1—Se1—Pd1	116.92 (3)		

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

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

Pd1—Cl1	2.3100 (7)	Pd1—Se2	2.4573 (4)
Pd1—Cl2	2.3169 (7)	Se1—P1	2.1870 (8)
Pd1—Se1	2.4411 (4)	Se2—P2	2.1936 (8)
Cl1—Pd1—Cl2	174.61 (3)	P1—Se1—Pd1	107.78 (2)
Cl1—Pd1—Se1	86.97 (2)	P2—Se2—Pd1	117.93 (3)
Cl2—Pd1—Se1	92.46 (2)	C3—P1—Se1	109.18 (10)
Cl1—Pd1—Se2	99.50 (2)	C2—P1—Se1	111.22 (9)
Cl2—Pd1—Se2	80.81 (2)	C1—P1—Se1	103.40 (10)
Se1—Pd1—Se2	172.772 (14)		

0.052 Å. The ligands adopt a *trans* configuration. Compounds **2** and **3** are isotypic. The molecules of **1** and **4** display crystallographic inversion symmetry; compound **5** involves two independent molecules, each with inversion symmetry. With one exception, one of the three torsion angles $E\cdots E—P—Cn$ (omitting the metal atom as the central atom of a linear group), which all differ by ca. 120°, is close to ±180°, and this atom, belonging to a *tert*-butyl group, is given the lowest numbering (C1 or C4). The exception is the second independent molecule of compound **5**, for which all the torsion angles of this type are some 30° greater than for the first molecule; the isopropyl groups are at C3 and C5 and thus formally change places in the rotational sequence (see Fig. 10). Chemically equivalent bond lengths all lie in narrow ranges, whereby the values for palladium (**1**–**4**) and platinum (**5**) compounds scarcely differ. Average bond lengths for the ligands are P—S = 2.028 and P—Se = 2.190 Å; these are closely similar to those in the related gold(I) derivatives LAuX (2.037, 2.194 Å; Upmann *et al.*, 2024*a*) and [L_2Au]-[AuX_4] (2.032, 2.193 Å; Upmann *et al.*, 2024*d*) but slightly shorter than the values of 2.060, 2.218 Å for the gold(III) series LAuX_3 (Upmann *et al.*, 2024*b*), probably reflecting a somewhat greater contribution of the resonance form with a purely single P—E bond in the latter. The bond angles P—E—M were found to vary appreciably for the three series of gold compounds, with ranges of around 5° (and one outlier for ^{t}Bu$_3$PSAuCl$_3$, attributed tentatively to steric effects), although the average values were consistently smaller for Se than for S derivatives. For compounds **1**–**5**, the average

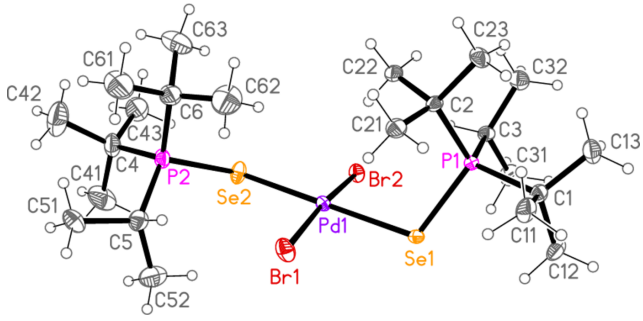

Figure 3
The molecule of compound **3** in the crystal.

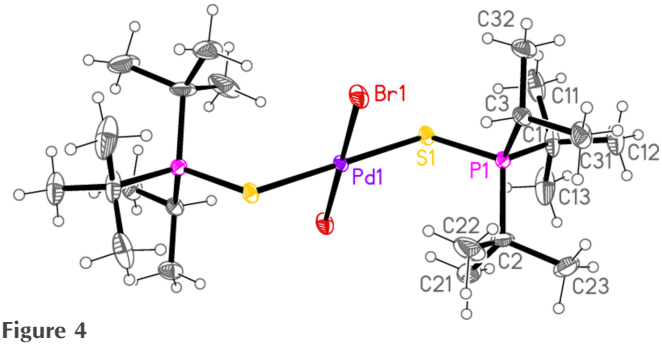

Figure 4
The molecule of compound **4** in the crystal. Only the asymmetric unit is labelled.

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

Pd1—Br1	2.4395 (5)	Se1—P1	2.1890 (11)
Pd1—Se1	2.4416 (5)	Se2—P2	2.1935 (12)
Pd1—Br2	2.4503 (5)	P1—C3	1.847 (4)
Pd1—Se2	2.4628 (5)		
Br1—Pd1—Se1	86.332 (18)	P1—Se1—Pd1	108.83 (3)
Br1—Pd1—Br2	172.66 (2)	P2—Se2—Pd1	119.30 (4)
Se1—Pd1—Br2	92.395 (17)	C3—P1—Se1	109.36 (14)
Br1—Pd1—Se2	100.149 (18)	C2—P1—Se1	111.31 (13)
Se1—Pd1—Se2	172.54 (2)	C1—P1—Se1	103.26 (14)
Br2—Pd1—Se2	80.706 (17)		

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

Pd1—S1	2.3317 (6)	S1—P1	2.0202 (10)
Pd1—Br1	2.4501 (3)		
S1^{i}—Pd1—S1	180.0	P1—S1—Pd1	117.53 (4)
S1—Pd1—Br1	91.133 (17)	C3—P1—S1	111.18 (10)
S1—Pd1—Br1^{i}	88.867 (17)	C2—P1—S1	111.32 (11)
Br1—Pd1—Br1^{i}	180.0	C1—P1—S1	101.95 (11)

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

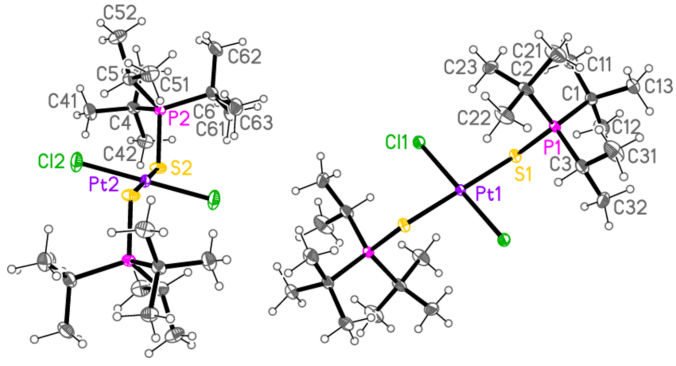

Figure 5
The two independent molecules of compound **5** in the crystal. Each has inversion symmetry; only the asymmetric unit is labelled.

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

Pt1—Cl1	2.3129 (7)	Pt2—S2	2.3278 (7)
Pt1—S1	2.3369 (7)	S1—P1	2.0322 (10)
Pt2—Cl2	2.3070 (7)	S2—P2	2.0323 (10)
Cl1—Pt1—Cl1^{i}	180.0	P1—S1—Pt1	113.49 (3)
Cl1—Pt1—S1	87.04 (2)	P2—S2—Pt2	113.27 (4)
Cl1—Pt1—S1^{i}	92.96 (2)	C3—P1—S1	108.40 (10)
S1—Pt1—S1^{i}	180.0	C2—P1—S1	111.07 (10)
Cl2—Pt2—Cl2^{ii}	180.0	C1—P1—S1	104.54 (10)
Cl2—Pt2—S2^{ii}	89.24 (3)	C5—P2—S2	110.77 (10)
Cl2—Pt2—S2	90.75 (3)	C6—P2—S2	109.37 (10)
S2^{ii}—Pt2—S2	180.0	C4—P2—S2	104.01 (10)

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

Table 6
Selected geometric parameters (Å, °) for **6**.

Pd1—Cl1	2.2799 (6)	Pd1—Cl2^{i}	2.3623 (6)
Pd1—S1	2.2882 (6)	S1—P1	2.0350 (7)
Pd1—Cl2	2.3349 (5)		
Cl1—Pd1—S1	93.95 (2)	Pd1—Cl2—Pd1^{i}	94.46 (2)
Cl1—Pd1—Cl2	175.53 (2)	P1—S1—Pd1	107.34 (3)
S1—Pd1—Cl2	89.38 (2)	C3—P1—S1	110.54 (7)
Cl1—Pd1—Cl2^{i}	91.03 (2)	C2—P1—S1	113.53 (7)
S1—Pd1—Cl2^{i}	174.586 (19)	C1—P1—S1	105.38 (8)
Cl2—Pd1—Cl2^{i}	85.54 (2)		

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

Table 7
Selected geometric parameters (Å, °) for **7**.

Br1—S1	2.2027 (14)	Pd1—Br2	2.4199 (6)
Br1—Br2	3.2387 (7)	Pd1—Br3^{i}	2.4447 (6)
P1—S1	2.0941 (18)	Pd1—Br3	2.4514 (6)
Pd1—Br4	2.4131 (6)		
S1—Br1—Br2	175.04 (4)	Br2—Pd1—Br3^{i}	176.25 (2)
C2—P1—S1	107.50 (17)	Br4—Pd1—Br3	177.07 (2)
C1—P1—S1	100.83 (16)	Br2—Pd1—Br3	91.29 (2)
C3—P1—S1	109.19 (17)	Br3^{i}—Pd1—Br3	85.00 (2)
P1—S1—Br1	103.52 (7)	Pd1—Br2—Br1	71.340 (18)
Br4—Pd1—Br2	91.64 (2)	Pd1^{i}—Br3—Pd1	95.00 (2)
Br4—Pd1—Br3^{i}	92.07 (2)		

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

Table 8
Selected geometric parameters (Å, °) for **8**.

P1—Se1	2.2505 (8)	Pd1—Br3	2.4413 (4)
Se1—Br1	2.3310 (4)	Pd2—Br4	2.4157 (4)
Br1—Br2	3.2510 (5)	Pd2—Br3	2.4562 (4)
Pd1—Br2	2.4218 (4)		
C2—P1—Se1	109.72 (10)	Br3—Pd1—Br3^{i}	85.785 (18)
C3—P1—Se1	109.24 (10)	Br4^{i}—Pd2—Br4	92.26 (2)
C1—P1—Se1	100.47 (10)	Br4—Pd2—Br3^{i}	176.053 (14)
P1—Se1—Br1	100.30 (2)	Br4—Pd2—Br3	91.324 (11)
Se1—Br1—Br2	176.810 (16)	Br3^{i}—Pd2—Br3	85.139 (18)
Br2—Pd1—Br2^{i}	92.495 (19)	Pd1—Br2—Br1	75.133 (10)
Br2—Pd1—Br3	90.936 (11)	Pd1—Br3—Pd2	94.538 (13)
Br2—Pd1—Br3^{i}	175.531 (13)		

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

P—*E*—*M* angles are 114.8° and 114.2° for S and Se, respectively, several degrees higher than those for the gold compounds. Furthermore, the Se values range from 107–119° and differ by over 10° for the two independent P—Se—Pd bond angles of **2** and **3**; perhaps this is in some way connected with the irregular Se—Pd—*X* angles in these compounds, but we can think of no simple reason for this. The *M*—*X* bond lengths are closely similar, with averages of 2.311 Å for *X* = Cl (with no significant difference for the Pd and Pt derivatives) and 2.447 Å for *X* = Br; the same applies to the *E*—*X* bond lengths, with averages of 2.332 Å for *E* = S and 2.447 Å for *E* = Se.

The dinuclear complex **6** (Fig. 6) has the composition $L_2Pd_2Cl_4$ rather than the expected L_2PdCl_2 and displays crystallographic inversion symmetry (with the inversion centre at the centre of the four-membered ring). The bonds to the bridging chlorine atom Cl2 in the central Pd_2Cl_2 ring are, as expected, longer than those to the terminal atom Cl1, with lengths of 2.3623 (6) and 2.3349 (5) Å for the former and 2.2799 (6) Å for the latter. The Pd1—S1 bond is, at 2.2882 (6) Å, shorter than the *M*—S bonds in **4** and **5**, reflecting the weaker *trans* influence of (bridging) chlorine compared to sulfur. The P1—S1—Pd1 angle of 107.34 (3)°, several degrees narrower than for **4** and **5**, underlines the highly variable nature of the P—E—M angles in these compounds. This compound shows the shortest intramolecular H···M contact, namely H22*A*···Pd1 = 2.52 Å.

Compounds **7** and **8**, although differing only in the atom *E*, are not isotypic. The $[Pd_2Br_6]^{2-}$ dianion of compound **7** (Fig. 7) displays crystallographic inversion symmetry, with the

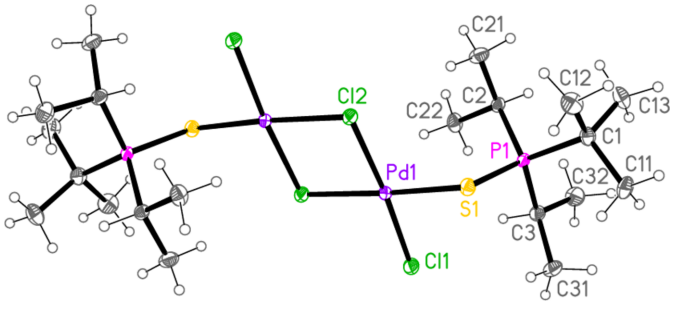

Figure 6
The molecule of compound **6** in the crystal. Only the asymmetric unit is labelled.

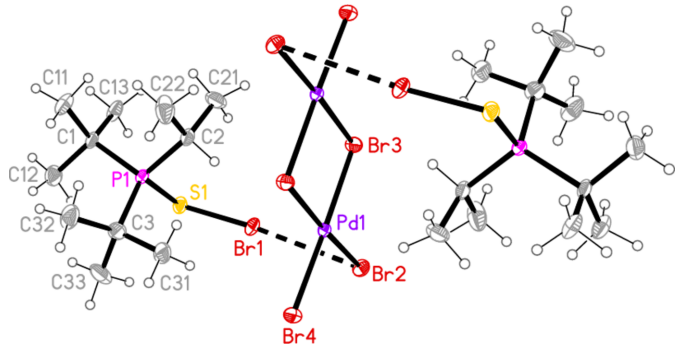

Figure 7
The structure of compound **7** in the crystal. Only the asymmetric unit is labelled. Only the major sites of the disordered methyl groups are shown. The dashed lines indicate short Br···Br contacts.

Table 9
Selected geometric parameters (Å, °) for **9**.

Pd1—Se1	2.4642 (2)	Se2—P2	2.1863 (4)
Pd1—Se2	2.4662 (2)	P1—O1	1.5340 (13)
Se1—P1	2.1894 (5)	P2—O2	1.5287 (12)
Se1—Pd1—Se1^{i}	180.0	Se2^{i}—Pd1—Se2	180.0
Se1—Pd1—Se2^{i}	82.184 (5)	P1—Se1—Pd1	114.028 (13)
Se1—Pd1—Se2	97.817 (5)	P2—Se2—Pd1	105.840 (13)

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

inversion centre at the centre of the four-membered ring, whereas the corresponding dianion of compound **8** (Fig. 8) shows crystallographic twofold symmetry, with both Pd atoms lying on the twofold axis at 0.5, *y*, 0.25. Both anions are essentially planar, with mean deviations of 0.006 and 0.032 Å, respectively. Again, the bonds to the bridging bromine atoms in the central ring are longer than those to the terminal atoms, with average lengths of 2.448 and 2.417 Å, respectively. The halochalcogenyltrialkylphosphonium cations have P—*E* and *E*—Br bond lengths [**7**: P1—S1 = 2.0941 (7), S1—Br1 = 2.2027 (14); **8**: P1—Se1 = 2.2505 (8), Se1—Br1 = 2.3310 (4) Å]

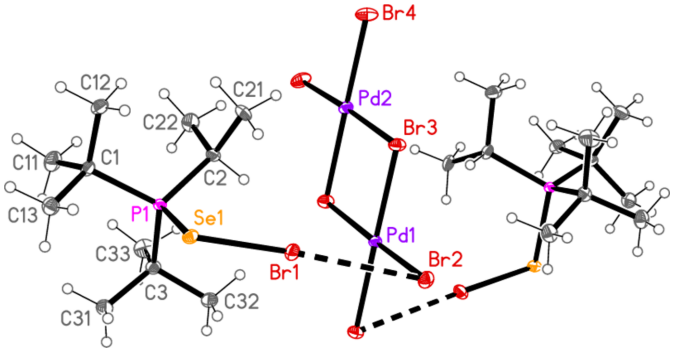

Figure 8
The structure of compound **8** in the crystal. Only the asymmetric unit is labelled. The dashed lines indicate short Br···Br contacts.

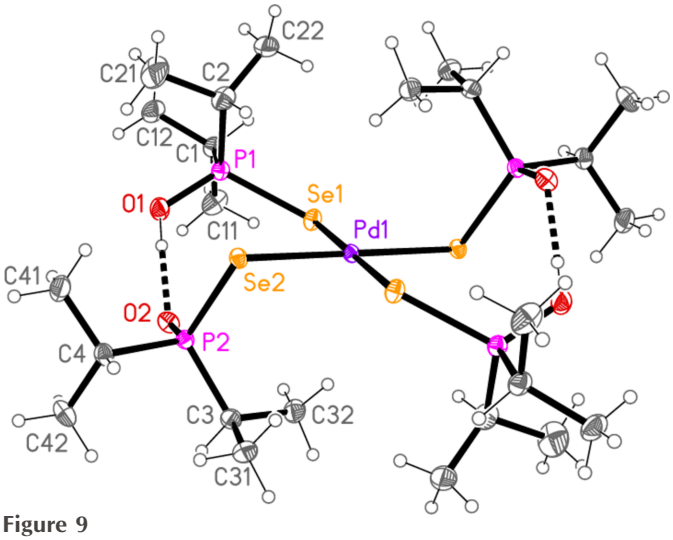

Figure 9
The molecule of compound **9** in the crystal. Only one position of the disordered bridging hydrogen atom is shown. Dashed lines indicate hydrogen bonds.

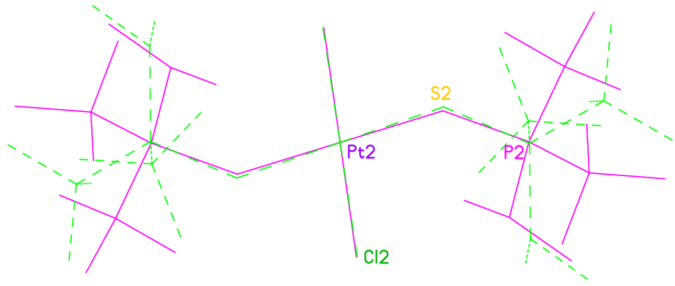

Figure 10
A least-squares fit of the two independent molecules of compound **5**, showing the mutual rotation of the alkyl groups. Molecule 1 is green with dashed bonds; molecule 2 is violet. Fitted atoms are labelled; their symmetry-equivalent atoms were also fitted. Hydrogen atoms are omitted.

that are closely similar to the average values (P—S = 2.095, P—Se = 2.248, S—Br = 2.200, Se—Br = 2.322 Å) for the same cations in the tetrahalogenidoaurate(III) salts, reported in Part 8 (Upmann *et al.*, 2024*c*). The more variable P—*E*—Br angles, which correlated well with increasing steric bulk for the much more numerous gold derivatives, can best be compared to the derivatives with the same (^{t}Bu$_2$^{i}PrP*E*Br)$^{+}$ cations; P1—S1—Br1 in **7** = 103.52 (7), P1—Se1—Br1 in **8** = 100.30 (2)°, compared to their [AuBr$_4$]$^{-}$ salts with 104.48 (6) and 101.91 (6)°, respectively.

The molecule of compound **9** (Fig. 9), which presumably arose under the influence of adventitious water, displays crystallographic inversion symmetry. As observed for **2** and **3** (see above), the two independent P—Se—Pd angles differ appreciably. The 'half' hydrogen atoms at O1 and O2 (see *Refinement*) are disordered; the oxygen atoms show no signs of disorder and the P—O bond lengths are effectively equal. The phosphorus atoms are displaced to opposite sides of the coordination plane (defined by the atoms Pd1, Se1 and Se2), P1 by 0.8076 (5) and P2 by 1.9183 (5) Å. The torsion angle P1—Se1···Se2—P2 (omitting the Pd atom) is 83.83 (2)°. The short intramolecular contact H32*A*···Pd1, 2.69 Å, is noteworthy.

3. Supramolecular features

Tables 10–18 list short contacts that might be interpreted as 'weak' hydrogen bonds; these include some borderline cases that are not further discussed, together with short intramolecular contacts, which may be regarded as a result of the steric crowding, and include contacts of the type H···*M* that are as short as 2.52 Å. In the packing diagrams, the labelling denotes atoms of the asymmetric unit.

In marked contrast to the various series of gold compounds, structures **1**–**6** and **9** show few short intermolecular contacts (*e.g.* H···*X* or *E*···*X*), presumably because of the increased steric effects of having two bulky ligands per molecule rather than one. For compound **1**, for instance, the methine hydrogen atoms, which were generally prolific in forming short contacts in the gold complexes, only form one such contact, which is intramolecular (H2···Cl1= 2.85 Å), and this is also true for

Table 10
Hydrogen-bond geometry (Å, °) for **1**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1	1.00	2.85	3.501 (4)	123
C31—H31*A*···Cl1	0.98	2.70	3.629 (4)	158
C21—H21*B*···Se1	0.98	3.07	3.547 (4)	112
C11—H11*A*···Se1	0.98	2.93	3.419 (4)	112
C12—H12*C*···Se1	0.98	3.05	3.574 (4)	115

Table 11
Hydrogen-bond geometry (Å, °) for **2**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···Se1	0.98	3.08	3.633 (3)	117
C12—H12*C*···Se1	0.98	2.72	3.282 (3)	117
C21—H21*C*···Cl1	0.98	2.77	3.741 (3)	170
C21—H21*C*···Se1	0.98	2.92	3.478 (3)	117
C3—H3···Cl2	1.00	2.77	3.608 (3)	142
C31—H31*B*···Se1	0.98	2.99	3.606 (3)	122
C41—H41*C*···Se2	0.98	3.08	3.632 (4)	117
C43—H43*A*···Se2	0.98	2.67	3.300 (4)	123
C5—H5···Cl1	1.00	2.83	3.484 (4)	124
C51—H51*C*···Cl1^{i}	0.98	2.76	3.642 (3)	150
C52—H52*B*···Cl1	0.98	2.81	3.439 (4)	123
C52—H52*B*···Se2	0.98	2.97	3.507 (4)	116
C62—H62*B*···Se2	0.98	2.91	3.456 (4)	116
C62—H62*C*···Cl1	0.98	2.82	3.552 (4)	132
C63—H63*A*···Se1^{ii}	0.98	3.05	3.948 (4)	153
C63—H63*A*···Pd1^{ii}	0.98	2.87	3.793 (4)	158

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

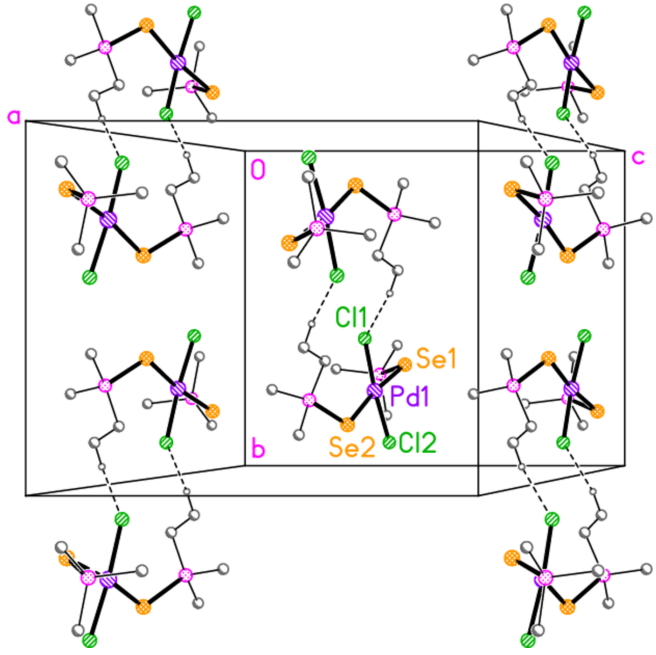

Figure 12
The packing of compound **2**, showing the formation of inversion-symmetric dimers *via* H···Cl contacts (dashed lines). The view direction is perpendicular to the *bc* plane.

several of the other structures. The intramolecular contact H31···Cl1′ is shorter, at 2.70 Å. The program *XP* (Bruker, 1998) found no intermolecular contacts shorter than (sum of atomic radii + 1.7 Å), using the 'PACK 1.7' command, which deliberately ignores H···H contacts; it is probable that the packing is determined by a large number of weak van der Waals contacts such as H···H. Fig. 11 shows a layer of molecules parallel to $(\bar{1}01)$, which can be interpreted as consisting of chains of molecules parallel to $[1\bar{1}1]$.

In compound **2**, the hydrogen bond H51*C*···Cl1$(1-x, 1-y, 1-z)$ links the molecules to form inversion-symmetric dimers (Fig. 12). A further packing motif, the connection of molecules by the three-centre contacts H63*A*···(Se1, Pd1) $(x, \frac{3}{2}-y, -\frac{1}{2}+z)$, leads to ribbons of molecules parallel to the *c* axis (Fig. 13). The concept of hydrogen bonds H···*M*, where *M* is a noble metal, is well-established for *M* = Au (Schmidbaur *et al.*, 2014; Schmidbaur, 2019), but we are not aware of any systematic survey for *M* = Pd or Pt. Compound **3** is isotypic to **2** and the packing motifs are therefore analogous.

For compound **4**, the packing involves no markedly short contacts, but may be interpreted as a layer structure parallel to

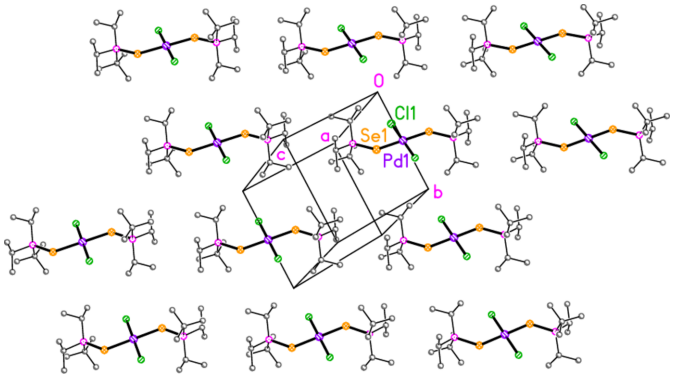

Figure 11
The packing of compound **1**, tentatively interpreted as a layer of molecules parallel to $(\bar{1}01)$. The view direction is perpendicular to the layer. All hydrogen atoms are omitted.

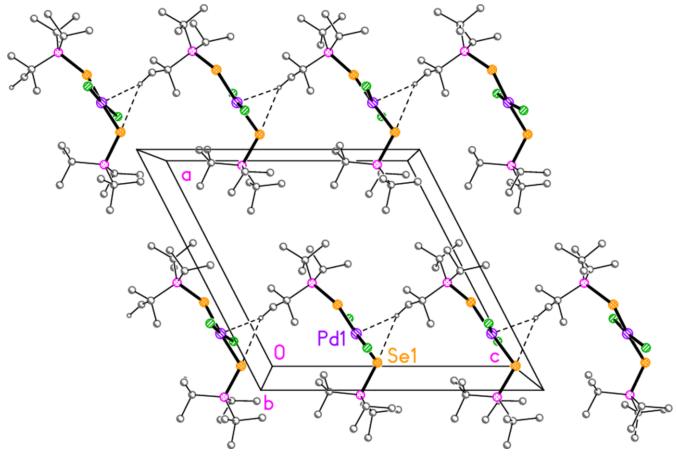

Figure 13
The packing of compound **2**, showing the short three-centre contacts H63*A*···(Pd1, Se1) (dashed lines). The view direction is parallel to the *ac* plane, and the region $y \simeq 0.25$ is depicted. Note that neighbouring molecules are connected by the *c* glide operator and *not* by translation, despite their very similar orientation.

Table 12
Hydrogen-bond geometry (Å, °) for **3**.

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11B···Se1	0.98	3.07	3.630 (4)	117
C12—H12C···Se1	0.98	2.74	3.286 (5)	116
C21—H21C···Br1	0.98	2.88	3.843 (4)	168
C21—H21C···Se1	0.98	2.94	3.491 (4)	117
C22—H22A···Pd1	0.98	2.73	3.641 (4)	155
C3—H3···Br2	1.00	2.84	3.678 (4)	142
C31—H31B···Se1	0.98	3.00	3.612 (5)	122
C41—H41C···Se2	0.98	3.05	3.610 (6)	118
C43—H43A···Se2	0.98	2.70	3.307 (5)	120
C5—H5···Br1	1.00	2.90	3.588 (5)	127
C51—H51C···Br1^{i}	0.98	2.87	3.706 (5)	144
C52—H52B···Br1	0.98	3.01	3.558 (6)	116
C52—H52B···Se2	0.98	2.96	3.545 (6)	120
C62—H62B···Se2	0.98	2.89	3.481 (6)	120
C62—H62C···Br1	0.98	3.03	3.678 (6)	124
C63—H63A···Se1^{ii}	0.98	3.09	4.032 (6)	162
C63—H63A···Pd1^{ii}	0.98	3.03	3.918 (6)	151

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

$(10\bar{1})$ (Fig. 14) in which molecules are connected by the borderline interaction H12A···S1$(\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z)$. The latter contact should perhaps be regarded as an aid to interpretation of the pattern rather than a definite interaction, a caveat that applies to several of the structures described here.

For compound **5**, the contacts H32A···Cl2′$(-1+x, 1+y, z)$ and the borderline H43A···S1$(2-x, 1-y, 1-z)$ combine to form layers parallel to the *ab* plane (Fig. 15).

Compound **6** may be interpreted as a layer structure parallel to the *ac* plane (Fig. 16) involving the borderline contacts H32C···Cl1$(1-x, -y, -z)$ and H22C···Cl2$(-1+x, 1+y, z)$ together with the trio of contacts (H2, H22C, H21A)···Pd1$(-1+x, y, z)$.

In compound **7**, the cation and anion are connected by an extremely short halogen bond (for a review see Metrangolo *et al.*, 2008) Br1···Br2 of 3.2387 (7) Å, with a linear grouping S1—Br1···Br2 = 175.04 (3)° (Fig. 7), which is approximately perpendicular to the coordination plane of the metal, with

Table 13
Hydrogen-bond geometry (Å, °) for **4**.

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···Br1	1.00	2.81	3.638 (3)	140
C11—H11C···S1	0.98	2.63	3.132 (3)	112
C21—H21C···S1	0.98	2.85	3.366 (4)	114
C32—H32C···S1	0.98	3.02	3.567 (3)	117
C22—H22C···Pd1	0.98	2.87	3.778 (4)	154
C12—H12A···S1^{ii}	0.98	2.95	3.443 (3)	112
C21—H21C···Br1^{i}	0.98	2.82	3.782 (4)	168

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

Table 14
Hydrogen-bond geometry (Å, °) for **5**.

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···Cl1^{i}	1.00	2.61	3.382 (3)	134
C5—H6···Cl2	1.00	2.65	3.470 (3)	140
C41—H41A···S2	0.98	3.01	3.541 (3)	115
C42—H42C···S2	0.98	2.64	3.195 (3)	116
C63—H63B···Cl2^{ii}	0.98	2.85	3.666 (3)	141
C63—H63B···S2	0.98	2.85	3.374 (3)	114
C12—H12A···S1	0.98	2.67	3.188 (3)	113
C23—H23B···Cl1	0.98	2.73	3.708 (4)	175
C23—H23B···S1	0.98	2.84	3.370 (3)	115
C32—H32B···S1	0.98	2.87	3.467 (3)	121
C22—H22C···Pt1^{i}	0.98	2.77	3.691 (3)	156
C51—H51C···Pt2^{ii}	0.98	2.64	3.475 (3)	143
C32—H32A···Cl2^{iii}	0.98	2.76	3.716 (3)	165
C43—H43A···S1^{iv}	0.98	2.98	3.779 (3)	139

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

Pd1—Br2···Br1 = 71.34 (2)°. Analogous halogen bonds were common, but not ubiquitous, features of the corresponding Au^{III} derivatives $(R^1R^2R^3PEX)[AuX_4]$ (Upmann *et al.*, 2024*c*).

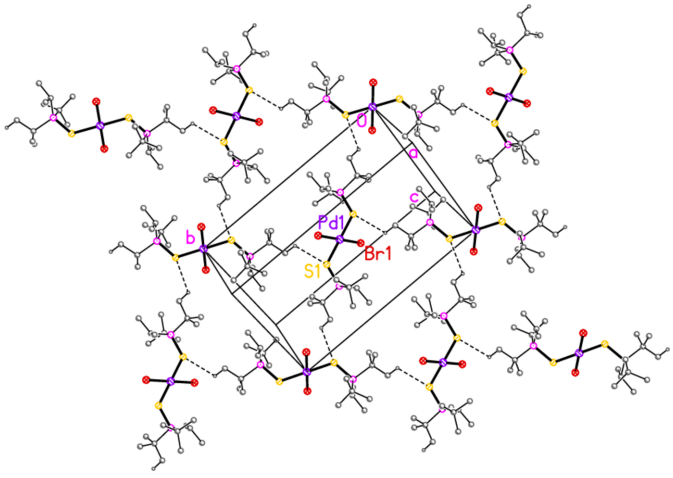

Figure 14
The packing of compound **4**, interpreted as a layer structure parallel to $(10\bar{1})$ involving H···S interactions (dashed lines). The view direction is perpendicular to the layer.

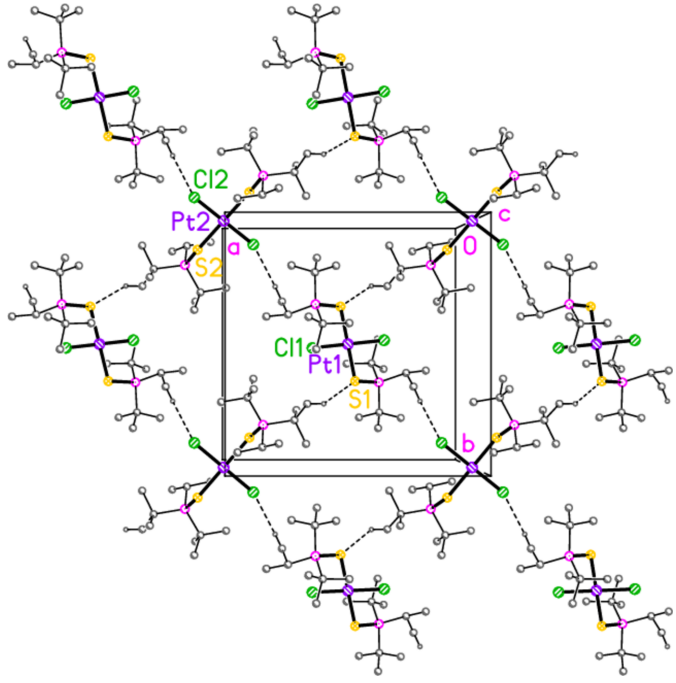

Figure 15
The packing of compound **5**, interpreted as a layer structure parallel to the *ab* plane in the region $z \simeq 0.5$. Dashed lines indicate H···Cl and H···S contacts. The view direction is perpendicular to the layer.

Table 15
Hydrogen-bond geometry (Å, °) for **6**.

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···Cl1	1.00	2.74	3.466 (2)	130
C13—H13*C*···Cl1^{ii}	0.98	2.95	3.893 (2)	162
C31—H31*B*···S1	0.98	2.93	3.523 (2)	120
C32—H32*C*···Cl1^{iii}	0.98	2.89	3.728 (2)	144
C21—H21*C*···Cl2	0.98	2.90	3.844 (2)	161
C22—H22*C*···Cl2^{iv}	0.98	2.99	3.960 (2)	172
C22—H22*A*···Pd1	0.98	2.52	3.383 (2)	147
C2—H2···Pd1^{v}	1.00	3.09	3.688 (2)	120
C22—H22*C*···Pd1^{v}	0.98	3.04	3.740 (2)	129
C21—H21*A*···Pd1^{v}	0.98	3.16	3.784 (2)	123

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

Table 16
Hydrogen-bond geometry (Å, °) for **7**.

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11*C*···Br4^{ii}	0.98	3.04	4.005 (7)	167
C12—H12*C*···S1	0.98	3.03	3.547 (7)	114
C12—H12*C*···Br4^{iii}	0.98	3.10	3.689 (6)	120
C13—H13*B*···S1	0.98	2.70	3.190 (7)	111
C2—H2···Br1	1.00	2.95	3.468 (5)	113
C2—H2···Br3^{i}	1.00	3.12	3.928 (5)	139
C21—H21*B*···Br1	0.98	3.03	3.668 (6)	124
C21—H21*B*···S1	0.98	2.88	3.456 (6)	119
C21—H21*B*···Br3^{iv}	0.98	3.10	3.968 (6)	149
C31—H31*C*···Br1	0.98	2.94	3.732 (11)	139
C33—H33*B*···S1	0.98	2.73	3.298 (10)	117

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x+1, y, z+1$; (iii) $x+1, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $-x+1, -y+1, -z+1$.

The ions are further linked by the contacts S1···Br3 ($1-x$, $1-y$, $1-z$) 3.5463 (14) Å and the rather longer S1···Br4($1+x, y, z$) = 3.8042 (14) Å, with P1—S1···Br′ angles of 116.90 (6) and 173.51 (7)°, respectively, to produce ribbons of residues parallel to the *a* axis (Fig. 17). Despite the differing crystallographic symmetry, the packing of compound **8** is similar to that of **7**, with the halogen bond Br1···Br2 [3.2510 (5) Å, with Se1—Br1···Br2 = 176.81 (2) and Pd1—Br2···Br1 = 75.133 (10)°; see Fig. 8] and the Se···Br contacts Se1···Br4 and Se1···Br3 [3.5655 (5) and 3.6692 (5) Å, respectively, P—Se···Br′ angles of 167.14 (2) and 109.58 (2) Å, respectively, operator $\frac{1}{2}-x, \frac{3}{2}-y, -z$] combining to produce ribbons parallel to [101] (Fig. 18).

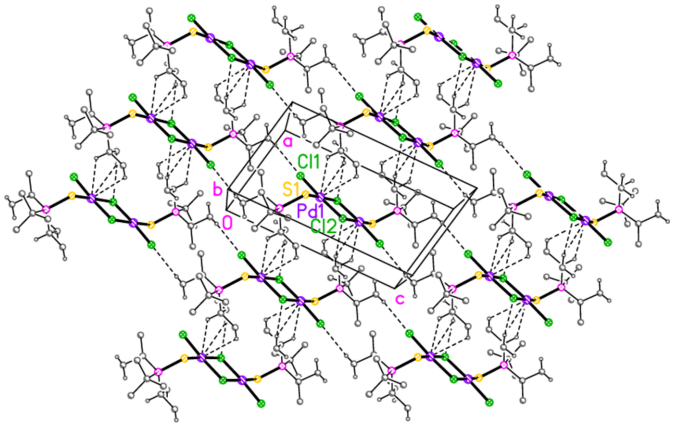

Figure 16
The packing of compound **6**, interpreted as a layer structure parallel to the *ac* plane in the region $y \simeq 0$. Dashed lines indicate H···Cl and H···Pd contacts. The view direction is perpendicular to the layer.

Table 17
Hydrogen-bond geometry (Å, °) for **8**.

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12*C*···Se1	0.98	3.12	3.640 (3)	115
C13—H13*B*···Se1	0.98	2.61	3.181 (3)	117
C21—H21*A*···Br3	0.98	3.14	3.822 (3)	128
C21—H21*B*···Se1	0.98	2.90	3.511 (3)	121
C21—H21*B*···Br1	0.98	2.80	3.574 (3)	137
C31—H31*B*···Se1	0.98	3.16	3.740 (3)	119
C32—H32*A*···Se1	0.98	2.97	3.526 (3)	117
C32—H32*A*···Br1	0.98	2.85	3.452 (3)	120
C12—H12*A*···Br2^{ii}	0.98	2.98	3.877 (3)	152
C12—H12*C*···Br3^{iii}	0.98	3.04	4.009 (3)	170
C2—H2···Br3^{i}	1.00	3.03	3.928 (3)	151
C32—H32*C*···Br2^{iv}	0.98	2.95	3.885 (3)	160

Symmetry codes: (i) $-x+1, y, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, y-\frac{1}{2}, z$; (iii) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z$; (iv) $x, -y+2, z+\frac{1}{2}$.

The packing of compound **9**, like those of **1**–**6**, is almost featureless. The main pattern involves ribbons parallel to the *a* axis *via* the 'weak' hydrogen bond H21*A*···O2′ (Fig. 19).

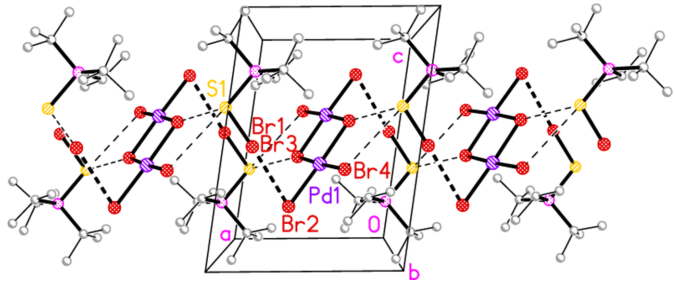

Figure 17
The packing of compound **7**, viewed parallel to the *b* axis in the region $y \simeq 0.5$. All hydrogen atoms are omitted. The dashed lines indicate Br···Br (thick) or Br···S (thin) contacts.

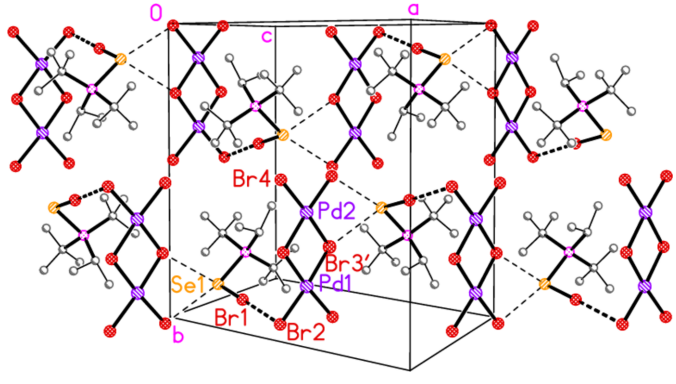

Figure 18
The packing of compound **8**, showing two ribbons of residues running horizontally, The view direction is approximately perpendicular to (10$\bar{1}$), but was rotated slightly about the horizontal axis to minimize overlap of the ribbons. All hydrogen atoms are omitted. The dashed lines indicate Br···Br (thick) or Br···Se (thin) contacts. For clarity, the atom Br3 is represented by its equivalent Br3′.

Table 18
Hydrogen-bond geometry (Å, °) for **9**.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H01···O2	0.78 (2)	1.63 (2)	2.4156 (17)	178 (4)
O2—H02···O1	0.78 (2)	1.64 (2)	2.4156 (17)	172 (5)
C11—H11B···Se1	0.98	2.95	3.4748 (18)	115
C21—H21A···O2^{ii}	0.98	2.52	3.407 (2)	150
C32—H32C···Se2^{iii}	0.98	3.13	3.8999 (17)	136
C41—H41B···Se2	0.98	2.94	3.4825 (18)	116
C42—H42A···Se1^{iv}	0.98	2.98	3.8370 (19)	146
C32—H32A···Pd1	0.98	2.69	3.4897 (18)	139

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

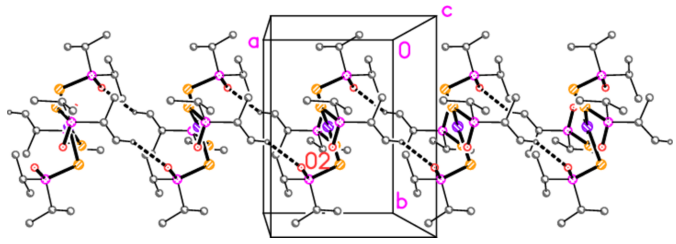

Figure 19
The packing of compound **9**, viewed perpendicular to the *ab* plane in the region $z \simeq 0$. Dashed lines indicate the short contact H21A···O2.

4. Database survey

The searches employed the routine ConQuest (Bruno *et al.*, 2002), part of Version 2024.1.0 of the Cambridge Structural Database (Groom *et al.*, 2016).

A search for compounds containing the moiety $(C_3PE)_2MX_2$, with E = S or Se, X = any halogen, M = Pd or Pt, and coordination number 2 for E, gave seven hits, of which six were unique; all had X = Cl and all but one had M = Pd. Only two involved monodentate phosphane chalcogenide ligands (three were bidentate and one tridentate), namely *trans*-dichloridobis(tri-isobutyl phosphane sulfide)palladium(II) (Richardson, 1985; refcode COSWUC) and *trans*-bis(diethylphenylphosphane sulfide)dichloridopalladium(II) (Satek *et al.*, 1975; EPPTPD). Both have crystallographic inversion symmetry, with Pd—S = 2.334 (1), Pd—Cl = 2.297 (1) and P—S = 2.014 (1) Å for the former and 2.350 (1), 2.302 (1) and 2.013 (2) Å, respectively, for the latter.

A search for the $M_2X_4(E{=}PC_3)_2$ core, as in **6**, gave only one hit, namely $Pd_2Cl_4(S{=}PCy_2Ar)$, where Cy = cyclohexyl and Ar is a 1,4-dimethoxy-3-trimethoxyphenyl-2-naphthyl group (Miroslaw *et al.*, 2023; ROGZEW). The bond lengths of the core are closely similar to those of **6**.

Finally, a search for the $[Pd_2Br_6]^{2-}$ anion gave 22 hits with 24 independent anions. The terminal Pd—Br bond lengths were 2.369–2.438, av. 2.407 (11), and the bridging bonds were as expected significantly longer, at 2.423–2.513, av. 2.453 (13) Å.

5. Synthesis and crystallization

Full details of the preparations (including NMR data) are given in the PhD thesis of Upmann (2015). Here we present three representative syntheses.

Compound **2**: Palladium dichloride (95 mg, 0.5 mmol) was refluxed for 1 h in 25 mL of acetonitrile to give an orange solution. After cooling to r.t., ^{t}Bu$_2^i$PrPSe (286 mg, 1.0 mmol) was added, causing an immediate colour change to reddish-brown. After stirring overnight, the solvent was removed *in vacuo*, the brown residue was washed with *n*-pentane (3 × 3 mL) and diethyl ether (2 × 3 mL) and dried *in vacuo*. The product was recrystallized from dichloromethane/*n*-pentane. ^{31}P NMR (81 MHz, $CDCl_3$): δ = 79.93 (singlet with P—Se satellites, J_{PSe} = 577 Hz).

Compound **6**: Palladium dichloride (454 mg, 2.6 mmol) was refluxed for 1 h in 100 mL of acetonitrile to give an orange solution. After cooling to r.t., ^{t}Bu^{i}Pr$_2$PS (1.057 g, 5.2 mmol) was added, causing an immediate colour change to brown. After stirring overnight, the solvent was removed *in vacuo*, the brown residue was washed with *n*-pentane (2 × 5 mL) and diethyl ether (2 × 5 mL) and dried *in vacuo*. ^{31}P NMR (81 MHz, $CDCl_3$): δ = 81.54 (*s*).

Compound **8**: Compound **3** (103 mg, 0.1 mmol) was dissolved in 5 mL of dichloromethane. The solution was carefully overlayered with *n*-pentane, and two drops of elemental bromine were immediately added. Red crystals of **8** formed overnight. Elemental analysis: calculated: C 19.06, H 3.63. Found: C 18.79, H 3.77%. ^{31}P NMR (81 MHz, $CDCl_3$): δ = 83.77 (*s*). The solubility was too poor to detect P—Se coupling.

6. Refinement

Details of the measurements and refinements are given in Table 19. Structures were refined anisotropically on F^2. Methine hydrogens were included at calculated positions and refined using a riding model with C—H = 1.00 Å and U_{iso}(H) = 1.2 × U_{eq}(C). Methyl groups were refined, using the command 'AFIX 137', as idealized rigid groups allowed to rotate but not tip, with C—H = 0.98 Å, H—C—H = 109.5° and U_{iso}(H) = 1.5 × U_{eq}(C). This procedure, relying as it does on the location of electron-density maxima corresponding to the H-atom sites, is less reliable for heavy-atom structures, so that any postulated hydrogen bonds involving methyl hydrogen atoms (especially for the disordered methyl groups of compound **7**) should be interpreted with caution; however, clear maxima in the electron density were generally found.

Exceptions and special features. For compound **6**, two reflections with $\Delta/\sigma > 6$ were omitted from the refinement; for compound **7**, three reflections with Δ/σ = 7–13 were similarly omitted. For compound **7**, the *tert*-butyl groups at C1 and C3 are disordered over two positions; the occupation factors of the isotropically refined minor components were 0.19 (1) at C1 and 0.20 (3) at C3. Appropriate restraints were applied to improve refinement stability; additionally, the isotropic U value of the minor component of C31 had to be fixed to prevent it becoming negative. The dimensions of disordered groups (especially the minor components) should always be interpreted with caution. Only the major components were

Table 19
Experimental details.

	1	**2**	**3**	**4**	**5**
Crystal data					
Chemical formula	$[PdCl_2(C_{10}H_{23}PSe)_2]$	$[PdCl_2(C_{11}H_{25}PSe)_2]$	$[PdBr_2(C_{11}H_{25}PSe)_2]$	$[PdBr_2(C_{11}H_{25}PS)_2]$	$[PdCl_2(C_{11}H_{25}PS)_2]$
M_r	683.73	711.78	800.70	706.90	706.67
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$
Temperature (K)	100	100	100	100	100
a, b, c (Å)	7.9312 (5), 8.5664 (6), 10.1483 (7)	15.3744 (4), 13.2969 (2), 16.0503 (3)	15.3561 (6), 13.4695 (4), 16.1371 (6)	7.8595 (3), 17.5019 (6), 10.6740 (3)	14.5283 (3), 14.4191 (3), 13.9428 (4)
α, β, γ (°)	88.993 (6), 88.010 (6), 77.063 (6)	90, 117.306 (3), 90	90, 116.558 (5), 90	90, 94.551 (3), 90	90, 94.571 (3), 90
V (Å^3)	671.55 (8)	2915.56 (12)	2985.6 (2)	1463.65 (9)	2911.53 (12)
Z	1	4	4	2	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	3.73	3.44	5.85	3.63	5.27
Crystal size (mm)	0.2 × 0.1 × 0.01	0.20 × 0.10 × 0.02	0.18 × 0.05 × 0.02	0.2 × 0.08 × 0.05	0.2 × 0.1 × 0.07
Data collection					
Diffractometer	Oxford Diffraction Xcalibur, Eos	Oxford Diffraction Xcalibur, Eos	Oxford Diffraction Xcalibur, Eos	Oxford Diffraction Xcalibur, Eos	Oxford Diffraction Xcalibur, Eos
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)
T_{min}, T_{max}	0.841, 1.000	0.713, 1.000	0.419, 0.892	0.865, 1.000	0.650, 1.000
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	20956, 3275, 2604	134870, 7231, 5841	120046, 7402, 5854	45160, 4422, 3832	79369, 8721, 6105
R_{int}	0.072	0.091	0.104	0.050	0.044
θ values (°)	θ_{max} = 28.3, θ_{min} = 2.4	θ_{max} = 28.3, θ_{min} = 2.1	θ_{max} = 28.3, θ_{min} = 2.1	θ_{max} = 30.9, θ_{min} = 2.2	θ_{max} = 30.9, θ_{min} = 2.4
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.667	0.667	0.667	0.721	0.722
Refinement					
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.078, 1.04	0.032, 0.068, 1.04	0.039, 0.083, 1.04	0.039, 0.065, 1.22	0.026, 0.055, 1.04
No. of reflections	3275	7231	7402	4422	8721
No. of parameters	131	278	278	141	281
No. of restraints	0	0	0	0	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.93, −0.98	1.35, −0.78	2.68, −1.32	1.02, −0.70	1.14, −0.97

	6	**7**)	**8**	**9**
Crystal data				
Chemical formula	$[PdCl_2(C_{10}H_{23}PS)_2]$	$(C_{11}H_{25}BrPS)_2[Pd_2Br_6]$	$(C_{11}H_{25}BrPS_2)_2[Pd_2Br_6]$	$[Pd(C_6H_{14}OP)_2(C_6H_{15}OP)_2]$
M_r	767.23	1292.76	1386.56	956.82
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$	Monoclinic, $C2/c$	Monoclinic, $P2_1/n$
Temperature (K)	100	100	100	100
a, b, c (Å)	6.9753 (4), 8.7642 (5), 13.0718 (7)	7.8691 (4), 22.7255 (8), 10.5879 (3)	19.3550 (6), 14.8165 (2), 16.3047 (5)	7.56435 (6), 10.09140 (9), 24.13960 (19)
α, β, γ (°)	88.930 (6), 78.488 (6), 79.804 (7)	90, 98.386 (3), 90	90, 125.957 (5), 90	90, 92.7641 (8), 90
V (Å^3)	770.56 (8)	1873.18 (13)	3784.8 (3)	1840.55 (3)
Z	1	2	4	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	1.76	9.70	11.42	4.66
Crystal size (mm)	0.17 × 0.06 × 0.02	0.2 × 0.1 × 0.01	0.2 × 0.06 × 0.02	0.10 × 0.08 × 0.04
Data collection				
Diffractometer	Oxford Diffraction Xcalibur, Eos	Oxford Diffraction Xcalibur, Eos	Oxford Diffraction Xcalibur, Eos	Oxford Diffraction Xcalibur, Eos
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2012)
T_{min}, T_{max}	0.754, 0.966	0.495, 1.000	0.327, 1.000	0.650, 1.000
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	48215, 4550, 3905	52903, 4622, 3740	51432, 5622, 4433	86102, 5558, 4939
R_{int}	0.052	0.082	0.074	0.047
θ values (°)	θ_{max} = 30.8, θ_{min} = 2.4	θ_{max} = 28.3, θ_{min} = 2.1	θ_{max} = 30.9, θ_{min} = 2.6	θ_{max} = 30.9, θ_{min} = 2.2
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.721	0.667	0.722	0.722

Table 19 (continued)

	6	7)	8	9
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.059, 1.05	0.042, 0.072, 1.11	0.031, 0.057, 1.04	0.022, 0.041, 1.07
No. of reflections	4550	4622	5622	5558
No. of parameters	143	196	172	185
No. of restraints	0	87	0	2
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.29, −0.58	0.71, −0.95	0.97, −1.02	0.47, −0.54

Computer programs: *CrysAlis PRO* (Rigaku OD, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2019/3* (Sheldrick, 2015), *XP* (Bruker, 1998) and *publCIF* (Westrip, 2010).

considered for the discussion and the figures. For compound **9**, the hydrogen atom of the O—H···O moiety was refined on two alternative, half-occupied positions (the occupations were fixed; refining them led to values within 1σ of 0.5). There is no evidence (on the basis of *U* values or residual electron density) that the corresponding oxygen atoms are disordered, and the P—O bond lengths are effectively equal, so that localized P—O and P═O bonds are unlikely.

7. Some comments on the SFAC command

Users of *SHELXL* will be familiar with the SFAC command, which defines the element types (and implicitly the scattering factors) to be used for each atom in the refinement. Thus the command 'SFAC C H P SE BR PD' defines carbon to be element type 1, hydrogen 2, phosphorus 3, *etc*., and these numbers are given explicitly for each atom in the refinement (coming immediately after the atom name), *e.g.*

```
BR1 5 0.275182 0.581682 0.432225 11.00000
0.01922 0.01540 0.03417 − 0.00115 0.01030
0.00275
```

where the atom type 'BR' is the fifth element of the SFAC command (in this section, long lines of computer text have been split into more than one line).

The commonest convention for a standard order of SFAC elements is: C, then H, then other elements in order of atomic number. However, another possibility is: C, then H, then other elements alphabetically. Clearly, the refinement results will be the same in both cases, as long as the SFAC element numbers are given correctly for each atom. The user can change the SFAC order if required, making sure to change the SFAC numbers accordingly; the obvious danger is that, if errors are made, atoms may be refined with the wrong scattering factors. This is usually recognized easily, because the results will be entirely, and often disastrously, wrong (*e.g.* in terms of divergent refinement, high *R* factors, impossibly high or low *U* values, or major features in the residual electron density). A typical example would be an amine complex of gold, for which the third SFAC element would usually be N according to atomic number but Au alphabetically; erroneously using the scattering factors of nitrogen to refine a gold atom, or *vice versa*, would result in nonsense, although the program *SHELXL* would not give an explicit error message.

In this age of automation, SFAC commands are generally set automatically by the program systems. Thus the Rigaku OD *CrysAlis PRO* (Rigaku, OD, 2012) system generates an INS file with an SFAC command corresponding to the 'atomic number' option. However, the *Autochem* option, which solves and refines the structure automatically during and after the data collection, using the *Olex2* platform of *SHELXL* (Dolomanov *et al.*, 2009), employs the 'alphabetic' option (in the version 1.171.43.143a that we currently use). A common first step in refining a structure is thus to extract the atom information from the *Olex2* RES file, edit it into the INS file and *change the SFAC command or the element type numbers appropriately* (if necessary; for many organic structures, *e.g.* those containing only C, H, N, O, P and S, there is no difference between the two options). Even experienced users occasionally forget to do this, but no permanent harm is done if the error is immediately recognized and corrected. *Note added during finalization of the manuscript*: Rigaku OD has informed us that the SFAC commands will be made consistent in the next version of the program, using the IUPAC recommendation for chemical formulae (Connelly & Damhus, 2005), the 'alphabetic' option, which was originally suggested by Hill (1900).

The structures reported here were determined some years ago, in an era where SFAC commands were often set by hand, and were re-refined for publication, using the most recent version of *SHELXL*. When preparing the structure of compound **3** for publication, a curious feature was noticed in the generation of both the figures and the tables; for elements with two letters in the atom symbol, the second letter remained a capital, although both *XP* (Bruker, 1998) and the tables program CIFTAB (as implemented in various *SHELX* platforms) usually convert the second letter automatically to lower case.

checkCIF (Spek, 2020 and references therein) gave no serious alerts of the type A or B; the solution to the conundrum was found in the list of 'less serious' alerts of type G:

```
PLAT017_ALERT_1_G Check Scattering Type
Consistency of BR1 as SE
PLAT017_ALERT_1_G Check Scattering Type
Consistency of BR2 as SE
PLAT017_ALERT_1_G Check Scattering Type
Consistency of SE1 as BR
PLAT017_ALERT_1_G Check Scattering Type
Consistency of SE2 as BR
```

These were generated because of corresponding inconsistencies in the CIF atom sites:

```
  BR1 Se 0.27518 (3) 0.58168 (3) 0.43222 (3)
0.02212 (11) Uani 1 1 d . . . . .
  BR2 Se 0.17265 (3) 0.91841 (3) 0.45811 (3)
0.01627 (10) Uani 1 1 d . . . . .
  SE1 Br 0.08503 (3) 0.67520 (3) 0.45392 (3)
0.01664 (10) Uani 1 1 d  . . . .
  SE2 Br 0.35224 (3) 0.84539 (3) 0.42599 (3)
0.02206 (11) Uani 1 1 d . . . . .
```

The CIF atom list contains the explicit element symbols rather than the SFAC numbers, and it can thus be seen that the bromine atoms had been refined as selenium and *vice versa*, because the wrong SFAC numbers had been used in the refinement. Because the scattering factors of the two atom types, with atomic numbers 34 and 35, are not wildly different, the usual symptoms were not as obvious. The SFAC numbers were corrected and the refinement successfully completed. The *R* value thereby decreased only slightly (by *ca*. 0.2%), as did the residual electron density.

We conclude: (1) Even experienced users can make mistakes in the use of SFAC. (2) These errors are flagged by *checkCIF*, but only as 'ALERT G' (we feel that the severity should be upgraded to 'ALERT B' at least). *Note added during finalization of the manuscript*: The author of *checkCIF*, Professor A. L. Spek, has informed us that this change will soon be implemented. (3) Authors should check not only the serious A and B alerts, but also the 'less serious' alerts C and G; authors (and we include ourselves here!) have a natural tendency to screen the latter lists less conscientiously. (4) For reasons about which we do not speculate, such errors may be indicated by atom symbols with two capital letters when using *XP* or CIFTAB.

Acknowledgements

We thank the Open Access Publication Funds of the Technical University of Braunschweig for financial support. We also express gratitude to Professor A. L. Spek for providing the crystallographic community with the essential checking tool *checkCIF* and for maintaining and improving this program on a regular basis; to Dr Mathias Meyer (Rigaku OD) for helpful discussions; and to both for their patience in dealing with users' comments.

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supporting information

Acta Cryst. (2025). E**81** [https://doi.org/10.1107/S2056989025000805]

Crystal structures of six complexes of phosphane chalcogenides $R^1R^2R^3PE$ (*R* = *tert*-butyl or isopropyl, *E* = S or Se) with the metal halides MX_2 (*M* = Pd or Pt, *X* = Cl or Br), two halochalcogenylphosphonium derivatives $(^tBu_2{}^iPrPEBr)_2[Pd_2Br_6]$ and one hydrolysis product

Daniel Upmann and Peter G. Jones

Computing details

(*tert*-Butyldiisopropylphosphine selenide-κ*Se*)dichloridopalladium(II) (1)

Crystal data

$[PdCl_2(C_{10}H_{23}PSe)_2]$
M_r = 683.73
Triclinic, $P\overline{1}$
a = 7.9312 (5) Å
b = 8.5664 (6) Å
c = 10.1483 (7) Å
α = 88.993 (6)°
β = 88.010 (6)°
γ = 77.063 (6)°
V = 671.55 (8) Å^3
Z = 1
F(000) = 344
D_x = 1.691 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 4517 reflections
θ = 2.4–29.3°
μ = 3.73 mm^{-1}
T = 100 K
Thin plate, dichroic yellow orange
0.2 × 0.1 × 0.01 mm

Data collection

Oxford Diffraction Xcalibur, Eos diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.841, T_{max} = 1.000
20956 measured reflections
3275 independent reflections
2604 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 28.3°, θ_{min} = 2.4°
h = −10→10
k = −11→10
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.078
S = 1.04
3275 reflections
131 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
$w = 1/[\sigma^2(F_o^2) + (0.0307P)^2 + 0.4792P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.93 e Å^{-3}
$\Delta\rho_{min}$ = −0.98 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.000000	0.500000	0.000000	0.01285 (10)
Se1	0.07012 (4)	0.44262 (4)	0.22978 (4)	0.01727 (11)
P1	0.29069 (11)	0.24483 (11)	0.26911 (9)	0.01228 (19)
Cl1	0.17759 (11)	0.27541 (10)	−0.09307 (9)	0.0190 (2)
C1	0.2805 (4)	0.2272 (4)	0.4510 (3)	0.0147 (7)
H1	0.385348	0.145781	0.477693	0.018*
C11	0.2856 (5)	0.3841 (4)	0.5188 (4)	0.0203 (8)
H11A	0.180031	0.464600	0.500005	0.030*
H11B	0.386789	0.422371	0.485510	0.030*
H11C	0.293216	0.365997	0.614217	0.030*
C12	0.1217 (5)	0.1681 (5)	0.5024 (4)	0.0201 (8)
H12A	0.120148	0.164458	0.598972	0.030*
H12B	0.127039	0.060539	0.468581	0.030*
H12C	0.016410	0.241323	0.472417	0.030*
C2	0.2789 (4)	0.0519 (4)	0.1971 (4)	0.0165 (7)
H2	0.327920	0.052510	0.104964	0.020*
C21	0.0930 (5)	0.0285 (5)	0.1851 (4)	0.0223 (8)
H21A	0.096198	−0.072961	0.140989	0.034*
H21B	0.025579	0.117118	0.133495	0.034*
H21C	0.039096	0.026597	0.273322	0.034*
C22	0.3941 (5)	−0.0906 (4)	0.2679 (4)	0.0230 (9)
H22A	0.351376	−0.096425	0.359266	0.034*
H22B	0.513302	−0.076338	0.267243	0.034*
H22C	0.391017	−0.189887	0.222544	0.034*
C3	0.5088 (4)	0.2865 (4)	0.2197 (3)	0.0158 (7)
C31	0.5749 (5)	0.2080 (5)	0.0875 (4)	0.0213 (8)
H31A	0.486334	0.241179	0.021383	0.032*
H31B	0.599828	0.091189	0.097984	0.032*
H31C	0.680742	0.241374	0.058531	0.032*
C32	0.6447 (4)	0.2221 (5)	0.3233 (4)	0.0221 (8)
H32A	0.756830	0.242050	0.293247	0.033*
H32B	0.654933	0.106606	0.335441	0.033*
H32C	0.609030	0.276447	0.407183	0.033*
C33	0.4890 (5)	0.4678 (4)	0.2038 (4)	0.0229 (8)
H33A	0.442647	0.520661	0.286371	0.034*
H33B	0.409494	0.507633	0.132710	0.034*
H33C	0.602237	0.491191	0.181993	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.00951 (19)	0.0118 (2)	0.0158 (2)	0.00070 (14)	−0.00103 (14)	0.00080 (15)
Se1	0.01435 (19)	0.0168 (2)	0.0165 (2)	0.00561 (14)	−0.00110 (14)	−0.00058 (15)
P1	0.0096 (4)	0.0118 (4)	0.0143 (4)	0.0003 (3)	−0.0011 (3)	0.0002 (3)
Cl1	0.0177 (4)	0.0153 (4)	0.0202 (5)	0.0042 (3)	−0.0011 (3)	−0.0009 (3)

C1	0.0154 (17)	0.0149 (18)	0.0130 (17)	−0.0015 (14)	0.0004 (14)	−0.0022 (14)
C11	0.0192 (19)	0.023 (2)	0.0169 (19)	−0.0019 (16)	0.0009 (15)	−0.0029 (15)
C12	0.0211 (19)	0.022 (2)	0.0168 (19)	−0.0048 (16)	0.0016 (15)	0.0004 (15)
C2	0.0150 (17)	0.0149 (18)	0.0175 (19)	0.0008 (14)	0.0014 (14)	−0.0003 (14)
C21	0.022 (2)	0.024 (2)	0.023 (2)	−0.0087 (16)	−0.0015 (16)	−0.0014 (16)
C22	0.026 (2)	0.0118 (18)	0.029 (2)	−0.0001 (15)	−0.0001 (17)	−0.0015 (16)
C3	0.0110 (17)	0.0223 (19)	0.0141 (18)	−0.0045 (14)	0.0016 (13)	−0.0001 (14)
C31	0.0179 (19)	0.027 (2)	0.019 (2)	−0.0064 (16)	0.0032 (15)	−0.0019 (16)
C32	0.0117 (17)	0.030 (2)	0.025 (2)	−0.0054 (16)	−0.0002 (15)	0.0011 (17)
C33	0.024 (2)	0.020 (2)	0.026 (2)	−0.0088 (16)	0.0010 (16)	0.0018 (16)

Geometric parameters (Å, º)

Pd1—Cl1^{i}	2.3099 (8)	C2—H2	1.0000
Pd1—Cl1	2.3099 (8)	C21—H21A	0.9800
Pd1—Se1^{i}	2.4322 (4)	C21—H21B	0.9800
Pd1—Se1	2.4322 (4)	C21—H21C	0.9800
Se1—P1	2.1881 (9)	C22—H22A	0.9800
P1—C2	1.842 (4)	C22—H22B	0.9800
P1—C1	1.850 (3)	C22—H22C	0.9800
P1—C3	1.892 (3)	C3—C33	1.532 (5)
C1—C11	1.530 (5)	C3—C31	1.534 (5)
C1—C12	1.531 (5)	C3—C32	1.538 (5)
C1—H1	1.0000	C31—H31A	0.9800
C11—H11A	0.9800	C31—H31B	0.9800
C11—H11B	0.9800	C31—H31C	0.9800
C11—H11C	0.9800	C32—H32A	0.9800
C12—H12A	0.9800	C32—H32B	0.9800
C12—H12B	0.9800	C32—H32C	0.9800
C12—H12C	0.9800	C33—H33A	0.9800
C2—C22	1.535 (5)	C33—H33B	0.9800
C2—C21	1.541 (5)	C33—H33C	0.9800
Cl1^{i}—Pd1—Cl1	180.0	C2—C21—H21A	109.5
Cl1^{i}—Pd1—Se1^{i}	98.63 (2)	C2—C21—H21B	109.5
Cl1—Pd1—Se1^{i}	81.37 (2)	H21A—C21—H21B	109.5
Cl1^{i}—Pd1—Se1	81.37 (2)	C2—C21—H21C	109.5
Cl1—Pd1—Se1	98.63 (2)	H21A—C21—H21C	109.5
Se1^{i}—Pd1—Se1	180.0	H21B—C21—H21C	109.5
P1—Se1—Pd1	116.92 (3)	C2—C22—H22A	109.5
C2—P1—C1	109.10 (16)	C2—C22—H22B	109.5
C2—P1—C3	107.77 (16)	H22A—C22—H22B	109.5
C1—P1—C3	107.62 (16)	C2—C22—H22C	109.5
C2—P1—Se1	114.92 (11)	H22A—C22—H22C	109.5
C1—P1—Se1	102.53 (11)	H22B—C22—H22C	109.5
C3—P1—Se1	114.48 (12)	C33—C3—C31	108.2 (3)
C11—C1—C12	109.5 (3)	C33—C3—C32	109.4 (3)
C11—C1—P1	112.4 (2)	C31—C3—C32	108.1 (3)

C12—C1—P1	112.7 (2)	C33—C3—P1	108.7 (2)
C11—C1—H1	107.3	C31—C3—P1	110.8 (2)
C12—C1—H1	107.3	C32—C3—P1	111.5 (2)
P1—C1—H1	107.3	C3—C31—H31A	109.5
C1—C11—H11A	109.5	C3—C31—H31B	109.5
C1—C11—H11B	109.5	H31A—C31—H31B	109.5
H11A—C11—H11B	109.5	C3—C31—H31C	109.5
C1—C11—H11C	109.5	H31A—C31—H31C	109.5
H11A—C11—H11C	109.5	H31B—C31—H31C	109.5
H11B—C11—H11C	109.5	C3—C32—H32A	109.5
C1—C12—H12A	109.5	C3—C32—H32B	109.5
C1—C12—H12B	109.5	H32A—C32—H32B	109.5
H12A—C12—H12B	109.5	C3—C32—H32C	109.5
C1—C12—H12C	109.5	H32A—C32—H32C	109.5
H12A—C12—H12C	109.5	H32B—C32—H32C	109.5
H12B—C12—H12C	109.5	C3—C33—H33A	109.5
C22—C2—C21	111.5 (3)	C3—C33—H33B	109.5
C22—C2—P1	112.1 (3)	H33A—C33—H33B	109.5
C21—C2—P1	113.9 (2)	C3—C33—H33C	109.5
C22—C2—H2	106.2	H33A—C33—H33C	109.5
C21—C2—H2	106.2	H33B—C33—H33C	109.5
P1—C2—H2	106.2		

Cl1^{i}—Pd1—Se1—P1	−169.36 (4)	Se1—P1—C2—C22	−158.3 (2)
Cl1—Pd1—Se1—P1	10.64 (4)	C1—P1—C2—C21	84.0 (3)
Pd1—Se1—P1—C2	−55.00 (14)	C3—P1—C2—C21	−159.4 (3)
Pd1—Se1—P1—C1	−173.22 (11)	Se1—P1—C2—C21	−30.5 (3)
Pd1—Se1—P1—C3	70.55 (13)	C2—P1—C3—C33	147.4 (3)
C2—P1—C1—C11	−178.8 (2)	C1—P1—C3—C33	−95.1 (3)
C3—P1—C1—C11	64.6 (3)	Se1—P1—C3—C33	18.2 (3)
Se1—P1—C1—C11	−56.5 (3)	C2—P1—C3—C31	28.6 (3)
C2—P1—C1—C12	−54.5 (3)	C1—P1—C3—C31	146.1 (3)
C3—P1—C1—C12	−171.2 (2)	Se1—P1—C3—C31	−100.6 (2)
Se1—P1—C1—C12	67.8 (2)	C2—P1—C3—C32	−91.9 (3)
C1—P1—C2—C22	−43.8 (3)	C1—P1—C3—C32	25.7 (3)
C3—P1—C2—C22	72.8 (3)	Se1—P1—C3—C32	138.9 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1	1.00	2.85	3.501 (4)	123
C31—H31*A*···Cl1	0.98	2.70	3.629 (4)	158
C21—H21*B*···Se1	0.98	3.07	3.547 (4)	112
C11—H11*A*···Se1	0.98	2.93	3.419 (4)	112
C12—H12*C*···Se1	0.98	3.05	3.574 (4)	115

(Di-*tert*-butylisopropylphosphine selenide-*κSe*)dichloridopalladium(II) (2)

Crystal data

$[PdCl_2(C_{11}H_{25}PSe)_2]$
M_r = 711.78
Monoclinic, $P2_1/c$
a = 15.3744 (4) Å
b = 13.2969 (2) Å
c = 16.0503 (3) Å
β = 117.306 (3)°
V = 2915.56 (12) Å^3
Z = 4
$F(000)$ = 1440
D_x = 1.622 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 15039 reflections
θ = 2.1–30.8°
μ = 3.44 mm^{-1}
T = 100 K
Plate, dichroic red orange
0.20 × 0.10 × 0.02 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 (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.713, T_{max} = 1.000
134870 measured reflections
7231 independent reflections
5841 reflections with $I > 2\sigma(I)$
R_{int} = 0.091
θ_{max} = 28.3°, θ_{min} = 2.1°
h = −20→20
k = −17→17
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.068
S = 1.04
7231 reflections
278 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
$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 4.2293P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.78 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	U_{iso}*/U_{eq}
Pd1	0.21631 (2)	0.75350 (2)	0.43474 (2)	0.01454 (6)
Cl1	0.27119 (6)	0.59216 (5)	0.43300 (6)	0.02429 (16)
Cl2	0.16916 (5)	0.91456 (5)	0.45175 (5)	0.02071 (15)
Se1	0.08372 (2)	0.67680 (2)	0.45622 (2)	0.01735 (7)
Se2	0.34701 (2)	0.84936 (2)	0.42322 (2)	0.02034 (7)
P1	−0.05382 (5)	0.71107 (5)	0.33209 (5)	0.01336 (14)
P2	0.43631 (6)	0.77169 (6)	0.36886 (5)	0.02029 (17)
C1	−0.1433 (2)	0.6199 (2)	0.3408 (2)	0.0179 (6)

C11	−0.1219 (2)	0.5134 (2)	0.3182 (2)	0.0234 (7)
H11A	−0.160709	0.464688	0.332998	0.035*
H11B	−0.052156	0.498536	0.355833	0.035*
H11C	−0.139325	0.508954	0.251481	0.035*
C12	−0.1301 (2)	0.6188 (2)	0.4422 (2)	0.0257 (7)
H12A	−0.176767	0.571550	0.446459	0.039*
H12B	−0.141840	0.686386	0.459183	0.039*
H12C	−0.063198	0.597785	0.485175	0.039*
C13	−0.2503 (2)	0.6480 (2)	0.2754 (2)	0.0259 (7)
H13A	−0.260177	0.651640	0.210622	0.039*
H13B	−0.265127	0.713400	0.293923	0.039*
H13C	−0.293788	0.596718	0.279834	0.039*
C2	−0.0416 (2)	0.6943 (2)	0.2212 (2)	0.0176 (6)
C21	0.0215 (2)	0.6025 (2)	0.2280 (2)	0.0228 (7)
H21A	0.031078	0.598683	0.171795	0.034*
H21B	−0.011447	0.541326	0.232944	0.034*
H21C	0.085160	0.608671	0.283618	0.034*
C22	0.0092 (2)	0.7880 (2)	0.2074 (2)	0.0205 (6)
H22A	0.070588	0.800034	0.264343	0.031*
H22B	−0.033787	0.846468	0.194614	0.031*
H22C	0.023270	0.776957	0.154365	0.031*
C23	−0.1426 (2)	0.6809 (2)	0.1351 (2)	0.0222 (6)
H23A	−0.134537	0.679690	0.077960	0.033*
H23B	−0.185338	0.736972	0.131925	0.033*
H23C	−0.171914	0.617463	0.140730	0.033*
C3	−0.0861 (2)	0.8448 (2)	0.3365 (2)	0.0173 (6)
H3	−0.029117	0.883940	0.339530	0.021*
C31	−0.0912 (3)	0.8716 (2)	0.4271 (2)	0.0264 (7)
H31A	−0.088046	0.944789	0.435029	0.040*
H31B	−0.035950	0.840529	0.480612	0.040*
H31C	−0.152747	0.846544	0.423459	0.040*
C32	−0.1759 (2)	0.8883 (2)	0.2515 (2)	0.0241 (7)
H32A	−0.235194	0.854274	0.244931	0.036*
H32B	−0.168594	0.877950	0.194552	0.036*
H32C	−0.180919	0.960508	0.260924	0.036*
C4	0.5372 (3)	0.8675 (3)	0.3899 (3)	0.0310 (8)
C41	0.6073 (3)	0.8667 (3)	0.4940 (3)	0.0420 (10)
H41A	0.655858	0.920298	0.508305	0.063*
H41B	0.640662	0.801528	0.511255	0.063*
H41C	0.570742	0.877751	0.529645	0.063*
C42	0.5944 (3)	0.8430 (3)	0.3349 (3)	0.0440 (10)
H42A	0.645149	0.894025	0.348349	0.066*
H42B	0.549428	0.842777	0.267542	0.066*
H42C	0.625043	0.776703	0.353712	0.066*
C43	0.4951 (3)	0.9741 (3)	0.3629 (3)	0.0359 (8)
H43A	0.453950	0.989210	0.393325	0.054*
H43B	0.455536	0.978337	0.294732	0.054*
H43C	0.548795	1.022783	0.383513	0.054*

C5	0.4908 (3)	0.6542 (3)	0.4317 (2)	0.0287 (7)
H5	0.438287	0.602317	0.402417	0.034*
C51	0.5791 (3)	0.6127 (3)	0.4210 (3)	0.0400 (9)
H51A	0.636018	0.656369	0.454863	0.060*
H51B	0.563010	0.611008	0.354459	0.060*
H51C	0.594383	0.544578	0.446984	0.060*
C52	0.5152 (3)	0.6556 (3)	0.5356 (2)	0.0375 (9)
H52A	0.535191	0.588088	0.562099	0.056*
H52B	0.457333	0.676426	0.541816	0.056*
H52C	0.568761	0.703035	0.569321	0.056*
C6	0.3601 (3)	0.7436 (3)	0.2415 (2)	0.0294 (7)
C61	0.4087 (3)	0.6709 (3)	0.2026 (3)	0.0425 (10)
H61A	0.419072	0.606082	0.234969	0.064*
H61B	0.471833	0.698348	0.212523	0.064*
H61C	0.366455	0.661231	0.135372	0.064*
C62	0.2628 (3)	0.6983 (3)	0.2264 (3)	0.0362 (9)
H62A	0.222619	0.683661	0.159586	0.054*
H62B	0.228373	0.746102	0.247212	0.054*
H62C	0.275005	0.635872	0.262518	0.054*
C63	0.3368 (3)	0.8428 (3)	0.1851 (2)	0.0372 (9)
H63A	0.289663	0.829621	0.119645	0.056*
H63B	0.397166	0.870319	0.187677	0.056*
H63C	0.308914	0.891380	0.212100	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.01474 (11)	0.01251 (10)	0.01476 (11)	0.00092 (8)	0.00538 (9)	0.00004 (8)
Cl1	0.0229 (4)	0.0153 (3)	0.0320 (4)	0.0039 (3)	0.0103 (3)	0.0004 (3)
Cl2	0.0209 (4)	0.0151 (3)	0.0267 (4)	0.0009 (3)	0.0115 (3)	−0.0021 (3)
Se1	0.01708 (15)	0.01778 (14)	0.01473 (14)	−0.00054 (11)	0.00516 (12)	0.00452 (11)
Se2	0.02171 (16)	0.01730 (15)	0.02555 (17)	−0.00316 (12)	0.01387 (14)	−0.00411 (12)
P1	0.0149 (4)	0.0115 (3)	0.0129 (3)	−0.0003 (3)	0.0056 (3)	0.0005 (3)
P2	0.0180 (4)	0.0242 (4)	0.0187 (4)	0.0048 (3)	0.0084 (3)	0.0027 (3)
C1	0.0193 (15)	0.0139 (14)	0.0196 (15)	−0.0036 (11)	0.0081 (13)	−0.0002 (11)
C11	0.0243 (17)	0.0143 (14)	0.0305 (18)	−0.0033 (12)	0.0118 (15)	0.0015 (12)
C12	0.0312 (18)	0.0243 (16)	0.0279 (17)	−0.0056 (13)	0.0190 (16)	0.0021 (13)
C13	0.0201 (16)	0.0254 (16)	0.0333 (18)	−0.0030 (13)	0.0133 (15)	0.0013 (14)
C2	0.0189 (15)	0.0191 (15)	0.0143 (14)	0.0008 (11)	0.0071 (12)	0.0013 (11)
C21	0.0225 (16)	0.0265 (17)	0.0196 (16)	0.0024 (13)	0.0099 (14)	−0.0026 (13)
C22	0.0240 (16)	0.0221 (15)	0.0169 (15)	−0.0010 (12)	0.0106 (13)	0.0020 (12)
C23	0.0211 (16)	0.0264 (16)	0.0169 (15)	0.0001 (13)	0.0068 (13)	0.0001 (12)
C3	0.0200 (15)	0.0127 (13)	0.0203 (15)	0.0007 (11)	0.0101 (13)	0.0007 (11)
C31	0.0347 (19)	0.0201 (16)	0.0276 (17)	0.0025 (14)	0.0171 (16)	−0.0037 (13)
C32	0.0247 (17)	0.0186 (15)	0.0280 (17)	0.0033 (13)	0.0112 (15)	0.0034 (13)
C4	0.0270 (18)	0.0299 (18)	0.039 (2)	0.0024 (14)	0.0177 (17)	0.0086 (15)
C41	0.026 (2)	0.040 (2)	0.048 (2)	−0.0067 (16)	0.0070 (18)	0.0027 (18)
C42	0.038 (2)	0.044 (2)	0.062 (3)	0.0066 (18)	0.033 (2)	0.011 (2)

C43	0.036 (2)	0.0285 (19)	0.046 (2)	−0.0031 (15)	0.0219 (19)	0.0064 (16)
C5	0.0279 (18)	0.0256 (17)	0.0322 (19)	0.0050 (14)	0.0134 (16)	0.0062 (14)
C51	0.035 (2)	0.034 (2)	0.051 (2)	0.0194 (17)	0.0195 (19)	0.0160 (18)
C52	0.036 (2)	0.041 (2)	0.031 (2)	0.0045 (17)	0.0115 (17)	0.0150 (16)
C6	0.0308 (18)	0.0340 (19)	0.0226 (17)	0.0049 (15)	0.0116 (15)	−0.0014 (14)
C61	0.046 (2)	0.049 (2)	0.034 (2)	0.0139 (19)	0.0201 (19)	−0.0064 (18)
C62	0.030 (2)	0.043 (2)	0.0292 (19)	−0.0094 (17)	0.0088 (16)	−0.0113 (16)
C63	0.044 (2)	0.044 (2)	0.0217 (18)	0.0098 (18)	0.0131 (17)	0.0049 (16)

Geometric parameters (Å, º)

Pd1—Cl1	2.3100 (7)	C31—H31A	0.9800
Pd1—Cl2	2.3169 (7)	C31—H31B	0.9800
Pd1—Se1	2.4411 (4)	C31—H31C	0.9800
Pd1—Se2	2.4573 (4)	C32—H32A	0.9800
Se1—P1	2.1870 (8)	C32—H32B	0.9800
Se2—P2	2.1936 (8)	C32—H32C	0.9800
P1—C3	1.856 (3)	C4—C41	1.517 (5)
P1—C2	1.886 (3)	C4—C43	1.535 (5)
P1—C1	1.887 (3)	C4—C42	1.541 (5)
P2—C5	1.840 (3)	C41—H41A	0.9800
P2—C6	1.869 (3)	C41—H41B	0.9800
P2—C4	1.913 (4)	C41—H41C	0.9800
C1—C11	1.534 (4)	C42—H42A	0.9800
C1—C13	1.537 (4)	C42—H42B	0.9800
C1—C12	1.544 (4)	C42—H42C	0.9800
C11—H11A	0.9800	C43—H43A	0.9800
C11—H11B	0.9800	C43—H43B	0.9800
C11—H11C	0.9800	C43—H43C	0.9800
C12—H12A	0.9800	C5—C52	1.532 (5)
C12—H12B	0.9800	C5—C51	1.547 (5)
C12—H12C	0.9800	C5—H5	1.0000
C13—H13A	0.9800	C51—H51A	0.9800
C13—H13B	0.9800	C51—H51B	0.9800
C13—H13C	0.9800	C51—H51C	0.9800
C2—C21	1.532 (4)	C52—H52A	0.9800
C2—C22	1.540 (4)	C52—H52B	0.9800
C2—C23	1.543 (4)	C52—H52C	0.9800
C21—H21A	0.9800	C6—C61	1.520 (5)
C21—H21B	0.9800	C6—C62	1.524 (5)
C21—H21C	0.9800	C6—C63	1.547 (5)
C22—H22A	0.9800	C61—H61A	0.9800
C22—H22B	0.9800	C61—H61B	0.9800
C22—H22C	0.9800	C61—H61C	0.9800
C23—H23A	0.9800	C62—H62A	0.9800
C23—H23B	0.9800	C62—H62B	0.9800
C23—H23C	0.9800	C62—H62C	0.9800
C3—C31	1.534 (4)	C63—H63A	0.9800

C3—C32	1.541 (4)	C63—H63B	0.9800
C3—H3	1.0000	C63—H63C	0.9800
Cl1—Pd1—Cl2	174.61 (3)	H31A—C31—H31B	109.5
Cl1—Pd1—Se1	86.97 (2)	C3—C31—H31C	109.5
Cl2—Pd1—Se1	92.46 (2)	H31A—C31—H31C	109.5
Cl1—Pd1—Se2	99.50 (2)	H31B—C31—H31C	109.5
Cl2—Pd1—Se2	80.81 (2)	C3—C32—H32A	109.5
Se1—Pd1—Se2	172.772 (14)	C3—C32—H32B	109.5
P1—Se1—Pd1	107.78 (2)	H32A—C32—H32B	109.5
P2—Se2—Pd1	117.93 (3)	C3—C32—H32C	109.5
C3—P1—C2	107.01 (13)	H32A—C32—H32C	109.5
C3—P1—C1	113.37 (13)	H32B—C32—H32C	109.5
C2—P1—C1	112.67 (13)	C41—C4—C43	108.2 (3)
C3—P1—Se1	109.18 (10)	C41—C4—C42	109.0 (3)
C2—P1—Se1	111.22 (9)	C43—C4—C42	108.1 (3)
C1—P1—Se1	103.40 (10)	C41—C4—P2	107.4 (2)
C5—P2—C6	109.22 (16)	C43—C4—P2	111.2 (2)
C5—P2—C4	109.91 (16)	C42—C4—P2	112.8 (3)
C6—P2—C4	112.17 (16)	C4—C41—H41A	109.5
C5—P2—Se2	113.08 (11)	C4—C41—H41B	109.5
C6—P2—Se2	109.66 (11)	H41A—C41—H41B	109.5
C4—P2—Se2	102.73 (11)	C4—C41—H41C	109.5
C11—C1—C13	109.9 (2)	H41A—C41—H41C	109.5
C11—C1—C12	107.5 (2)	H41B—C41—H41C	109.5
C13—C1—C12	107.7 (3)	C4—C42—H42A	109.5
C11—C1—P1	109.5 (2)	C4—C42—H42B	109.5
C13—C1—P1	112.4 (2)	H42A—C42—H42B	109.5
C12—C1—P1	109.6 (2)	C4—C42—H42C	109.5
C1—C11—H11A	109.5	H42A—C42—H42C	109.5
C1—C11—H11B	109.5	H42B—C42—H42C	109.5
H11A—C11—H11B	109.5	C4—C43—H43A	109.5
C1—C11—H11C	109.5	C4—C43—H43B	109.5
H11A—C11—H11C	109.5	H43A—C43—H43B	109.5
H11B—C11—H11C	109.5	C4—C43—H43C	109.5
C1—C12—H12A	109.5	H43A—C43—H43C	109.5
C1—C12—H12B	109.5	H43B—C43—H43C	109.5
H12A—C12—H12B	109.5	C52—C5—C51	109.6 (3)
C1—C12—H12C	109.5	C52—C5—P2	114.2 (2)
H12A—C12—H12C	109.5	C51—C5—P2	116.3 (2)
H12B—C12—H12C	109.5	C52—C5—H5	105.2
C1—C13—H13A	109.5	C51—C5—H5	105.2
C1—C13—H13B	109.5	P2—C5—H5	105.2
H13A—C13—H13B	109.5	C5—C51—H51A	109.5
C1—C13—H13C	109.5	C5—C51—H51B	109.5
H13A—C13—H13C	109.5	H51A—C51—H51B	109.5
H13B—C13—H13C	109.5	C5—C51—H51C	109.5
C21—C2—C22	107.9 (2)	H51A—C51—H51C	109.5

C21—C2—C23	108.4 (2)	H51B—C51—H51C	109.5
C22—C2—C23	109.5 (2)	C5—C52—H52A	109.5
C21—C2—P1	111.1 (2)	C5—C52—H52B	109.5
C22—C2—P1	108.51 (19)	H52A—C52—H52B	109.5
C23—C2—P1	111.4 (2)	C5—C52—H52C	109.5
C2—C21—H21A	109.5	H52A—C52—H52C	109.5
C2—C21—H21B	109.5	H52B—C52—H52C	109.5
H21A—C21—H21B	109.5	C61—C6—C62	108.3 (3)
C2—C21—H21C	109.5	C61—C6—C63	109.1 (3)
H21A—C21—H21C	109.5	C62—C6—C63	107.3 (3)
H21B—C21—H21C	109.5	C61—C6—P2	113.4 (3)
C2—C22—H22A	109.5	C62—C6—P2	109.3 (2)
C2—C22—H22B	109.5	C63—C6—P2	109.3 (2)
H22A—C22—H22B	109.5	C6—C61—H61A	109.5
C2—C22—H22C	109.5	C6—C61—H61B	109.5
H22A—C22—H22C	109.5	H61A—C61—H61B	109.5
H22B—C22—H22C	109.5	C6—C61—H61C	109.5
C2—C23—H23A	109.5	H61A—C61—H61C	109.5
C2—C23—H23B	109.5	H61B—C61—H61C	109.5
H23A—C23—H23B	109.5	C6—C62—H62A	109.5
C2—C23—H23C	109.5	C6—C62—H62B	109.5
H23A—C23—H23C	109.5	H62A—C62—H62B	109.5
H23B—C23—H23C	109.5	C6—C62—H62C	109.5
C31—C3—C32	109.7 (2)	H62A—C62—H62C	109.5
C31—C3—P1	112.9 (2)	H62B—C62—H62C	109.5
C32—C3—P1	118.2 (2)	C6—C63—H63A	109.5
C31—C3—H3	104.9	C6—C63—H63B	109.5
C32—C3—H3	104.9	H63A—C63—H63B	109.5
P1—C3—H3	104.9	C6—C63—H63C	109.5
C3—C31—H31A	109.5	H63A—C63—H63C	109.5
C3—C31—H31B	109.5	H63B—C63—H63C	109.5

Cl1—Pd1—Se1—P1	−115.18 (3)	C1—P1—C3—C31	59.3 (3)
Cl2—Pd1—Se1—P1	70.19 (3)	Se1—P1—C3—C31	−55.4 (2)
Cl1—Pd1—Se2—P2	20.17 (3)	C2—P1—C3—C32	54.2 (3)
Cl2—Pd1—Se2—P2	−165.29 (3)	C1—P1—C3—C32	−70.6 (3)
Pd1—Se1—P1—C3	−74.49 (10)	Se1—P1—C3—C32	174.7 (2)
Pd1—Se1—P1—C2	43.37 (10)	C5—P2—C4—C41	−45.7 (3)
Pd1—Se1—P1—C1	164.54 (9)	C6—P2—C4—C41	−167.4 (2)
Pd1—Se2—P2—C5	−53.52 (13)	Se2—P2—C4—C41	74.9 (2)
Pd1—Se2—P2—C6	68.63 (12)	C5—P2—C4—C43	−163.9 (2)
Pd1—Se2—P2—C4	−171.93 (11)	C6—P2—C4—C43	74.4 (3)
C3—P1—C1—C11	169.5 (2)	Se2—P2—C4—C43	−43.3 (3)
C2—P1—C1—C11	47.7 (2)	C5—P2—C4—C42	74.4 (3)
Se1—P1—C1—C11	−72.4 (2)	C6—P2—C4—C42	−47.3 (3)
C3—P1—C1—C13	47.0 (3)	Se2—P2—C4—C42	−165.0 (2)
C2—P1—C1—C13	−74.7 (2)	C6—P2—C5—C52	−154.2 (3)
Se1—P1—C1—C13	165.09 (19)	C4—P2—C5—C52	82.4 (3)

C3—P1—C1—C12	−72.8 (2)	Se2—P2—C5—C52	−31.8 (3)
C2—P1—C1—C12	165.5 (2)	C6—P2—C5—C51	76.6 (3)
Se1—P1—C1—C12	45.3 (2)	C4—P2—C5—C51	−46.9 (3)
C3—P1—C2—C21	158.0 (2)	Se2—P2—C5—C51	−161.0 (2)
C1—P1—C2—C21	−76.8 (2)	C5—P2—C6—C61	−43.2 (3)
Se1—P1—C2—C21	38.8 (2)	C4—P2—C6—C61	78.9 (3)
C3—P1—C2—C22	39.5 (2)	Se2—P2—C6—C61	−167.7 (2)
C1—P1—C2—C22	164.73 (19)	C5—P2—C6—C62	77.6 (3)
Se1—P1—C2—C22	−79.7 (2)	C4—P2—C6—C62	−160.2 (2)
C3—P1—C2—C23	−81.1 (2)	Se2—P2—C6—C62	−46.8 (3)
C1—P1—C2—C23	44.2 (2)	C5—P2—C6—C63	−165.2 (2)
Se1—P1—C2—C23	159.75 (18)	C4—P2—C6—C63	−43.1 (3)
C2—P1—C3—C31	−175.8 (2)	Se2—P2—C6—C63	70.4 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···Se1	0.98	3.08	3.633 (3)	117
C12—H12*C*···Se1	0.98	2.72	3.282 (3)	117
C21—H21*C*···Cl1	0.98	2.77	3.741 (3)	170
C21—H21*C*···Se1	0.98	2.92	3.478 (3)	117
C3—H3···Cl2	1.00	2.77	3.608 (3)	142
C31—H31*B*···Se1	0.98	2.99	3.606 (3)	122
C41—H41*C*···Se2	0.98	3.08	3.632 (4)	117
C43—H43*A*···Se2	0.98	2.67	3.300 (4)	123
C5—H5···Cl1	1.00	2.83	3.484 (4)	124
C51—H51*C*···Cl1^{i}	0.98	2.76	3.642 (3)	150
C52—H52*B*···Cl1	0.98	2.81	3.439 (4)	123
C52—H52*B*···Se2	0.98	2.97	3.507 (4)	116
C62—H62*B*···Se2	0.98	2.91	3.456 (4)	116
C62—H62*C*···Cl1	0.98	2.82	3.552 (4)	132
C63—H63*A*···Se1^{ii}	0.98	3.05	3.948 (4)	153
C63—H63*A*···Pd1^{ii}	0.98	2.87	3.793 (4)	158

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*, −*y*+3/2, *z*−1/2.

Dibromido(di-*tert*-butylisopropylphosphine selenide-κ*Se*)palladium(II) (3)

Crystal data

$[PdBr_2(C_{11}H_{25}PSe)_2]$
M_r = 800.70
Monoclinic, $P2_1/c$
a = 15.3561 (6) Å
b = 13.4695 (4) Å
c = 16.1371 (6) Å
β = 116.558 (5)°
V = 2985.6 (2) Å^3
Z = 4
$F(000)$ = 1584
D_x = 1.781 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 11858 reflections
θ = 2.1–30.8°
μ = 5.85 mm^{-1}
T = 100 K
Plate, dichroic orange yellow
0.18 × 0.05 × 0.02 mm

Data collection

Oxford Diffraction Xcalibur, Eos diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.419, T_{max} = 0.892

120046 measured reflections
7402 independent reflections
5854 reflections with $I > 2\sigma(I)$
R_{int} = 0.104
θ_{max} = 28.3°, θ_{min} = 2.1°
h = −20→20
k = −17→17
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.083
S = 1.04
7402 reflections
278 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
$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 9.8278P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.68 e Å^{-3}
$\Delta\rho_{min}$ = −1.32 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Pd1	0.21918 (2)	0.75073 (2)	0.43434 (2)	0.01287 (7)
Br1	0.27518 (3)	0.58168 (3)	0.43222 (3)	0.02354 (11)
Br2	0.17265 (3)	0.91841 (3)	0.45811 (3)	0.01765 (9)
Se1	0.08502 (3)	0.67520 (3)	0.45392 (3)	0.01525 (9)
Se2	0.35225 (3)	0.84539 (3)	0.42600 (3)	0.02064 (10)
P1	−0.05189 (7)	0.71084 (8)	0.33294 (7)	0.0120 (2)
P2	0.44090 (8)	0.77472 (9)	0.36726 (8)	0.0192 (2)
C1	−0.1407 (3)	0.6195 (3)	0.3414 (3)	0.0182 (9)
C11	−0.1193 (3)	0.5150 (3)	0.3172 (3)	0.0240 (10)
H11A	−0.156840	0.466156	0.332898	0.036*
H11B	−0.049659	0.500810	0.352519	0.036*
H11C	−0.137787	0.511447	0.250767	0.036*
C12	−0.1279 (3)	0.6164 (4)	0.4421 (3)	0.0257 (10)
H12A	−0.175427	0.570368	0.445869	0.039*
H12B	−0.138252	0.682963	0.460610	0.039*
H12C	−0.061899	0.593931	0.483685	0.039*
C13	−0.2468 (3)	0.6477 (4)	0.2783 (3)	0.0261 (10)
H13A	−0.256585	0.652786	0.214177	0.039*
H13B	−0.261527	0.711689	0.298098	0.039*
H13C	−0.290132	0.596587	0.282483	0.039*
C2	−0.0403 (3)	0.6968 (3)	0.2225 (3)	0.0161 (8)
C21	0.0218 (3)	0.6064 (3)	0.2255 (3)	0.0207 (9)
H21A	0.030884	0.604683	0.169173	0.031*
H21B	−0.011140	0.545654	0.229574	0.031*
H21C	0.085417	0.611226	0.279842	0.031*

C22	0.0110 (3)	0.7902 (3)	0.2100 (3)	0.0200 (9)
H22A	0.073163	0.799598	0.265212	0.030*
H22B	−0.030508	0.848400	0.201391	0.030*
H22C	0.022835	0.781899	0.155587	0.030*
C23	−0.1407 (3)	0.6866 (4)	0.1375 (3)	0.0223 (9)
H23A	−0.132414	0.687864	0.080729	0.033*
H23B	−0.182575	0.741859	0.136722	0.033*
H23C	−0.170713	0.623666	0.141251	0.033*
C3	−0.0841 (3)	0.8417 (3)	0.3399 (3)	0.0160 (8)
H3	−0.027467	0.880820	0.342719	0.019*
C31	−0.0892 (4)	0.8665 (4)	0.4308 (3)	0.0270 (10)
H31A	−0.086566	0.938682	0.439388	0.041*
H31B	−0.034074	0.835710	0.482964	0.041*
H31C	−0.150280	0.840984	0.427894	0.041*
C32	−0.1734 (3)	0.8863 (3)	0.2577 (3)	0.0235 (10)
H32A	−0.232344	0.851460	0.250726	0.035*
H32B	−0.165869	0.878896	0.200851	0.035*
H32C	−0.178865	0.956897	0.269306	0.035*
C4	0.5400 (3)	0.8700 (4)	0.3894 (4)	0.0290 (11)
C41	0.6093 (4)	0.8676 (5)	0.4926 (4)	0.0457 (15)
H41A	0.656464	0.921832	0.507720	0.069*
H41B	0.643857	0.803925	0.508191	0.069*
H41C	0.572109	0.875352	0.528170	0.069*
C42	0.5960 (5)	0.8510 (5)	0.3324 (5)	0.0551 (17)
H42A	0.642049	0.905379	0.342308	0.083*
H42B	0.550002	0.847280	0.266478	0.083*
H42C	0.631709	0.788233	0.351925	0.083*
C43	0.4983 (4)	0.9747 (4)	0.3662 (4)	0.0401 (13)
H43A	0.460652	0.989064	0.400297	0.060*
H43B	0.455855	0.979673	0.299479	0.060*
H43C	0.551666	1.022634	0.383809	0.060*
C5	0.4953 (3)	0.6558 (3)	0.4234 (3)	0.0241 (10)
H5	0.441791	0.605898	0.394996	0.029*
C51	0.5784 (4)	0.6157 (4)	0.4052 (4)	0.0397 (14)
H51A	0.637091	0.655526	0.439550	0.060*
H51B	0.559936	0.619523	0.338762	0.060*
H51C	0.591391	0.546434	0.425604	0.060*
C52	0.5245 (4)	0.6523 (4)	0.5272 (3)	0.0385 (13)
H52A	0.538156	0.583456	0.548987	0.058*
H52B	0.471159	0.678218	0.538551	0.058*
H52C	0.582848	0.692822	0.560611	0.058*
C6	0.3644 (4)	0.7522 (4)	0.2406 (3)	0.0287 (11)
C61	0.4136 (5)	0.6876 (5)	0.1969 (4)	0.0529 (18)
H61A	0.427936	0.622348	0.226977	0.079*
H61B	0.474323	0.719151	0.204913	0.079*
H61C	0.370228	0.679586	0.130734	0.079*
C62	0.2698 (4)	0.7024 (5)	0.2255 (4)	0.0472 (15)
H62A	0.227124	0.696604	0.158951	0.071*

H62B	0.237428	0.742320	0.254450	0.071*
H62C	0.283632	0.636084	0.253488	0.071*
C63	0.3361 (4)	0.8524 (4)	0.1882 (4)	0.0455 (15)
H63A	0.285766	0.841135	0.124748	0.068*
H63B	0.393630	0.881876	0.186418	0.068*
H63C	0.311119	0.897575	0.220092	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.01204 (15)	0.01246 (14)	0.01323 (14)	0.00053 (11)	0.00486 (12)	−0.00056 (12)
Br1	0.0192 (2)	0.0154 (2)	0.0342 (3)	0.00275 (17)	0.0103 (2)	−0.00115 (18)
Br2	0.0166 (2)	0.01380 (19)	0.0229 (2)	0.00086 (16)	0.00917 (17)	−0.00232 (16)
Se1	0.0140 (2)	0.0174 (2)	0.01274 (19)	−0.00010 (16)	0.00447 (16)	0.00418 (16)
Se2	0.0205 (2)	0.0203 (2)	0.0266 (2)	−0.00533 (18)	0.01539 (19)	−0.00853 (18)
P1	0.0118 (5)	0.0126 (5)	0.0118 (5)	0.0001 (4)	0.0054 (4)	0.0009 (4)
P2	0.0163 (5)	0.0234 (6)	0.0195 (6)	0.0023 (4)	0.0094 (5)	−0.0018 (4)
C1	0.018 (2)	0.015 (2)	0.022 (2)	−0.0022 (17)	0.0094 (18)	0.0017 (17)
C11	0.025 (2)	0.014 (2)	0.036 (3)	−0.0052 (18)	0.016 (2)	0.0000 (19)
C12	0.028 (3)	0.030 (3)	0.028 (2)	−0.003 (2)	0.020 (2)	0.005 (2)
C13	0.018 (2)	0.025 (2)	0.034 (3)	−0.0013 (19)	0.011 (2)	0.001 (2)
C2	0.016 (2)	0.020 (2)	0.0115 (19)	−0.0045 (17)	0.0054 (16)	−0.0004 (16)
C21	0.022 (2)	0.024 (2)	0.016 (2)	0.0012 (18)	0.0093 (19)	−0.0020 (17)
C22	0.018 (2)	0.029 (2)	0.013 (2)	−0.0025 (18)	0.0063 (17)	−0.0001 (17)
C23	0.018 (2)	0.029 (2)	0.016 (2)	−0.0013 (19)	0.0037 (18)	−0.0045 (18)
C3	0.018 (2)	0.014 (2)	0.016 (2)	−0.0003 (16)	0.0069 (17)	0.0009 (16)
C31	0.033 (3)	0.022 (2)	0.031 (3)	0.005 (2)	0.019 (2)	−0.001 (2)
C32	0.023 (2)	0.018 (2)	0.028 (2)	0.0042 (18)	0.011 (2)	0.0040 (18)
C4	0.021 (2)	0.031 (3)	0.040 (3)	0.000 (2)	0.018 (2)	0.002 (2)
C41	0.025 (3)	0.045 (4)	0.046 (3)	−0.013 (3)	−0.003 (3)	0.007 (3)
C42	0.044 (4)	0.056 (4)	0.082 (5)	−0.005 (3)	0.044 (4)	0.000 (4)
C43	0.034 (3)	0.034 (3)	0.053 (4)	−0.002 (2)	0.020 (3)	0.013 (3)
C5	0.025 (2)	0.025 (2)	0.024 (2)	0.0012 (19)	0.013 (2)	−0.0006 (19)
C51	0.035 (3)	0.042 (3)	0.045 (3)	0.024 (3)	0.020 (3)	0.019 (3)
C52	0.045 (3)	0.039 (3)	0.031 (3)	−0.002 (3)	0.016 (3)	0.008 (2)
C6	0.028 (3)	0.039 (3)	0.019 (2)	0.005 (2)	0.010 (2)	−0.002 (2)
C61	0.050 (4)	0.078 (5)	0.026 (3)	0.023 (3)	0.013 (3)	−0.009 (3)
C62	0.045 (4)	0.061 (4)	0.031 (3)	−0.015 (3)	0.013 (3)	−0.015 (3)
C63	0.045 (4)	0.046 (4)	0.031 (3)	0.013 (3)	0.004 (3)	0.004 (3)

Geometric parameters (Å, º)

Pd1—Br1	2.4395 (5)	C31—H31A	0.9800
Pd1—Se1	2.4416 (5)	C31—H31B	0.9800
Pd1—Br2	2.4503 (5)	C31—H31C	0.9800
Pd1—Se2	2.4628 (5)	C32—H32A	0.9800
Se1—P1	2.1890 (11)	C32—H32B	0.9800
Se2—P2	2.1935 (12)	C32—H32C	0.9800

P1—C3	1.847 (4)	C4—C41	1.524 (7)
P1—C2	1.879 (4)	C4—C43	1.524 (7)
P1—C1	1.886 (4)	C4—C42	1.536 (7)
P2—C5	1.846 (5)	C41—H41A	0.9800
P2—C6	1.871 (5)	C41—H41B	0.9800
P2—C4	1.899 (5)	C41—H41C	0.9800
C1—C11	1.534 (6)	C42—H42A	0.9800
C1—C13	1.534 (6)	C42—H42B	0.9800
C1—C12	1.549 (6)	C42—H42C	0.9800
C11—H11A	0.9800	C43—H43A	0.9800
C11—H11B	0.9800	C43—H43B	0.9800
C11—H11C	0.9800	C43—H43C	0.9800
C12—H12A	0.9800	C5—C52	1.529 (6)
C12—H12B	0.9800	C5—C51	1.530 (6)
C12—H12C	0.9800	C5—H5	1.0000
C13—H13A	0.9800	C51—H51A	0.9800
C13—H13B	0.9800	C51—H51B	0.9800
C13—H13C	0.9800	C51—H51C	0.9800
C2—C21	1.534 (6)	C52—H52A	0.9800
C2—C22	1.544 (6)	C52—H52B	0.9800
C2—C23	1.544 (6)	C52—H52C	0.9800
C21—H21A	0.9800	C6—C61	1.517 (7)
C21—H21B	0.9800	C6—C62	1.517 (7)
C21—H21C	0.9800	C6—C63	1.549 (7)
C22—H22A	0.9800	C61—H61A	0.9800
C22—H22B	0.9800	C61—H61B	0.9800
C22—H22C	0.9800	C61—H61C	0.9800
C23—H23A	0.9800	C62—H62A	0.9800
C23—H23B	0.9800	C62—H62B	0.9800
C23—H23C	0.9800	C62—H62C	0.9800
C3—C32	1.541 (6)	C63—H63A	0.9800
C3—C31	1.541 (6)	C63—H63B	0.9800
C3—H3	1.0000	C63—H63C	0.9800

Br1—Pd1—Se1	86.332 (18)	H31A—C31—H31B	109.5
Br1—Pd1—Br2	172.66 (2)	C3—C31—H31C	109.5
Se1—Pd1—Br2	92.395 (17)	H31A—C31—H31C	109.5
Br1—Pd1—Se2	100.149 (18)	H31B—C31—H31C	109.5
Se1—Pd1—Se2	172.54 (2)	C3—C32—H32A	109.5
Br2—Pd1—Se2	80.706 (17)	C3—C32—H32B	109.5
P1—Se1—Pd1	108.83 (3)	H32A—C32—H32B	109.5
P2—Se2—Pd1	119.30 (4)	C3—C32—H32C	109.5
C3—P1—C2	107.14 (19)	H32A—C32—H32C	109.5
C3—P1—C1	113.37 (19)	H32B—C32—H32C	109.5
C2—P1—C1	112.42 (19)	C41—C4—C43	107.5 (5)
C3—P1—Se1	109.36 (14)	C41—C4—C42	110.3 (5)
C2—P1—Se1	111.31 (13)	C43—C4—C42	107.1 (5)
C1—P1—Se1	103.26 (14)	C41—C4—P2	107.4 (3)

C5—P2—C6	108.7 (2)	C43—C4—P2	111.4 (3)
C5—P2—C4	110.2 (2)	C42—C4—P2	113.0 (4)
C6—P2—C4	112.0 (2)	C4—C41—H41A	109.5
C5—P2—Se2	113.36 (14)	C4—C41—H41B	109.5
C6—P2—Se2	109.52 (16)	H41A—C41—H41B	109.5
C4—P2—Se2	103.04 (16)	C4—C41—H41C	109.5
C11—C1—C13	110.1 (4)	H41A—C41—H41C	109.5
C11—C1—C12	107.5 (4)	H41B—C41—H41C	109.5
C13—C1—C12	107.5 (4)	C4—C42—H42A	109.5
C11—C1—P1	109.7 (3)	C4—C42—H42B	109.5
C13—C1—P1	112.2 (3)	H42A—C42—H42B	109.5
C12—C1—P1	109.7 (3)	C4—C42—H42C	109.5
C1—C11—H11A	109.5	H42A—C42—H42C	109.5
C1—C11—H11B	109.5	H42B—C42—H42C	109.5
H11A—C11—H11B	109.5	C4—C43—H43A	109.5
C1—C11—H11C	109.5	C4—C43—H43B	109.5
H11A—C11—H11C	109.5	H43A—C43—H43B	109.5
H11B—C11—H11C	109.5	C4—C43—H43C	109.5
C1—C12—H12A	109.5	H43A—C43—H43C	109.5
C1—C12—H12B	109.5	H43B—C43—H43C	109.5
H12A—C12—H12B	109.5	C52—C5—C51	109.8 (4)
C1—C12—H12C	109.5	C52—C5—P2	114.3 (3)
H12A—C12—H12C	109.5	C51—C5—P2	116.4 (3)
H12B—C12—H12C	109.5	C52—C5—H5	105.0
C1—C13—H13A	109.5	C51—C5—H5	105.0
C1—C13—H13B	109.5	P2—C5—H5	105.0
H13A—C13—H13B	109.5	C5—C51—H51A	109.5
C1—C13—H13C	109.5	C5—C51—H51B	109.5
H13A—C13—H13C	109.5	H51A—C51—H51B	109.5
H13B—C13—H13C	109.5	C5—C51—H51C	109.5
C21—C2—C22	107.8 (3)	H51A—C51—H51C	109.5
C21—C2—C23	108.5 (3)	H51B—C51—H51C	109.5
C22—C2—C23	108.8 (3)	C5—C52—H52A	109.5
C21—C2—P1	111.7 (3)	C5—C52—H52B	109.5
C22—C2—P1	108.2 (3)	H52A—C52—H52B	109.5
C23—C2—P1	111.7 (3)	C5—C52—H52C	109.5
C2—C21—H21A	109.5	H52A—C52—H52C	109.5
C2—C21—H21B	109.5	H52B—C52—H52C	109.5
H21A—C21—H21B	109.5	C61—C6—C62	108.2 (5)
C2—C21—H21C	109.5	C61—C6—C63	108.7 (5)
H21A—C21—H21C	109.5	C62—C6—C63	106.5 (5)
H21B—C21—H21C	109.5	C61—C6—P2	113.6 (4)
C2—C22—H22A	109.5	C62—C6—P2	109.6 (3)
C2—C22—H22B	109.5	C63—C6—P2	110.0 (4)
H22A—C22—H22B	109.5	C6—C61—H61A	109.5
C2—C22—H22C	109.5	C6—C61—H61B	109.5
H22A—C22—H22C	109.5	H61A—C61—H61B	109.5
H22B—C22—H22C	109.5	C6—C61—H61C	109.5

C2—C23—H23A	109.5	H61A—C61—H61C	109.5
C2—C23—H23B	109.5	H61B—C61—H61C	109.5
H23A—C23—H23B	109.5	C6—C62—H62A	109.5
C2—C23—H23C	109.5	C6—C62—H62B	109.5
H23A—C23—H23C	109.5	H62A—C62—H62B	109.5
H23B—C23—H23C	109.5	C6—C62—H62C	109.5
C32—C3—C31	109.2 (4)	H62A—C62—H62C	109.5
C32—C3—P1	118.6 (3)	H62B—C62—H62C	109.5
C31—C3—P1	113.2 (3)	C6—C63—H63A	109.5
C32—C3—H3	104.8	C6—C63—H63B	109.5
C31—C3—H3	104.8	H63A—C63—H63B	109.5
P1—C3—H3	104.8	C6—C63—H63C	109.5
C3—C31—H31A	109.5	H63A—C63—H63C	109.5
C3—C31—H31B	109.5	H63B—C63—H63C	109.5

Br1—Pd1—Se1—P1	−116.19 (3)	C1—P1—C3—C32	−70.6 (4)
Br2—Pd1—Se1—P1	71.05 (3)	Se1—P1—C3—C32	174.8 (3)
Br1—Pd1—Se2—P2	23.37 (4)	C2—P1—C3—C31	−176.0 (3)
Br2—Pd1—Se2—P2	−164.03 (4)	C1—P1—C3—C31	59.4 (4)
Pd1—Se1—P1—C3	−74.90 (14)	Se1—P1—C3—C31	−55.2 (3)
Pd1—Se1—P1—C2	43.29 (15)	C5—P2—C4—C41	−47.9 (4)
Pd1—Se1—P1—C1	164.12 (14)	C6—P2—C4—C41	−169.0 (4)
Pd1—Se2—P2—C5	−54.43 (18)	Se2—P2—C4—C41	73.4 (4)
Pd1—Se2—P2—C6	67.15 (18)	C5—P2—C4—C43	−165.3 (4)
Pd1—Se2—P2—C4	−173.53 (16)	C6—P2—C4—C43	73.5 (4)
C3—P1—C1—C11	169.6 (3)	Se2—P2—C4—C43	−44.1 (4)
C2—P1—C1—C11	47.9 (4)	C5—P2—C4—C42	74.0 (4)
Se1—P1—C1—C11	−72.2 (3)	C6—P2—C4—C42	−47.1 (5)
C3—P1—C1—C13	47.0 (4)	Se2—P2—C4—C42	−164.7 (4)
C2—P1—C1—C13	−74.7 (4)	C6—P2—C5—C52	−156.7 (4)
Se1—P1—C1—C13	165.2 (3)	C4—P2—C5—C52	80.3 (4)
C3—P1—C1—C12	−72.5 (3)	Se2—P2—C5—C52	−34.6 (4)
C2—P1—C1—C12	165.8 (3)	C6—P2—C5—C51	73.6 (4)
Se1—P1—C1—C12	45.7 (3)	C4—P2—C5—C51	−49.4 (4)
C3—P1—C2—C21	158.5 (3)	Se2—P2—C5—C51	−164.3 (3)
C1—P1—C2—C21	−76.3 (3)	C5—P2—C6—C61	−45.5 (5)
Se1—P1—C2—C21	38.9 (3)	C4—P2—C6—C61	76.5 (5)
C3—P1—C2—C22	40.0 (3)	Se2—P2—C6—C61	−169.9 (4)
C1—P1—C2—C22	165.2 (3)	C5—P2—C6—C62	75.6 (4)
Se1—P1—C2—C22	−79.5 (3)	C4—P2—C6—C62	−162.4 (4)
C3—P1—C2—C23	−79.7 (3)	Se2—P2—C6—C62	−48.7 (4)
C1—P1—C2—C23	45.5 (4)	C5—P2—C6—C63	−167.7 (4)
Se1—P1—C2—C23	160.8 (3)	C4—P2—C6—C63	−45.7 (4)
C2—P1—C3—C32	54.0 (4)	Se2—P2—C6—C63	68.0 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···Se1	0.98	3.07	3.630 (4)	117
C12—H12*C*···Se1	0.98	2.74	3.286 (5)	116
C21—H21*C*···Br1	0.98	2.88	3.843 (4)	168
C21—H21*C*···Se1	0.98	2.94	3.491 (4)	117
C22—H22*A*···Pd1	0.98	2.73	3.641 (4)	155
C3—H3···Br2	1.00	2.84	3.678 (4)	142
C31—H31*B*···Se1	0.98	3.00	3.612 (5)	122
C41—H41*C*···Se2	0.98	3.05	3.610 (6)	118
C43—H43*A*···Se2	0.98	2.70	3.307 (5)	120
C5—H5···Br1	1.00	2.90	3.588 (5)	127
C51—H51*C*···Br1^{i}	0.98	2.87	3.706 (5)	144
C52—H52*B*···Br1	0.98	3.01	3.558 (6)	116
C52—H52*B*···Se2	0.98	2.96	3.545 (6)	120
C62—H62*B*···Se2	0.98	2.89	3.481 (6)	120
C62—H62*C*···Br1	0.98	3.03	3.678 (6)	124
C63—H63*A*···Se1^{ii}	0.98	3.09	4.032 (6)	162
C63—H63*A*···Pd1^{ii}	0.98	3.03	3.918 (6)	151

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*, −*y*+3/2, *z*−1/2.

Dibromido(di-*tert*-butylisopropylphosphine sulfide-κ*S*)palladium(II) (4)

Crystal data

$[PdBr_2(C_{11}H_{25}PS)_2]$
M_r = 706.90
Monoclinic, $P2_1/n$
a = 7.8595 (3) Å
b = 17.5019 (6) Å
c = 10.6740 (3) Å
β = 94.551 (3)°
V = 1463.65 (9) Å^3
Z = 2
$F(000)$ = 720
D_x = 1.604 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9329 reflections
θ = 2.2–30.8°
μ = 3.63 mm^{-1}
T = 100 K
Prism, red
0.2 × 0.08 × 0.05 mm

Data collection

Oxford Diffraction Xcalibur, Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Detector resolution: 16.1419 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.865, T_{max} = 1.000
45160 measured reflections
4422 independent reflections
3832 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 30.9°, θ_{min} = 2.2°
h = −10→11
k = −25→24
l = −15→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.065
S = 1.22
4422 reflections
141 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0106P)^2 + 2.7056P]$ 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	$\Delta\rho_{max} = 1.02$ e Å^{-3}
	$\Delta\rho_{min} = -0.70$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.500000	0.500000	0.500000	0.01288 (6)
Br1	0.42664 (4)	0.43715 (2)	0.69362 (2)	0.02184 (7)
S1	0.50672 (9)	0.61904 (4)	0.59836 (6)	0.01631 (13)
P1	0.69438 (9)	0.63805 (4)	0.73533 (6)	0.01672 (14)
C1	0.6766 (5)	0.74449 (17)	0.7604 (3)	0.0303 (7)
C2	0.9064 (4)	0.6062 (3)	0.6863 (3)	0.0394 (9)
C3	0.6566 (4)	0.58401 (15)	0.8794 (2)	0.0177 (5)
H3	0.643819	0.529579	0.851762	0.021*
C11	0.4869 (5)	0.76795 (18)	0.7592 (3)	0.0370 (9)
H11A	0.479014	0.822916	0.775306	0.055*
H11B	0.432726	0.739706	0.824649	0.055*
H11C	0.428534	0.756105	0.676939	0.055*
C12	0.7681 (5)	0.76860 (19)	0.8859 (3)	0.0360 (8)
H12A	0.767553	0.824455	0.892452	0.054*
H12B	0.886187	0.750204	0.890819	0.054*
H12C	0.709110	0.746614	0.954975	0.054*
C13	0.7540 (6)	0.7875 (2)	0.6522 (3)	0.0526 (12)
H13A	0.875993	0.775669	0.652991	0.079*
H13B	0.739078	0.842626	0.663040	0.079*
H13C	0.696157	0.771508	0.571718	0.079*
C21	0.9235 (5)	0.6266 (3)	0.5471 (3)	0.0556 (13)
H21A	1.031394	0.606305	0.520980	0.083*
H21B	0.921907	0.682258	0.537176	0.083*
H21C	0.828103	0.604191	0.494919	0.083*
C22	0.9172 (5)	0.5191 (3)	0.6991 (4)	0.0543 (13)
H22A	0.914052	0.504892	0.787721	0.082*
H22B	1.024007	0.501008	0.667953	0.082*
H22C	0.820267	0.495659	0.649915	0.082*
C23	1.0582 (5)	0.6450 (3)	0.7640 (4)	0.0599 (14)
H23A	1.053169	0.632649	0.853171	0.090*
H23B	1.051007	0.700531	0.752609	0.090*
H23C	1.166053	0.626419	0.735231	0.090*
C31	0.7987 (4)	0.58306 (19)	0.9882 (3)	0.0278 (7)
H31A	0.763606	0.550379	1.056143	0.042*
H31B	0.818221	0.635141	1.019900	0.042*
H31C	0.904217	0.563072	0.957619	0.042*
C32	0.4866 (4)	0.60471 (18)	0.9302 (3)	0.0262 (6)
H32A	0.493743	0.656222	0.966213	0.039*
H32B	0.460738	0.567997	0.995351	0.039*

H32C	0.395990	0.603216	0.861626	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.01454 (13)	0.01546 (12)	0.00854 (12)	0.00066 (11)	0.00023 (9)	−0.00076 (10)
Br1	0.03258 (17)	0.02237 (13)	0.01082 (12)	−0.00486 (12)	0.00328 (11)	0.00052 (10)
S1	0.0192 (3)	0.0167 (3)	0.0125 (3)	0.0023 (2)	−0.0020 (2)	−0.0014 (2)
P1	0.0159 (3)	0.0239 (3)	0.0104 (3)	−0.0046 (3)	0.0015 (3)	−0.0018 (3)
C1	0.050 (2)	0.0214 (14)	0.0183 (14)	−0.0136 (14)	−0.0064 (14)	−0.0016 (11)
C2	0.0112 (15)	0.086 (3)	0.0220 (16)	0.0006 (16)	0.0038 (12)	−0.0152 (17)
C3	0.0209 (14)	0.0188 (12)	0.0136 (12)	0.0014 (10)	0.0019 (10)	0.0009 (10)
C11	0.061 (3)	0.0201 (15)	0.0269 (17)	0.0095 (15)	−0.0133 (16)	−0.0059 (12)
C12	0.055 (2)	0.0295 (16)	0.0225 (16)	−0.0164 (16)	−0.0039 (15)	−0.0035 (13)
C13	0.089 (3)	0.044 (2)	0.0234 (17)	−0.041 (2)	−0.0055 (19)	0.0074 (15)
C21	0.029 (2)	0.119 (4)	0.0205 (17)	−0.021 (2)	0.0115 (15)	−0.021 (2)
C22	0.030 (2)	0.095 (4)	0.037 (2)	0.035 (2)	−0.0038 (16)	−0.022 (2)
C23	0.0173 (18)	0.132 (5)	0.031 (2)	−0.013 (2)	0.0039 (15)	−0.020 (2)
C31	0.0313 (18)	0.0345 (17)	0.0164 (14)	0.0034 (13)	−0.0056 (12)	−0.0003 (12)
C32	0.0258 (16)	0.0356 (17)	0.0181 (14)	0.0023 (13)	0.0077 (12)	0.0040 (12)

Geometric parameters (Å, º)

Pd1—S1^{i}	2.3317 (6)	C12—H12B	0.9800
Pd1—S1	2.3317 (6)	C12—H12C	0.9800
Pd1—Br1	2.4501 (3)	C13—H13A	0.9800
Pd1—Br1^{i}	2.4501 (3)	C13—H13B	0.9800
S1—P1	2.0202 (10)	C13—H13C	0.9800
P1—C3	1.849 (3)	C21—H21A	0.9800
P1—C2	1.871 (3)	C21—H21B	0.9800
P1—C1	1.889 (3)	C21—H21C	0.9800
C1—C12	1.529 (4)	C22—H22A	0.9800
C1—C13	1.544 (5)	C22—H22B	0.9800
C1—C11	1.546 (5)	C22—H22C	0.9800
C2—C22	1.532 (6)	C23—H23A	0.9800
C2—C21	1.544 (5)	C23—H23B	0.9800
C2—C23	1.555 (5)	C23—H23C	0.9800
C3—C32	1.525 (4)	C31—H31A	0.9800
C3—C31	1.546 (4)	C31—H31B	0.9800
C3—H3	1.0000	C31—H31C	0.9800
C11—H11A	0.9800	C32—H32A	0.9800
C11—H11B	0.9800	C32—H32B	0.9800
C11—H11C	0.9800	C32—H32C	0.9800
C12—H12A	0.9800		
S1^{i}—Pd1—S1	180.0	C1—C12—H12C	109.5
S1^{i}—Pd1—Br1	88.867 (17)	H12A—C12—H12C	109.5
S1—Pd1—Br1	91.133 (17)	H12B—C12—H12C	109.5

S1^{i}—Pd1—Br1^{i}	91.133 (17)	C1—C13—H13A	109.5
S1—Pd1—Br1^{i}	88.867 (17)	C1—C13—H13B	109.5
Br1—Pd1—Br1^{i}	180.0	H13A—C13—H13B	109.5
P1—S1—Pd1	117.53 (4)	C1—C13—H13C	109.5
C3—P1—C2	106.59 (16)	H13A—C13—H13C	109.5
C3—P1—C1	111.60 (13)	H13B—C13—H13C	109.5
C2—P1—C1	114.28 (18)	C2—C21—H21A	109.5
C3—P1—S1	111.18 (10)	C2—C21—H21B	109.5
C2—P1—S1	111.32 (11)	H21A—C21—H21B	109.5
C1—P1—S1	101.95 (11)	C2—C21—H21C	109.5
C12—C1—C13	109.6 (3)	H21A—C21—H21C	109.5
C12—C1—C11	108.6 (3)	H21B—C21—H21C	109.5
C13—C1—C11	107.6 (3)	C2—C22—H22A	109.5
C12—C1—P1	111.2 (2)	C2—C22—H22B	109.5
C13—C1—P1	109.7 (2)	H22A—C22—H22B	109.5
C11—C1—P1	110.0 (2)	C2—C22—H22C	109.5
C22—C2—C21	108.0 (3)	H22A—C22—H22C	109.5
C22—C2—C23	110.7 (4)	H22B—C22—H22C	109.5
C21—C2—C23	106.9 (3)	C2—C23—H23A	109.5
C22—C2—P1	108.4 (3)	C2—C23—H23B	109.5
C21—C2—P1	110.3 (3)	H23A—C23—H23B	109.5
C23—C2—P1	112.5 (3)	C2—C23—H23C	109.5
C32—C3—C31	109.7 (2)	H23A—C23—H23C	109.5
C32—C3—P1	112.16 (19)	H23B—C23—H23C	109.5
C31—C3—P1	118.4 (2)	C3—C31—H31A	109.5
C32—C3—H3	105.1	C3—C31—H31B	109.5
C31—C3—H3	105.1	H31A—C31—H31B	109.5
P1—C3—H3	105.1	C3—C31—H31C	109.5
C1—C11—H11A	109.5	H31A—C31—H31C	109.5
C1—C11—H11B	109.5	H31B—C31—H31C	109.5
H11A—C11—H11B	109.5	C3—C32—H32A	109.5
C1—C11—H11C	109.5	C3—C32—H32B	109.5
H11A—C11—H11C	109.5	H32A—C32—H32B	109.5
H11B—C11—H11C	109.5	C3—C32—H32C	109.5
C1—C12—H12A	109.5	H32A—C32—H32C	109.5
C1—C12—H12B	109.5	H32B—C32—H32C	109.5
H12A—C12—H12B	109.5		

Br1—Pd1—S1—P1	67.56 (4)	C1—P1—C2—C22	167.0 (2)
Br1^{i}—Pd1—S1—P1	−112.44 (4)	S1—P1—C2—C22	−78.1 (3)
Pd1—S1—P1—C3	−72.48 (10)	C3—P1—C2—C21	161.3 (3)
Pd1—S1—P1—C2	46.20 (16)	C1—P1—C2—C21	−74.9 (3)
Pd1—S1—P1—C1	168.46 (11)	S1—P1—C2—C21	39.9 (3)
C3—P1—C1—C12	43.8 (3)	C3—P1—C2—C23	−79.4 (3)
C2—P1—C1—C12	−77.2 (3)	C1—P1—C2—C23	44.3 (4)
S1—P1—C1—C12	162.6 (2)	S1—P1—C2—C23	159.2 (3)
C3—P1—C1—C13	165.2 (2)	C2—P1—C3—C32	179.9 (2)
C2—P1—C1—C13	44.2 (3)	C1—P1—C3—C32	54.5 (3)

S1—P1—C1—C13	−76.0 (3)	S1—P1—C3—C32	−58.6 (2)
C3—P1—C1—C11	−76.6 (2)	C2—P1—C3—C31	50.6 (3)
C2—P1—C1—C11	162.3 (2)	C1—P1—C3—C31	−74.8 (3)
S1—P1—C1—C11	42.1 (2)	S1—P1—C3—C31	172.08 (19)
C3—P1—C2—C22	43.3 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···Br1	1.00	2.81	3.638 (3)	140
C11—H11*C*···S1	0.98	2.63	3.132 (3)	112
C21—H21*C*···S1	0.98	2.85	3.366 (4)	114
C32—H32*C*···S1	0.98	3.02	3.567 (3)	117
C22—H22*C*···Pd1	0.98	2.87	3.778 (4)	154
C12—H12*A*···S1^{ii}	0.98	2.95	3.443 (3)	112
C21—H21*C*···Br1^{i}	0.98	2.82	3.782 (4)	168

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*+1/2, −*y*+3/2, *z*+1/2.

Dichlorido(di-*tert*-butylisopropylphosphine sulfide-κ*S*)palladium(II) (5)

Crystal data

$[PdCl_2(C_{11}H_{25}PS)_2]$
M_r = 706.67
Monoclinic, $P2_1/c$
a = 14.5283 (3) Å
b = 14.4191 (3) Å
c = 13.9428 (4) Å
β = 94.571 (3)°
V = 2911.53 (12) Å^3
Z = 4
$F(000)$ = 1424
D_x = 1.612 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 16523 reflections
θ = 2.4–30.8°
μ = 5.27 mm^{-1}
T = 100 K
Plate, orange
0.2 × 0.1 × 0.07 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 (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.650, T_{max} = 1.000
79369 measured reflections
8721 independent reflections
6105 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 30.9°, θ_{min} = 2.4°
h = −20→19
k = −20→20
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.055
S = 1.04
8721 reflections
281 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

$w = 1/[\sigma^2(F_o^2) + (0.0154P)^2 + 5.0893P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 1.14$ e Å^{-3}
$\Delta\rho_{min} = -0.97$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pt1	0.500000	0.500000	0.500000	0.01098 (4)
Pt2	1.000000	0.000000	0.500000	0.01384 (4)
Cl1	0.63902 (5)	0.51548 (5)	0.43147 (5)	0.01956 (14)
Cl2	1.11830 (6)	−0.09779 (5)	0.46453 (7)	0.0331 (2)
S1	0.46640 (5)	0.65107 (5)	0.44755 (5)	0.01454 (13)
S2	1.10486 (5)	0.11932 (5)	0.53673 (5)	0.02066 (15)
P1	0.36073 (5)	0.65824 (5)	0.34317 (5)	0.01255 (13)
P2	1.15656 (5)	0.17568 (5)	0.41869 (5)	0.01434 (14)
C1	0.3559 (2)	0.78408 (19)	0.3069 (2)	0.0168 (6)
C2	0.3820 (2)	0.5802 (2)	0.2395 (2)	0.0210 (6)
C3	0.2543 (2)	0.6187 (2)	0.3944 (2)	0.0213 (6)
H3	0.268369	0.554165	0.417452	0.026*
C11	0.4342 (2)	0.8074 (2)	0.2434 (2)	0.0241 (7)
H11A	0.434372	0.874186	0.230651	0.036*
H11B	0.424701	0.773586	0.182419	0.036*
H11C	0.493434	0.789167	0.276544	0.036*
C12	0.3691 (2)	0.8452 (2)	0.3974 (2)	0.0259 (7)
H12A	0.429443	0.832118	0.431296	0.039*
H12B	0.320267	0.831639	0.439964	0.039*
H12C	0.366055	0.910682	0.378725	0.039*
C13	0.2617 (2)	0.8088 (2)	0.2546 (2)	0.0249 (7)
H13A	0.264392	0.871411	0.227656	0.037*
H13B	0.213872	0.806424	0.300297	0.037*
H13C	0.246881	0.764237	0.202563	0.037*
C21	0.3226 (3)	0.6059 (2)	0.1473 (2)	0.0356 (9)
H21A	0.338772	0.668409	0.126848	0.053*
H21B	0.257188	0.604125	0.159742	0.053*
H21C	0.333941	0.561417	0.096345	0.053*
C22	0.3590 (3)	0.4801 (2)	0.2688 (2)	0.0285 (8)
H22A	0.376003	0.436967	0.218822	0.043*
H22B	0.292668	0.475023	0.276190	0.043*
H22C	0.393688	0.464781	0.329878	0.043*
C23	0.4838 (2)	0.5823 (2)	0.2187 (2)	0.0293 (8)
H23A	0.494595	0.536750	0.168649	0.044*
H23B	0.522372	0.567268	0.277471	0.044*
H23C	0.499647	0.644401	0.196502	0.044*
C31	0.1662 (2)	0.6095 (3)	0.3268 (3)	0.0405 (10)
H31A	0.117957	0.579561	0.361093	0.061*
H31B	0.179136	0.571937	0.270916	0.061*
H31C	0.145316	0.671282	0.305321	0.061*
C32	0.2342 (2)	0.6725 (2)	0.4846 (2)	0.0312 (8)

H32A	0.205901	0.732244	0.465998	0.047*
H32B	0.291972	0.683219	0.524245	0.047*
H32C	0.191773	0.636716	0.521383	0.047*
C4	1.28268 (19)	0.1938 (2)	0.4566 (2)	0.0179 (6)
C5	1.1497 (2)	0.0919 (2)	0.3175 (2)	0.0208 (6)
H6	1.171530	0.031438	0.346156	0.025*
C6	1.0935 (2)	0.2867 (2)	0.3861 (2)	0.0211 (6)
C41	1.3291 (2)	0.0980 (2)	0.4566 (2)	0.0260 (7)
H41A	1.294399	0.054273	0.493665	0.039*
H41B	1.392548	0.102896	0.485921	0.039*
H41C	1.330114	0.075718	0.390301	0.039*
C42	1.2955 (2)	0.2306 (2)	0.5602 (2)	0.0249 (7)
H42A	1.266935	0.292009	0.563232	0.037*
H42B	1.361545	0.235329	0.580115	0.037*
H42C	1.266161	0.188009	0.603242	0.037*
C43	1.3306 (2)	0.2614 (2)	0.3912 (2)	0.0218 (6)
H43A	1.397265	0.261236	0.408971	0.033*
H43B	1.306219	0.324094	0.399079	0.033*
H43C	1.318924	0.242032	0.323990	0.033*
C51	1.0518 (2)	0.0737 (2)	0.2732 (2)	0.0285 (7)
H51A	1.051247	0.017243	0.234046	0.043*
H51B	1.031131	0.126376	0.232513	0.043*
H51C	1.010113	0.065921	0.324517	0.043*
C52	1.2114 (2)	0.1109 (2)	0.2353 (2)	0.0292 (7)
H52A	1.193601	0.170001	0.204498	0.044*
H52B	1.203986	0.060810	0.187854	0.044*
H52C	1.276058	0.114029	0.261225	0.044*
C61	1.1185 (2)	0.3602 (2)	0.4628 (2)	0.0289 (7)
H61A	1.107728	0.335411	0.526414	0.043*
H61B	1.079998	0.415351	0.449882	0.043*
H61C	1.183701	0.377119	0.461402	0.043*
C62	1.1152 (2)	0.3226 (2)	0.2865 (2)	0.0290 (7)
H62A	1.182092	0.329578	0.284636	0.043*
H62B	1.085109	0.382733	0.274379	0.043*
H62C	1.092081	0.278224	0.237003	0.043*
C63	0.9891 (2)	0.2686 (2)	0.3848 (3)	0.0300 (8)
H63A	0.955485	0.326305	0.369323	0.045*
H63B	0.974242	0.246472	0.448200	0.045*
H63C	0.971147	0.221626	0.336124	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.00922 (7)	0.01024 (7)	0.01332 (7)	−0.00095 (5)	−0.00001 (5)	0.00139 (5)
Pt2	0.01099 (7)	0.01107 (7)	0.02000 (8)	−0.00090 (6)	0.00461 (5)	0.00118 (6)
Cl1	0.0139 (3)	0.0204 (3)	0.0250 (4)	0.0013 (3)	0.0052 (3)	0.0077 (3)
Cl2	0.0249 (4)	0.0179 (4)	0.0592 (6)	0.0054 (3)	0.0209 (4)	0.0058 (4)
S1	0.0135 (3)	0.0108 (3)	0.0187 (3)	−0.0019 (2)	−0.0024 (3)	0.0010 (3)

S2	0.0246 (4)	0.0211 (4)	0.0165 (4)	−0.0097 (3)	0.0029 (3)	0.0013 (3)
P1	0.0142 (3)	0.0103 (3)	0.0129 (3)	−0.0006 (3)	−0.0003 (3)	0.0010 (3)
P2	0.0141 (3)	0.0145 (3)	0.0144 (3)	−0.0025 (3)	0.0010 (3)	0.0012 (3)
C1	0.0220 (15)	0.0108 (12)	0.0178 (14)	0.0014 (11)	0.0019 (11)	0.0013 (10)
C2	0.0367 (18)	0.0136 (13)	0.0118 (14)	0.0033 (12)	−0.0031 (12)	−0.0010 (11)
C3	0.0146 (14)	0.0235 (15)	0.0255 (16)	−0.0009 (12)	−0.0011 (12)	0.0105 (12)
C11	0.0313 (17)	0.0137 (13)	0.0280 (17)	0.0000 (12)	0.0070 (14)	0.0079 (12)
C12	0.0398 (19)	0.0119 (13)	0.0254 (16)	0.0015 (13)	−0.0001 (14)	−0.0030 (12)
C13	0.0270 (17)	0.0228 (15)	0.0249 (16)	0.0072 (13)	0.0021 (13)	0.0068 (13)
C21	0.062 (3)	0.0232 (17)	0.0197 (17)	0.0024 (17)	−0.0091 (16)	−0.0039 (13)
C22	0.047 (2)	0.0134 (14)	0.0229 (16)	−0.0028 (13)	−0.0073 (15)	−0.0033 (12)
C23	0.047 (2)	0.0210 (16)	0.0223 (16)	0.0117 (15)	0.0161 (15)	0.0023 (13)
C31	0.0197 (17)	0.048 (2)	0.051 (2)	−0.0112 (16)	−0.0102 (16)	0.0242 (19)
C32	0.0258 (17)	0.0306 (18)	0.039 (2)	0.0119 (14)	0.0170 (15)	0.0122 (15)
C4	0.0146 (14)	0.0179 (13)	0.0210 (15)	−0.0035 (11)	0.0007 (11)	0.0037 (11)
C5	0.0227 (15)	0.0208 (14)	0.0189 (15)	−0.0034 (12)	0.0016 (12)	−0.0017 (12)
C6	0.0209 (15)	0.0171 (14)	0.0253 (16)	0.0016 (12)	0.0013 (12)	0.0041 (12)
C41	0.0201 (16)	0.0212 (15)	0.0363 (19)	0.0014 (12)	−0.0003 (13)	0.0049 (13)
C42	0.0234 (16)	0.0271 (16)	0.0235 (16)	−0.0078 (13)	−0.0021 (13)	0.0007 (13)
C43	0.0180 (15)	0.0239 (15)	0.0239 (16)	−0.0060 (12)	0.0037 (12)	0.0012 (12)
C51	0.0283 (18)	0.0338 (18)	0.0226 (17)	−0.0085 (14)	−0.0027 (13)	−0.0063 (14)
C52	0.0323 (18)	0.0345 (18)	0.0218 (17)	−0.0049 (15)	0.0077 (14)	−0.0071 (14)
C61	0.0359 (19)	0.0190 (15)	0.0321 (18)	0.0069 (14)	0.0042 (15)	−0.0009 (13)
C62	0.0306 (18)	0.0266 (17)	0.0296 (18)	0.0050 (14)	0.0019 (14)	0.0124 (14)
C63	0.0187 (16)	0.0340 (19)	0.037 (2)	0.0052 (14)	0.0026 (14)	0.0068 (15)

Geometric parameters (Å, º)

Pt1—Cl1	2.3129 (7)	C23—H23B	0.9800
Pt1—Cl1^{i}	2.3129 (7)	C23—H23C	0.9800
Pt1—S1	2.3369 (7)	C31—H31A	0.9800
Pt1—S1^{i}	2.3369 (7)	C31—H31B	0.9800
Pt2—Cl2	2.3070 (7)	C31—H31C	0.9800
Pt2—Cl2^{ii}	2.3070 (7)	C32—H32A	0.9800
Pt2—S2^{ii}	2.3278 (7)	C32—H32B	0.9800
Pt2—S2	2.3278 (7)	C32—H32C	0.9800
S1—P1	2.0322 (10)	C4—C42	1.536 (4)
S2—P2	2.0323 (10)	C4—C41	1.537 (4)
P1—C3	1.844 (3)	C4—C43	1.539 (4)
P1—C2	1.877 (3)	C5—C51	1.528 (4)
P1—C1	1.883 (3)	C5—C52	1.535 (4)
P2—C5	1.854 (3)	C5—H6	1.0000
P2—C6	1.882 (3)	C6—C61	1.530 (4)
P2—C4	1.884 (3)	C6—C62	1.537 (4)
C1—C11	1.533 (4)	C6—C63	1.538 (4)
C1—C12	1.539 (4)	C41—H41A	0.9800
C1—C13	1.541 (4)	C41—H41B	0.9800
C2—C23	1.530 (5)	C41—H41C	0.9800

C2—C21	1.536 (4)	C42—H42A	0.9800
C2—C22	1.543 (4)	C42—H42B	0.9800
C3—C32	1.526 (4)	C42—H42C	0.9800
C3—C31	1.533 (4)	C43—H43A	0.9800
C3—H3	1.0000	C43—H43B	0.9800
C11—H11A	0.9800	C43—H43C	0.9800
C11—H11B	0.9800	C51—H51A	0.9800
C11—H11C	0.9800	C51—H51B	0.9800
C12—H12A	0.9800	C51—H51C	0.9800
C12—H12B	0.9800	C52—H52A	0.9800
C12—H12C	0.9800	C52—H52B	0.9800
C13—H13A	0.9800	C52—H52C	0.9800
C13—H13B	0.9800	C61—H61A	0.9800
C13—H13C	0.9800	C61—H61B	0.9800
C21—H21A	0.9800	C61—H61C	0.9800
C21—H21B	0.9800	C62—H62A	0.9800
C21—H21C	0.9800	C62—H62B	0.9800
C22—H22A	0.9800	C62—H62C	0.9800
C22—H22B	0.9800	C63—H63A	0.9800
C22—H22C	0.9800	C63—H63B	0.9800
C23—H23A	0.9800	C63—H63C	0.9800

Cl1—Pt1—Cl1^{i}	180.0	H23A—C23—H23C	109.5
Cl1—Pt1—S1	87.04 (2)	H23B—C23—H23C	109.5
Cl1^{i}—Pt1—S1	92.96 (2)	C3—C31—H31A	109.5
Cl1—Pt1—S1^{i}	92.96 (2)	C3—C31—H31B	109.5
Cl1^{i}—Pt1—S1^{i}	87.04 (2)	H31A—C31—H31B	109.5
S1—Pt1—S1^{i}	180.0	C3—C31—H31C	109.5
Cl2—Pt2—Cl2^{ii}	180.0	H31A—C31—H31C	109.5
Cl2—Pt2—S2^{ii}	89.24 (3)	H31B—C31—H31C	109.5
Cl2^{ii}—Pt2—S2^{ii}	90.76 (3)	C3—C32—H32A	109.5
Cl2—Pt2—S2	90.75 (3)	C3—C32—H32B	109.5
Cl2^{ii}—Pt2—S2	89.25 (3)	H32A—C32—H32B	109.5
S2^{ii}—Pt2—S2	180.0	C3—C32—H32C	109.5
P1—S1—Pt1^{i}	113.49 (3)	H32A—C32—H32C	109.5
P1—S1—Pt1	113.49 (3)	H32B—C32—H32C	109.5
P2—S2—Pt2^{ii}	113.27 (4)	C42—C4—C41	106.9 (2)
P2—S2—Pt2	113.27 (4)	C42—C4—C43	108.1 (2)
C3—P1—C2	107.91 (14)	C41—C4—C43	110.5 (2)
C3—P1—C1	112.81 (13)	C42—C4—P2	110.7 (2)
C2—P1—C1	112.08 (13)	C41—C4—P2	107.03 (19)
C3—P1—S1	108.40 (10)	C43—C4—P2	113.4 (2)
C2—P1—S1	111.07 (10)	C51—C5—C52	108.0 (3)
C1—P1—S1	104.54 (10)	C51—C5—P2	114.3 (2)
C5—P2—C6	112.01 (14)	C52—C5—P2	116.9 (2)
C5—P2—C4	107.21 (13)	C51—C5—H6	105.5
C6—P2—C4	113.18 (13)	C52—C5—H6	105.5
C5—P2—S2	110.77 (10)	P2—C5—H6	105.5

C6—P2—S2	109.37 (10)	C61—C6—C62	110.1 (3)
C4—P2—S2	104.01 (10)	C61—C6—C63	107.7 (3)
C11—C1—C12	107.4 (2)	C62—C6—C63	108.6 (3)
C11—C1—C13	110.3 (2)	C61—C6—P2	109.7 (2)
C12—C1—C13	107.7 (2)	C62—C6—P2	112.0 (2)
C11—C1—P1	110.65 (19)	C63—C6—P2	108.6 (2)
C12—C1—P1	109.38 (19)	C4—C41—H41A	109.5
C13—C1—P1	111.2 (2)	C4—C41—H41B	109.5
C23—C2—C21	108.9 (3)	H41A—C41—H41B	109.5
C23—C2—C22	107.6 (3)	C4—C41—H41C	109.5
C21—C2—C22	109.1 (3)	H41A—C41—H41C	109.5
C23—C2—P1	110.8 (2)	H41B—C41—H41C	109.5
C21—C2—P1	112.6 (2)	C4—C42—H42A	109.5
C22—C2—P1	107.7 (2)	C4—C42—H42B	109.5
C32—C3—C31	110.2 (3)	H42A—C42—H42B	109.5
C32—C3—P1	112.6 (2)	C4—C42—H42C	109.5
C31—C3—P1	118.2 (2)	H42A—C42—H42C	109.5
C32—C3—H3	104.9	H42B—C42—H42C	109.5
C31—C3—H3	104.9	C4—C43—H43A	109.5
P1—C3—H3	104.9	C4—C43—H43B	109.5
C1—C11—H11A	109.5	H43A—C43—H43B	109.5
C1—C11—H11B	109.5	C4—C43—H43C	109.5
H11A—C11—H11B	109.5	H43A—C43—H43C	109.5
C1—C11—H11C	109.5	H43B—C43—H43C	109.5
H11A—C11—H11C	109.5	C5—C51—H51A	109.5
H11B—C11—H11C	109.5	C5—C51—H51B	109.5
C1—C12—H12A	109.5	H51A—C51—H51B	109.5
C1—C12—H12B	109.5	C5—C51—H51C	109.5
H12A—C12—H12B	109.5	H51A—C51—H51C	109.5
C1—C12—H12C	109.5	H51B—C51—H51C	109.5
H12A—C12—H12C	109.5	C5—C52—H52A	109.5
H12B—C12—H12C	109.5	C5—C52—H52B	109.5
C1—C13—H13A	109.5	H52A—C52—H52B	109.5
C1—C13—H13B	109.5	C5—C52—H52C	109.5
H13A—C13—H13B	109.5	H52A—C52—H52C	109.5
C1—C13—H13C	109.5	H52B—C52—H52C	109.5
H13A—C13—H13C	109.5	C6—C61—H61A	109.5
H13B—C13—H13C	109.5	C6—C61—H61B	109.5
C2—C21—H21A	109.5	H61A—C61—H61B	109.5
C2—C21—H21B	109.5	C6—C61—H61C	109.5
H21A—C21—H21B	109.5	H61A—C61—H61C	109.5
C2—C21—H21C	109.5	H61B—C61—H61C	109.5
H21A—C21—H21C	109.5	C6—C62—H62A	109.5
H21B—C21—H21C	109.5	C6—C62—H62B	109.5
C2—C22—H22A	109.5	H62A—C62—H62B	109.5
C2—C22—H22B	109.5	C6—C62—H62C	109.5
H22A—C22—H22B	109.5	H62A—C62—H62C	109.5
C2—C22—H22C	109.5	H62B—C62—H62C	109.5

H22A—C22—H22C	109.5	C6—C63—H63A	109.5
H22B—C22—H22C	109.5	C6—C63—H63B	109.5
C2—C23—H23A	109.5	H63A—C63—H63B	109.5
C2—C23—H23B	109.5	C6—C63—H63C	109.5
H23A—C23—H23B	109.5	H63A—C63—H63C	109.5
C2—C23—H23C	109.5	H63B—C63—H63C	109.5

Cl1—Pt1—S1—P1	112.78 (4)	C1—P1—C2—C22	−165.1 (2)
Cl1^{i}—Pt1—S1—P1	−67.22 (4)	S1—P1—C2—C22	78.4 (2)
Cl2—Pt2—S2—P2	73.29 (5)	C2—P1—C3—C32	175.4 (2)
Cl2^{ii}—Pt2—S2—P2	−106.71 (5)	C1—P1—C3—C32	−60.2 (2)
Pt1^{i}—S1—P1—C3	64.50 (11)	S1—P1—C3—C32	55.1 (2)
Pt1—S1—P1—C3	64.50 (11)	C2—P1—C3—C31	−54.3 (3)
Pt1^{i}—S1—P1—C2	−53.88 (11)	C1—P1—C3—C31	70.1 (3)
Pt1—S1—P1—C2	−53.88 (11)	S1—P1—C3—C31	−174.7 (2)
Pt1^{i}—S1—P1—C1	−174.95 (9)	C5—P2—C4—C42	−158.5 (2)
Pt1—S1—P1—C1	−174.95 (9)	C6—P2—C4—C42	77.5 (2)
Pt2^{ii}—S2—P2—C5	−24.15 (12)	S2—P2—C4—C42	−41.1 (2)
Pt2—S2—P2—C5	−24.15 (12)	C5—P2—C4—C41	−42.4 (2)
Pt2^{ii}—S2—P2—C6	99.77 (11)	C6—P2—C4—C41	−166.4 (2)
Pt2—S2—P2—C6	99.77 (11)	S2—P2—C4—C41	75.0 (2)
Pt2^{ii}—S2—P2—C4	−139.04 (10)	C5—P2—C4—C43	79.8 (2)
Pt2—S2—P2—C4	−139.04 (10)	C6—P2—C4—C43	−44.2 (3)
C3—P1—C1—C11	−166.3 (2)	S2—P2—C4—C43	−162.85 (19)
C2—P1—C1—C11	−44.2 (3)	C6—P2—C5—C51	−51.5 (3)
S1—P1—C1—C11	76.2 (2)	C4—P2—C5—C51	−176.3 (2)
C3—P1—C1—C12	75.6 (2)	S2—P2—C5—C51	70.9 (2)
C2—P1—C1—C12	−162.3 (2)	C6—P2—C5—C52	76.0 (3)
S1—P1—C1—C12	−41.9 (2)	C4—P2—C5—C52	−48.7 (3)
C3—P1—C1—C13	−43.2 (2)	S2—P2—C5—C52	−161.6 (2)
C2—P1—C1—C13	78.8 (2)	C5—P2—C6—C61	−167.8 (2)
S1—P1—C1—C13	−160.78 (18)	C4—P2—C6—C61	−46.5 (3)
C3—P1—C2—C23	−157.8 (2)	S2—P2—C6—C61	69.0 (2)
C1—P1—C2—C23	77.4 (2)	C5—P2—C6—C62	−45.2 (3)
S1—P1—C2—C23	−39.1 (2)	C4—P2—C6—C62	76.1 (3)
C3—P1—C2—C21	80.1 (3)	S2—P2—C6—C62	−168.44 (19)
C1—P1—C2—C21	−44.7 (3)	C5—P2—C6—C63	74.7 (2)
S1—P1—C2—C21	−161.3 (2)	C4—P2—C6—C63	−164.0 (2)
C3—P1—C2—C22	−40.3 (2)	S2—P2—C6—C63	−48.5 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···Cl1^{i}	1.00	2.61	3.382 (3)	134
C5—H6···Cl2	1.00	2.65	3.470 (3)	140
C41—H41*A*···S2	0.98	3.01	3.541 (3)	115

C42—H42*C*···S2	0.98	2.64	3.195 (3)	116
C63—H63*B*···Cl2^{ii}	0.98	2.85	3.666 (3)	141
C63—H63*B*···S2	0.98	2.85	3.374 (3)	114
C12—H12*A*···S1	0.98	2.67	3.188 (3)	113
C23—H23*B*···Cl1	0.98	2.73	3.708 (4)	175
C23—H23*B*···S1	0.98	2.84	3.370 (3)	115
C32—H32*B*···S1	0.98	2.87	3.467 (3)	121
C22—H22*C*···Pt1^{i}	0.98	2.77	3.691 (3)	156
C51—H51*C*···Pt2^{ii}	0.98	2.64	3.475 (3)	143
C32—H32*A*···Cl2^{iii}	0.98	2.76	3.716 (3)	165
C43—H43*A*···S1^{iv}	0.98	2.98	3.779 (3)	139

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

Di-*μ*-chlorido-bis[(*tert*-butyldiisopropylphosphine sulfide-*κS*)chloridopalladium(II)] (6)

Crystal data

$[PdCl_2(C_{10}H_{23}PS)_2]$
M_r = 767.23
Triclinic, $P\overline{1}$
a = 6.9753 (4) Å
b = 8.7642 (5) Å
c = 13.0718 (7) Å
α = 88.930 (6)°
β = 78.488 (6)°
γ = 79.804 (7)°
V = 770.56 (8) Å^3
Z = 1
$F(000)$ = 388
D_x = 1.653 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 13225 reflections
θ = 2.4–30.8°
μ = 1.76 mm^{-1}
T = 100 K
Plate, dichroic yellow orange
0.17 × 0.06 × 0.02 mm

Data collection

Oxford Diffraction Xcalibur, Eos diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1419 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.754, T_{max} = 0.966
48215 measured reflections
4550 independent reflections
3905 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 30.8°, θ_{min} = 2.4°
h = −9→10
k = −12→12
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.059
S = 1.05
4550 reflections
143 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
$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + 0.6436P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.51394 (2)	0.06683 (2)	0.37430 (2)	0.01336 (5)
Cl1	0.65368 (8)	−0.04335 (6)	0.21405 (4)	0.02082 (12)
Cl2	0.39202 (8)	0.16888 (6)	0.54318 (4)	0.01794 (11)
S1	0.42186 (8)	0.30672 (6)	0.30898 (4)	0.01541 (11)
P1	0.18827 (8)	0.29587 (6)	0.23938 (4)	0.01113 (10)
C1	0.0763 (3)	0.5020 (2)	0.22052 (17)	0.0176 (4)
C2	−0.0026 (3)	0.1987 (2)	0.31853 (16)	0.0139 (4)
H2	−0.117975	0.212802	0.282397	0.017*
C3	0.2721 (3)	0.1824 (3)	0.11670 (16)	0.0165 (4)
H3	0.319466	0.074571	0.138464	0.020*
C11	0.1990 (4)	0.5677 (3)	0.12441 (19)	0.0261 (5)
H11A	0.150739	0.679261	0.120726	0.039*
H11B	0.185254	0.515820	0.061147	0.039*
H11C	0.339152	0.549753	0.130053	0.039*
C12	0.0766 (4)	0.6024 (3)	0.31521 (19)	0.0246 (5)
H12A	0.213851	0.608368	0.319203	0.037*
H12B	0.012994	0.556072	0.379020	0.037*
H12C	0.003033	0.706862	0.307981	0.037*
C13	−0.1395 (3)	0.5133 (3)	0.2055 (2)	0.0241 (5)
H13A	−0.220896	0.480355	0.269184	0.036*
H13B	−0.141633	0.446013	0.146814	0.036*
H13C	−0.192987	0.620778	0.190777	0.036*
C21	−0.0800 (3)	0.2700 (3)	0.42818 (17)	0.0210 (5)
H21A	−0.172218	0.208534	0.468672	0.032*
H21B	−0.149281	0.376538	0.423102	0.032*
H21C	0.031967	0.270646	0.462986	0.032*
C22	0.0679 (3)	0.0233 (3)	0.32575 (19)	0.0204 (5)
H22A	0.180471	0.004640	0.361567	0.031*
H22B	0.109462	−0.022640	0.255299	0.031*
H22C	−0.041055	−0.023900	0.364992	0.031*
C31	0.4524 (4)	0.2273 (3)	0.04244 (17)	0.0232 (5)
H31A	0.517504	0.140119	−0.005206	0.035*
H31B	0.546491	0.253151	0.083088	0.035*
H31C	0.408421	0.317434	0.002014	0.035*
C32	0.1047 (4)	0.1677 (3)	0.05945 (18)	0.0245 (5)
H32A	0.058487	0.268083	0.030222	0.037*
H32B	−0.005830	0.135317	0.108573	0.037*
H32C	0.154396	0.090228	0.002903	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.01149 (8)	0.01694 (8)	0.01083 (8)	−0.00036 (6)	−0.00233 (6)	0.00143 (6)
Cl1	0.0223 (3)	0.0237 (3)	0.0129 (2)	0.0047 (2)	−0.0027 (2)	−0.0012 (2)
Cl2	0.0195 (3)	0.0189 (2)	0.0127 (2)	0.0027 (2)	−0.00211 (19)	0.00072 (19)
S1	0.0153 (2)	0.0168 (2)	0.0159 (2)	−0.00509 (19)	−0.0054 (2)	0.00214 (19)
P1	0.0129 (2)	0.0110 (2)	0.0095 (2)	−0.00207 (19)	−0.00224 (19)	0.00125 (18)
C1	0.0202 (11)	0.0124 (9)	0.0198 (11)	−0.0021 (8)	−0.0039 (9)	0.0026 (8)
C2	0.0140 (9)	0.0145 (9)	0.0138 (9)	−0.0047 (8)	−0.0027 (8)	0.0035 (7)
C3	0.0194 (10)	0.0164 (10)	0.0126 (9)	−0.0011 (8)	−0.0021 (8)	−0.0016 (8)
C11	0.0326 (13)	0.0190 (11)	0.0254 (12)	−0.0050 (10)	−0.0032 (10)	0.0121 (9)
C12	0.0314 (13)	0.0139 (10)	0.0273 (12)	−0.0015 (9)	−0.0046 (10)	−0.0050 (9)
C13	0.0214 (11)	0.0178 (11)	0.0334 (13)	0.0005 (9)	−0.0101 (10)	0.0029 (10)
C21	0.0189 (11)	0.0273 (12)	0.0162 (10)	−0.0076 (9)	0.0010 (9)	0.0007 (9)
C22	0.0210 (11)	0.0157 (10)	0.0264 (12)	−0.0062 (9)	−0.0076 (9)	0.0070 (9)
C31	0.0250 (12)	0.0272 (12)	0.0140 (10)	−0.0026 (10)	0.0021 (9)	0.0013 (9)
C32	0.0259 (12)	0.0311 (13)	0.0176 (11)	−0.0040 (10)	−0.0073 (9)	−0.0046 (9)

Geometric parameters (Å, º)

Pd1—Cl1	2.2799 (6)	C11—H11C	0.9800
Pd1—S1	2.2882 (6)	C12—H12A	0.9800
Pd1—Cl2	2.3349 (5)	C12—H12B	0.9800
Pd1—Cl2^{i}	2.3623 (6)	C12—H12C	0.9800
S1—P1	2.0350 (7)	C13—H13A	0.9800
P1—C3	1.839 (2)	C13—H13B	0.9800
P1—C2	1.841 (2)	C13—H13C	0.9800
P1—C1	1.870 (2)	C21—H21A	0.9800
C1—C12	1.532 (3)	C21—H21B	0.9800
C1—C11	1.536 (3)	C21—H21C	0.9800
C1—C13	1.542 (3)	C22—H22A	0.9800
C2—C21	1.530 (3)	C22—H22B	0.9800
C2—C22	1.536 (3)	C22—H22C	0.9800
C2—H2	1.0000	C31—H31A	0.9800
C3—C32	1.531 (3)	C31—H31B	0.9800
C3—C31	1.534 (3)	C31—H31C	0.9800
C3—H3	1.0000	C32—H32A	0.9800
C11—H11A	0.9800	C32—H32B	0.9800
C11—H11B	0.9800	C32—H32C	0.9800
Cl1—Pd1—S1	93.95 (2)	H11B—C11—H11C	109.5
Cl1—Pd1—Cl2	175.53 (2)	C1—C12—H12A	109.5
S1—Pd1—Cl2	89.38 (2)	C1—C12—H12B	109.5
Cl1—Pd1—Cl2^{i}	91.03 (2)	H12A—C12—H12B	109.5
S1—Pd1—Cl2^{i}	174.586 (19)	C1—C12—H12C	109.5
Cl2—Pd1—Cl2^{i}	85.54 (2)	H12A—C12—H12C	109.5
Pd1—Cl2—Pd1^{i}	94.46 (2)	H12B—C12—H12C	109.5

P1—S1—Pd1	107.34 (3)	C1—C13—H13A	109.5
C3—P1—C2	105.26 (10)	C1—C13—H13B	109.5
C3—P1—C1	113.34 (10)	H13A—C13—H13B	109.5
C2—P1—C1	108.97 (10)	C1—C13—H13C	109.5
C3—P1—S1	110.54 (7)	H13A—C13—H13C	109.5
C2—P1—S1	113.53 (7)	H13B—C13—H13C	109.5
C1—P1—S1	105.38 (8)	C2—C21—H21A	109.5
C12—C1—C11	107.57 (19)	C2—C21—H21B	109.5
C12—C1—C13	108.94 (19)	H21A—C21—H21B	109.5
C11—C1—C13	109.29 (19)	C2—C21—H21C	109.5
C12—C1—P1	110.55 (15)	H21A—C21—H21C	109.5
C11—C1—P1	110.01 (15)	H21B—C21—H21C	109.5
C13—C1—P1	110.41 (15)	C2—C22—H22A	109.5
C21—C2—C22	109.89 (18)	C2—C22—H22B	109.5
C21—C2—P1	112.98 (15)	H22A—C22—H22B	109.5
C22—C2—P1	112.56 (15)	C2—C22—H22C	109.5
C21—C2—H2	107.0	H22A—C22—H22C	109.5
C22—C2—H2	107.0	H22B—C22—H22C	109.5
P1—C2—H2	107.0	C3—C31—H31A	109.5
C32—C3—C31	111.67 (18)	C3—C31—H31B	109.5
C32—C3—P1	113.93 (15)	H31A—C31—H31B	109.5
C31—C3—P1	115.39 (16)	C3—C31—H31C	109.5
C32—C3—H3	104.9	H31A—C31—H31C	109.5
C31—C3—H3	104.9	H31B—C31—H31C	109.5
P1—C3—H3	104.9	C3—C32—H32A	109.5
C1—C11—H11A	109.5	C3—C32—H32B	109.5
C1—C11—H11B	109.5	H32A—C32—H32B	109.5
H11A—C11—H11B	109.5	C3—C32—H32C	109.5
C1—C11—H11C	109.5	H32A—C32—H32C	109.5
H11A—C11—H11C	109.5	H32B—C32—H32C	109.5

S1—Pd1—Cl2—Pd1^{i}	−178.13 (2)	C2—P1—C1—C13	−37.80 (18)
Cl2^{i}—Pd1—Cl2—Pd1^{i}	0.0	S1—P1—C1—C13	−159.96 (14)
Cl1—Pd1—S1—P1	73.01 (3)	C3—P1—C2—C21	175.65 (16)
Cl2—Pd1—S1—P1	−109.98 (3)	C1—P1—C2—C21	−62.49 (18)
Pd1—S1—P1—C3	−72.23 (8)	S1—P1—C2—C21	54.62 (17)
Pd1—S1—P1—C2	45.78 (8)	C3—P1—C2—C22	50.45 (18)
Pd1—S1—P1—C1	164.96 (7)	C1—P1—C2—C22	172.32 (15)
C3—P1—C1—C12	−160.34 (16)	S1—P1—C2—C22	−70.58 (16)
C2—P1—C1—C12	82.83 (18)	C2—P1—C3—C32	56.71 (18)
S1—P1—C1—C12	−39.34 (17)	C1—P1—C3—C32	−62.28 (19)
C3—P1—C1—C11	−41.68 (19)	S1—P1—C3—C32	179.67 (14)
C2—P1—C1—C11	−158.51 (16)	C2—P1—C3—C31	−172.15 (16)
S1—P1—C1—C11	79.32 (16)	C1—P1—C3—C31	68.86 (19)
C3—P1—C1—C13	79.03 (18)	S1—P1—C3—C31	−49.19 (18)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···Cl1	1.00	2.74	3.466 (2)	130
C13—H13*C*···Cl1^{ii}	0.98	2.95	3.893 (2)	162
C31—H31*B*···S1	0.98	2.93	3.523 (2)	120
C32—H32*C*···Cl1^{iii}	0.98	2.89	3.728 (2)	144
C21—H21*C*···Cl2	0.98	2.90	3.844 (2)	161
C22—H22*C*···Cl2^{iv}	0.98	2.99	3.960 (2)	172
C22—H22*A*···Pd1	0.98	2.52	3.383 (2)	147
C2—H2···Pd1^{v}	1.00	3.09	3.688 (2)	120
C22—H22*C*···Pd1^{v}	0.98	3.04	3.740 (2)	129
C21—H21*A*···Pd1^{v}	0.98	3.16	3.784 (2)	123

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

(Bromosulfanyl)di-*tert*-butylisopropylphosphanium di-*μ*-bromido-bis[dibromidopalladium(II)] (7)

Crystal data

$(C_{11}H_{25}BrPS)_2[Pd_2Br_6]$
M_r = 1292.76
Monoclinic, $P2_1/c$
a = 7.8691 (4) Å
b = 22.7255 (8) Å
c = 10.5879 (3) Å
β = 98.386 (3)°
V = 1873.18 (13) Å^3
Z = 2
$F(000)$ = 1232
D_x = 2.292 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8375 reflections
θ = 2.1–29.2°
μ = 9.70 mm^{-1}
T = 100 K
Plate, red
0.2 × 0.1 × 0.01 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 (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.495, T_{max} = 1.000
52903 measured reflections
4622 independent reflections
3740 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
θ_{max} = 28.3°, θ_{min} = 2.1°
h = −10→10
k = −30→30
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.072
S = 1.11
4622 reflections
196 parameters
87 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
$w = 1/[\sigma^2(F_o^2) + (0.0177P)^2 + 6.208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.95 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br1	0.39593 (7)	0.61750 (2)	0.46899 (5)	0.02308 (12)	
P1	0.43599 (16)	0.63574 (5)	0.78836 (12)	0.0152 (3)	
S1	0.57414 (17)	0.64827 (6)	0.63578 (12)	0.0201 (3)	
Pd1	−0.00182 (5)	0.56159 (2)	0.39219 (3)	0.01429 (9)	
Br2	0.14330 (7)	0.58219 (2)	0.21071 (5)	0.02152 (12)	
Br3	0.13664 (6)	0.46495 (2)	0.42182 (5)	0.01861 (12)	
Br4	−0.14636 (7)	0.65550 (2)	0.37175 (5)	0.02252 (12)	
C1	0.6145 (6)	0.6373 (2)	0.9272 (4)	0.0195 (10)	
C11	0.5461 (9)	0.6149 (4)	1.0480 (6)	0.0291 (19)	0.806 (14)
H11A	0.516541	0.573107	1.037742	0.044*	0.806 (14)
H11B	0.443594	0.637366	1.060595	0.044*	0.806 (14)
H11C	0.634802	0.619982	1.122408	0.044*	0.806 (14)
C12	0.6801 (10)	0.7008 (3)	0.9529 (7)	0.030 (2)	0.806 (14)
H12A	0.784684	0.700238	1.015776	0.046*	0.806 (14)
H12B	0.591760	0.724207	0.986083	0.046*	0.806 (14)
H12C	0.705636	0.718364	0.873216	0.046*	0.806 (14)
C13	0.7632 (9)	0.5987 (4)	0.9015 (7)	0.028 (2)	0.806 (14)
H13A	0.849008	0.596782	0.978382	0.042*	0.806 (14)
H13B	0.815668	0.615459	0.831111	0.042*	0.806 (14)
H13C	0.720826	0.559044	0.878454	0.042*	0.806 (14)
C11′	0.749 (3)	0.6836 (11)	0.902 (3)	0.026 (8)*	0.194 (14)
H11D	0.782645	0.676778	0.817698	0.040*	0.194 (14)
H11E	0.699108	0.723087	0.904523	0.040*	0.194 (14)
H11F	0.849914	0.680470	0.967595	0.040*	0.194 (14)
C12′	0.708 (4)	0.5767 (8)	0.931 (3)	0.019 (7)*	0.194 (14)
H12D	0.748162	0.569631	0.848642	0.028*	0.194 (14)
H12E	0.806985	0.576953	0.998990	0.028*	0.194 (14)
H12F	0.628702	0.545347	0.947110	0.028*	0.194 (14)
C13′	0.556 (4)	0.6455 (14)	1.0569 (19)	0.022 (8)*	0.194 (14)
H13D	0.471188	0.615273	1.068942	0.033*	0.194 (14)
H13E	0.655300	0.641940	1.124171	0.033*	0.194 (14)
H13F	0.504494	0.684557	1.061098	0.033*	0.194 (14)
C2	0.3250 (6)	0.5645 (2)	0.7640 (5)	0.0189 (10)	
H2	0.240842	0.569285	0.684305	0.023*	
C21	0.4409 (7)	0.5136 (2)	0.7369 (6)	0.0305 (13)	
H21A	0.518108	0.503466	0.815075	0.046*	
H21B	0.508807	0.525260	0.670503	0.046*	
H21C	0.370096	0.479360	0.707790	0.046*	
C22	0.2171 (9)	0.5480 (3)	0.8679 (6)	0.0408 (16)	
H22A	0.155513	0.511174	0.844575	0.061*	
H22B	0.134199	0.579387	0.876382	0.061*	
H22C	0.292551	0.542783	0.949374	0.061*	
C3	0.2742 (7)	0.6967 (2)	0.7887 (5)	0.0249 (12)	
C31	0.1155 (13)	0.6835 (5)	0.6871 (9)	0.030 (2)	0.80 (3)
H31A	0.029423	0.714453	0.689240	0.045*	0.80 (3)

H31B	0.066127	0.645410	0.705724	0.045*	0.80 (3)
H31C	0.151004	0.682183	0.602136	0.045*	0.80 (3)
C32	0.2141 (15)	0.7059 (6)	0.9180 (7)	0.039 (3)	0.80 (3)
H32A	0.309512	0.720990	0.979274	0.059*	0.80 (3)
H32B	0.174793	0.668312	0.948854	0.059*	0.80 (3)
H32C	0.119269	0.734253	0.908965	0.059*	0.80 (3)
C33	0.3548 (12)	0.7548 (3)	0.7488 (14)	0.044 (3)	0.80 (3)
H33A	0.267982	0.786020	0.740752	0.066*	0.80 (3)
H33B	0.396150	0.749228	0.666732	0.066*	0.80 (3)
H33C	0.451165	0.765887	0.813872	0.066*	0.80 (3)
C31′	0.167 (4)	0.6839 (16)	0.897 (3)	0.020*	0.20 (3)
H31D	0.110910	0.645445	0.882746	0.030*	0.20 (3)
H31E	0.242663	0.683535	0.979350	0.030*	0.20 (3)
H31F	0.079489	0.714485	0.898118	0.030*	0.20 (3)
C32′	0.363 (4)	0.7560 (10)	0.808 (4)	0.029 (10)*	0.20 (3)
H32D	0.441494	0.755791	0.889056	0.044*	0.20 (3)
H32E	0.428090	0.763569	0.737704	0.044*	0.20 (3)
H32F	0.276691	0.786928	0.810399	0.044*	0.20 (3)
C33′	0.148 (5)	0.693 (2)	0.664 (2)	0.022 (10)*	0.20 (3)
H33D	0.094962	0.654067	0.656033	0.033*	0.20 (3)
H33E	0.059080	0.723190	0.663899	0.033*	0.20 (3)
H33F	0.210480	0.699831	0.591204	0.033*	0.20 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0307 (3)	0.0269 (3)	0.0112 (2)	−0.0003 (2)	0.0017 (2)	−0.0001 (2)
P1	0.0203 (6)	0.0153 (6)	0.0102 (6)	−0.0004 (5)	0.0028 (5)	0.0005 (5)
S1	0.0269 (7)	0.0211 (7)	0.0129 (6)	−0.0055 (5)	0.0049 (5)	−0.0007 (5)
Pd1	0.01969 (19)	0.01357 (18)	0.00967 (18)	0.00020 (14)	0.00232 (14)	−0.00032 (14)
Br2	0.0287 (3)	0.0237 (3)	0.0133 (2)	−0.0017 (2)	0.0067 (2)	0.0026 (2)
Br3	0.0251 (3)	0.0171 (2)	0.0154 (2)	0.0045 (2)	0.0088 (2)	0.00110 (19)
Br4	0.0297 (3)	0.0163 (3)	0.0210 (3)	0.0049 (2)	0.0020 (2)	0.0014 (2)
C1	0.026 (3)	0.021 (3)	0.011 (2)	−0.006 (2)	0.001 (2)	−0.0004 (19)
C11	0.029 (4)	0.042 (5)	0.015 (3)	−0.009 (4)	−0.001 (3)	0.003 (3)
C12	0.037 (4)	0.028 (4)	0.025 (4)	−0.010 (3)	0.000 (3)	−0.003 (3)
C13	0.022 (4)	0.044 (5)	0.016 (4)	0.005 (3)	−0.003 (3)	−0.004 (3)
C2	0.022 (3)	0.021 (3)	0.013 (2)	−0.006 (2)	−0.001 (2)	0.000 (2)
C21	0.034 (3)	0.016 (3)	0.039 (4)	0.000 (2)	0.000 (3)	0.002 (2)
C22	0.054 (4)	0.041 (4)	0.031 (3)	−0.024 (3)	0.016 (3)	−0.011 (3)
C3	0.028 (3)	0.024 (3)	0.023 (3)	0.010 (2)	0.004 (2)	−0.001 (2)
C31	0.036 (5)	0.032 (5)	0.021 (4)	0.013 (4)	0.004 (4)	−0.007 (4)
C32	0.045 (6)	0.049 (7)	0.023 (4)	0.019 (5)	0.003 (4)	−0.010 (4)
C33	0.071 (6)	0.017 (4)	0.046 (7)	0.007 (3)	0.014 (5)	−0.003 (4)

Geometric parameters (Å, º)

Br1—S1	2.2027 (14)	C2—C21	1.525 (7)
Br1—Br2	3.2387 (7)	C2—C22	1.531 (7)
P1—C2	1.840 (5)	C2—H2	1.0000
P1—C1	1.879 (5)	C21—H21A	0.9800
P1—C3	1.881 (5)	C21—H21B	0.9800
P1—S1	2.0941 (18)	C21—H21C	0.9800
Pd1—Br4	2.4131 (6)	C22—H22A	0.9800
Pd1—Br2	2.4199 (6)	C22—H22B	0.9800
Pd1—Br3^{i}	2.4447 (6)	C22—H22C	0.9800
Pd1—Br3	2.4514 (6)	C3—C32′	1.518 (14)
C1—C13	1.518 (7)	C3—C32	1.527 (8)
C1—C13′	1.522 (14)	C3—C33′	1.536 (14)
C1—C11′	1.542 (14)	C3—C31′	1.549 (14)
C1—C12	1.544 (7)	C3—C33	1.550 (8)
C1—C11	1.545 (7)	C3—C31	1.554 (8)
C1—C12′	1.562 (13)	C31—H31A	0.9800
C11—H11A	0.9800	C31—H31B	0.9800
C11—H11B	0.9800	C31—H31C	0.9800
C11—H11C	0.9800	C32—H32A	0.9800
C12—H12A	0.9800	C32—H32B	0.9800
C12—H12B	0.9800	C32—H32C	0.9800
C12—H12C	0.9800	C33—H33A	0.9800
C13—H13A	0.9800	C33—H33B	0.9800
C13—H13B	0.9800	C33—H33C	0.9800
C13—H13C	0.9800	C31′—H31D	0.9800
C11′—H11D	0.9800	C31′—H31E	0.9800
C11′—H11E	0.9800	C31′—H31F	0.9800
C11′—H11F	0.9800	C32′—H32D	0.9800
C12′—H12D	0.9800	C32′—H32E	0.9800
C12′—H12E	0.9800	C32′—H32F	0.9800
C12′—H12F	0.9800	C33′—H33D	0.9800
C13′—H13D	0.9800	C33′—H33E	0.9800
C13′—H13E	0.9800	C33′—H33F	0.9800
C13′—H13F	0.9800		
S1—Br1—Br2	175.04 (4)	C21—C2—P1	114.2 (4)
C2—P1—C1	114.3 (2)	C22—C2—P1	114.6 (4)
C2—P1—C3	109.9 (2)	C21—C2—H2	105.3
C1—P1—C3	114.4 (2)	C22—C2—H2	105.3
C2—P1—S1	107.50 (17)	P1—C2—H2	105.3
C1—P1—S1	100.83 (16)	C2—C21—H21A	109.5
C3—P1—S1	109.19 (17)	C2—C21—H21B	109.5
P1—S1—Br1	103.52 (7)	H21A—C21—H21B	109.5
Br4—Pd1—Br2	91.64 (2)	C2—C21—H21C	109.5
Br4—Pd1—Br3^{i}	92.07 (2)	H21A—C21—H21C	109.5
Br2—Pd1—Br3^{i}	176.25 (2)	H21B—C21—H21C	109.5

Br4—Pd1—Br3	177.07 (2)	C2—C22—H22A	109.5
Br2—Pd1—Br3	91.29 (2)	C2—C22—H22B	109.5
Br3^{i}—Pd1—Br3	85.00 (2)	H22A—C22—H22B	109.5
Pd1—Br2—Br1	71.340 (18)	C2—C22—H22C	109.5
Pd1^{i}—Br3—Pd1	95.00 (2)	H22A—C22—H22C	109.5
C13′—C1—C11′	112.2 (14)	H22B—C22—H22C	109.5
C13—C1—C12	109.0 (5)	C32′—C3—C33′	113.0 (18)
C13—C1—C11	109.1 (5)	C32′—C3—C31′	110.8 (15)
C12—C1—C11	108.1 (5)	C33′—C3—C31′	106.1 (16)
C13′—C1—C12′	106.8 (14)	C32—C3—C33	108.7 (5)
C11′—C1—C12′	105.9 (13)	C32—C3—C31	108.9 (6)
C13—C1—P1	110.9 (4)	C33—C3—C31	107.2 (6)
C13′—C1—P1	114.8 (12)	C32′—C3—P1	110.8 (15)
C11′—C1—P1	109.3 (11)	C32—C3—P1	113.6 (4)
C12—C1—P1	110.4 (4)	C33′—C3—P1	108.1 (19)
C11—C1—P1	109.3 (4)	C31′—C3—P1	107.7 (12)
C12′—C1—P1	107.4 (11)	C33—C3—P1	108.9 (4)
C1—C11—H11A	109.5	C31—C3—P1	109.4 (5)
C1—C11—H11B	109.5	C3—C31—H31A	109.5
H11A—C11—H11B	109.5	C3—C31—H31B	109.5
C1—C11—H11C	109.5	H31A—C31—H31B	109.5
H11A—C11—H11C	109.5	C3—C31—H31C	109.5
H11B—C11—H11C	109.5	H31A—C31—H31C	109.5
C1—C12—H12A	109.5	H31B—C31—H31C	109.5
C1—C12—H12B	109.5	C3—C32—H32A	109.5
H12A—C12—H12B	109.5	C3—C32—H32B	109.5
C1—C12—H12C	109.5	H32A—C32—H32B	109.5
H12A—C12—H12C	109.5	C3—C32—H32C	109.5
H12B—C12—H12C	109.5	H32A—C32—H32C	109.5
C1—C13—H13A	109.5	H32B—C32—H32C	109.5
C1—C13—H13B	109.5	C3—C33—H33A	109.5
H13A—C13—H13B	109.5	C3—C33—H33B	109.5
C1—C13—H13C	109.5	H33A—C33—H33B	109.5
H13A—C13—H13C	109.5	C3—C33—H33C	109.5
H13B—C13—H13C	109.5	H33A—C33—H33C	109.5
C1—C11′—H11D	109.5	H33B—C33—H33C	109.5
C1—C11′—H11E	109.5	C3—C31′—H31D	109.5
H11D—C11′—H11E	109.5	C3—C31′—H31E	109.5
C1—C11′—H11F	109.5	H31D—C31′—H31E	109.5
H11D—C11′—H11F	109.5	C3—C31′—H31F	109.5
H11E—C11′—H11F	109.5	H31D—C31′—H31F	109.5
C1—C12′—H12D	109.5	H31E—C31′—H31F	109.5
C1—C12′—H12E	109.5	C3—C32′—H32D	109.5
H12D—C12′—H12E	109.5	C3—C32′—H32E	109.5
C1—C12′—H12F	109.5	H32D—C32′—H32E	109.5
H12D—C12′—H12F	109.5	C3—C32′—H32F	109.5
H12E—C12′—H12F	109.5	H32D—C32′—H32F	109.5
C1—C13′—H13D	109.5	H32E—C32′—H32F	109.5

C1—C13′—H13E	109.5	C3—C33′—H33D	109.5
H13D—C13′—H13E	109.5	C3—C33′—H33E	109.5
C1—C13′—H13F	109.5	H33D—C33′—H33E	109.5
H13D—C13′—H13F	109.5	C3—C33′—H33F	109.5
H13E—C13′—H13F	109.5	H33D—C33′—H33F	109.5
C21—C2—C22	111.0 (5)	H33E—C33′—H33F	109.5
C2—P1—S1—Br1	41.39 (18)	C1—P1—C2—C21	−60.8 (4)
C1—P1—S1—Br1	161.38 (15)	C3—P1—C2—C21	169.0 (4)
C3—P1—S1—Br1	−77.83 (18)	S1—P1—C2—C21	50.2 (4)
Br4—Pd1—Br2—Br1	95.390 (19)	C1—P1—C2—C22	68.8 (5)
Br3—Pd1—Br2—Br1	−84.823 (19)	C3—P1—C2—C22	−61.4 (5)
Br2—Pd1—Br3—Pd1^{i}	179.44 (2)	S1—P1—C2—C22	179.8 (4)
Br3^{i}—Pd1—Br3—Pd1^{i}	0.0	C2—P1—C3—C32′	−179.8 (19)
C2—P1—C1—C13	68.1 (5)	C1—P1—C3—C32′	50.0 (19)
C3—P1—C1—C13	−163.9 (5)	S1—P1—C3—C32′	−62.1 (19)
S1—P1—C1—C13	−46.9 (5)	C2—P1—C3—C32	83.8 (8)
C2—P1—C1—C13′	−80.4 (14)	C1—P1—C3—C32	−46.4 (8)
C3—P1—C1—C13′	47.6 (14)	S1—P1—C3—C32	−158.5 (7)
S1—P1—C1—C13′	164.6 (14)	C2—P1—C3—C33′	−55.5 (17)
C2—P1—C1—C11′	152.6 (14)	C1—P1—C3—C33′	174.4 (17)
C3—P1—C1—C11′	−79.4 (14)	S1—P1—C3—C33′	62.2 (17)
S1—P1—C1—C11′	37.6 (14)	C2—P1—C3—C31′	58.8 (17)
C2—P1—C1—C12	−171.1 (4)	C1—P1—C3—C31′	−71.4 (17)
C3—P1—C1—C12	−43.1 (5)	S1—P1—C3—C31′	176.5 (16)
S1—P1—C1—C12	74.0 (4)	C2—P1—C3—C33	−155.0 (6)
C2—P1—C1—C11	−52.3 (5)	C1—P1—C3—C33	74.8 (7)
C3—P1—C1—C11	75.7 (5)	S1—P1—C3—C33	−37.3 (7)
S1—P1—C1—C11	−167.3 (4)	C2—P1—C3—C31	−38.2 (7)
C2—P1—C1—C12′	38.2 (13)	C1—P1—C3—C31	−168.4 (6)
C3—P1—C1—C12′	166.2 (13)	S1—P1—C3—C31	79.5 (6)
S1—P1—C1—C12′	−76.8 (13)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*C*···Br4^{ii}	0.98	3.04	4.005 (7)	167
C12—H12*C*···S1	0.98	3.03	3.547 (7)	114
C12—H12*C*···Br4^{iii}	0.98	3.10	3.689 (6)	120
C13—H13*B*···S1	0.98	2.70	3.190 (7)	111
C2—H2···Br1	1.00	2.95	3.468 (5)	113
C2—H2···Br3^{i}	1.00	3.12	3.928 (5)	139
C21—H21*B*···Br1	0.98	3.03	3.668 (6)	124
C21—H21*B*···S1	0.98	2.88	3.456 (6)	119
C21—H21*B*···Br3^{iv}	0.98	3.10	3.968 (6)	149

C31—H31*C*···Br1	0.98	2.94	3.732 (11)	139
C33—H33*B*···S1	0.98	2.73	3.298 (10)	117

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) *x*+1, *y*, *z*+1; (iii) *x*+1, −*y*+3/2, *z*+1/2; (iv) −*x*+1, −*y*+1, −*z*+1.

(Bromoselanyl)di-*tert*-butylisopropylphosphanium di-*μ*-bromido-bis[dibromidopalladium(II)] (8)

Crystal data

$(C_{11}H_{25}BrPS_2)_2[Pd_2Br_6]$
M_r = 1386.56
Monoclinic, *C*2/*c*
a = 19.3550 (6) Å
b = 14.8165 (2) Å
c = 16.3047 (5) Å
β = 125.957 (5)°
V = 3784.8 (3) Å^3
Z = 4
F(000) = 2608
D_x = 2.433 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 7808 reflections
θ = 2.6–30.8°
μ = 11.42 mm^{-1}
T = 100 K
Prism, red
0.2 × 0.06 × 0.02 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 (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.327, T_{max} = 1.000
51432 measured reflections
5622 independent reflections
4433 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 30.9°, θ_{min} = 2.6°
h = −26→27
k = −20→21
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.057
S = 1.04
5622 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0187P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.97 e Å^{-3}
$\Delta\rho_{min}$ = −1.02 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.22849 (5)	0.77872 (5)	0.24438 (6)	0.00853 (16)
Se1	0.16673 (2)	0.87965 (2)	0.11698 (2)	0.01074 (7)
Br1	0.28206 (2)	0.91565 (2)	0.11317 (2)	0.01416 (8)
C1	0.13164 (18)	0.7218 (2)	0.2202 (2)	0.0114 (6)
C11	0.1540 (2)	0.6693 (2)	0.3137 (2)	0.0195 (7)
H11A	0.171302	0.711590	0.368829	0.029*
H11B	0.201090	0.627609	0.334906	0.029*
H11C	0.104049	0.635007	0.297163	0.029*
C12	0.09563 (19)	0.6565 (2)	0.1310 (2)	0.0145 (7)

H12A	0.043484	0.628429	0.115900	0.022*
H12B	0.137963	0.609596	0.148605	0.022*
H12C	0.082397	0.689711	0.071368	0.022*
C13	0.06286 (19)	0.7918 (2)	0.1920 (2)	0.0154 (7)
H13A	0.013385	0.761667	0.182044	0.023*
H13B	0.045404	0.822396	0.129251	0.023*
H13C	0.085754	0.836281	0.246658	0.023*
C2	0.29749 (19)	0.7020 (2)	0.2326 (2)	0.0119 (6)
H2	0.351708	0.735864	0.260464	0.014*
C21	0.2632 (2)	0.6766 (2)	0.1232 (2)	0.0164 (7)
H21A	0.308543	0.647598	0.122918	0.025*
H21B	0.244019	0.731244	0.081387	0.025*
H21C	0.215056	0.634815	0.095559	0.025*
C22	0.3232 (2)	0.6161 (2)	0.2967 (2)	0.0180 (7)
H22A	0.273840	0.575778	0.266646	0.027*
H22B	0.343204	0.632082	0.365724	0.027*
H22C	0.369143	0.585433	0.298861	0.027*
C3	0.29514 (19)	0.8399 (2)	0.3681 (2)	0.0121 (6)
C31	0.2387 (2)	0.8964 (2)	0.3853 (2)	0.0167 (7)
H31A	0.205657	0.856415	0.398084	0.025*
H31B	0.199669	0.933141	0.325067	0.025*
H31C	0.274628	0.936049	0.443940	0.025*
C32	0.3559 (2)	0.9036 (2)	0.3639 (2)	0.0160 (7)
H32A	0.322466	0.947762	0.309403	0.024*
H32B	0.391368	0.868362	0.350777	0.024*
H32C	0.392649	0.935251	0.428725	0.024*
C33	0.3513 (2)	0.7756 (2)	0.4582 (2)	0.0201 (8)
H33A	0.381125	0.810013	0.521590	0.030*
H33B	0.393280	0.746527	0.451519	0.030*
H33C	0.315400	0.729465	0.458823	0.030*
Pd1	0.500000	0.86021 (2)	0.250000	0.00908 (7)
Pd2	0.500000	0.61741 (2)	0.250000	0.01122 (8)
Br2	0.44677 (2)	0.97324 (2)	0.11858 (2)	0.01413 (7)
Br3	0.45642 (2)	0.73949 (2)	0.12672 (2)	0.01349 (7)
Br4	0.45006 (2)	0.50442 (2)	0.11976 (3)	0.01982 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0104 (4)	0.0066 (4)	0.0094 (4)	0.0001 (3)	0.0062 (3)	0.0001 (3)
Se1	0.01218 (15)	0.00876 (16)	0.01024 (15)	−0.00039 (12)	0.00600 (13)	0.00079 (12)
Br1	0.01752 (16)	0.01363 (17)	0.01470 (16)	−0.00296 (13)	0.01135 (14)	−0.00010 (13)
C1	0.0094 (14)	0.0096 (16)	0.0147 (16)	−0.0003 (12)	0.0068 (13)	0.0013 (12)
C11	0.0223 (18)	0.0178 (19)	0.0210 (18)	0.0002 (14)	0.0142 (16)	0.0059 (14)
C12	0.0120 (16)	0.0117 (17)	0.0179 (17)	−0.0019 (12)	0.0077 (14)	−0.0014 (13)
C13	0.0136 (16)	0.0163 (18)	0.0155 (17)	0.0002 (13)	0.0081 (14)	0.0000 (13)
C2	0.0115 (15)	0.0105 (16)	0.0147 (16)	−0.0001 (12)	0.0083 (13)	−0.0029 (12)
C21	0.0205 (17)	0.0141 (18)	0.0214 (18)	0.0018 (13)	0.0161 (15)	−0.0031 (14)

C22	0.0181 (17)	0.0145 (18)	0.0221 (18)	0.0049 (13)	0.0123 (15)	0.0060 (14)
C3	0.0132 (16)	0.0124 (17)	0.0101 (15)	−0.0019 (12)	0.0064 (13)	−0.0018 (12)
C31	0.0175 (17)	0.0179 (19)	0.0161 (17)	−0.0039 (13)	0.0106 (15)	−0.0057 (13)
C32	0.0177 (17)	0.0160 (18)	0.0150 (17)	−0.0059 (13)	0.0101 (14)	−0.0061 (13)
C33	0.0200 (18)	0.022 (2)	0.0098 (16)	0.0001 (14)	0.0042 (14)	0.0014 (14)
Pd1	0.01055 (16)	0.00756 (17)	0.00919 (16)	0.000	0.00583 (14)	0.000
Pd2	0.01087 (16)	0.00836 (17)	0.01348 (17)	0.000	0.00661 (14)	0.000
Br2	0.01631 (16)	0.01205 (17)	0.01485 (16)	0.00145 (12)	0.00962 (13)	0.00405 (12)
Br3	0.01799 (16)	0.01008 (16)	0.01034 (15)	−0.00096 (12)	0.00716 (13)	−0.00139 (12)
Br4	0.02289 (18)	0.01048 (17)	0.01998 (18)	0.00065 (13)	0.00918 (15)	−0.00316 (13)

Geometric parameters (Å, º)

P1—C2	1.848 (3)	C22—H22A	0.9800
P1—C3	1.870 (3)	C22—H22B	0.9800
P1—C1	1.875 (3)	C22—H22C	0.9800
P1—Se1	2.2505 (8)	C3—C31	1.528 (4)
Se1—Br1	2.3310 (4)	C3—C33	1.540 (4)
Br1—Br2	3.2510 (5)	C3—C32	1.542 (4)
C1—C11	1.529 (4)	C31—H31A	0.9800
C1—C13	1.530 (4)	C31—H31B	0.9800
C1—C12	1.532 (4)	C31—H31C	0.9800
C11—H11A	0.9800	C32—H32A	0.9800
C11—H11B	0.9800	C32—H32B	0.9800
C11—H11C	0.9800	C32—H32C	0.9800
C12—H12A	0.9800	C33—H33A	0.9800
C12—H12B	0.9800	C33—H33B	0.9800
C12—H12C	0.9800	C33—H33C	0.9800
C13—H13A	0.9800	Pd1—Br2	2.4218 (4)
C13—H13B	0.9800	Pd1—Br2^{i}	2.4218 (4)
C13—H13C	0.9800	Pd1—Br3	2.4413 (4)
C2—C22	1.533 (4)	Pd1—Br3^{i}	2.4413 (4)
C2—C21	1.541 (4)	Pd2—Br4^{i}	2.4157 (4)
C2—H2	1.0000	Pd2—Br4	2.4157 (4)
C21—H21A	0.9800	Pd2—Br3^{i}	2.4562 (4)
C21—H21B	0.9800	Pd2—Br3	2.4562 (4)
C21—H21C	0.9800		

C2—P1—C3	109.11 (14)	C2—C22—H22A	109.5
C2—P1—C1	113.31 (14)	C2—C22—H22B	109.5
C3—P1—C1	114.58 (14)	H22A—C22—H22B	109.5
C2—P1—Se1	109.72 (10)	C2—C22—H22C	109.5
C3—P1—Se1	109.24 (10)	H22A—C22—H22C	109.5
C1—P1—Se1	100.47 (10)	H22B—C22—H22C	109.5
P1—Se1—Br1	100.30 (2)	C31—C3—C33	110.1 (3)
Se1—Br1—Br2	176.810 (16)	C31—C3—C32	108.6 (3)
C11—C1—C13	109.4 (3)	C33—C3—C32	106.9 (3)
C11—C1—C12	109.3 (3)	C31—C3—P1	110.4 (2)

C13—C1—C12	108.2 (2)	C33—C3—P1	112.5 (2)
C11—C1—P1	110.9 (2)	C32—C3—P1	108.0 (2)
C13—C1—P1	110.1 (2)	C3—C31—H31A	109.5
C12—C1—P1	108.9 (2)	C3—C31—H31B	109.5
C1—C11—H11A	109.5	H31A—C31—H31B	109.5
C1—C11—H11B	109.5	C3—C31—H31C	109.5
H11A—C11—H11B	109.5	H31A—C31—H31C	109.5
C1—C11—H11C	109.5	H31B—C31—H31C	109.5
H11A—C11—H11C	109.5	C3—C32—H32A	109.5
H11B—C11—H11C	109.5	C3—C32—H32B	109.5
C1—C12—H12A	109.5	H32A—C32—H32B	109.5
C1—C12—H12B	109.5	C3—C32—H32C	109.5
H12A—C12—H12B	109.5	H32A—C32—H32C	109.5
C1—C12—H12C	109.5	H32B—C32—H32C	109.5
H12A—C12—H12C	109.5	C3—C33—H33A	109.5
H12B—C12—H12C	109.5	C3—C33—H33B	109.5
C1—C13—H13A	109.5	H33A—C33—H33B	109.5
C1—C13—H13B	109.5	C3—C33—H33C	109.5
H13A—C13—H13B	109.5	H33A—C33—H33C	109.5
C1—C13—H13C	109.5	H33B—C33—H33C	109.5
H13A—C13—H13C	109.5	Br2—Pd1—Br2^{i}	92.495 (19)
H13B—C13—H13C	109.5	Br2—Pd1—Br3	90.936 (11)
C22—C2—C21	109.5 (3)	Br2^{i}—Pd1—Br3	175.531 (13)
C22—C2—P1	113.9 (2)	Br2—Pd1—Br3^{i}	175.531 (13)
C21—C2—P1	115.1 (2)	Br2^{i}—Pd1—Br3^{i}	90.936 (11)
C22—C2—H2	105.8	Br3—Pd1—Br3^{i}	85.785 (18)
C21—C2—H2	105.8	Br4^{i}—Pd2—Br4	92.26 (2)
P1—C2—H2	105.8	Br4^{i}—Pd2—Br3^{i}	91.326 (11)
C2—C21—H21A	109.5	Br4—Pd2—Br3^{i}	176.053 (14)
C2—C21—H21B	109.5	Br4^{i}—Pd2—Br3	176.054 (14)
H21A—C21—H21B	109.5	Br4—Pd2—Br3	91.324 (11)
C2—C21—H21C	109.5	Br3^{i}—Pd2—Br3	85.139 (18)
H21A—C21—H21C	109.5	Pd1—Br2—Br1	75.133 (10)
H21B—C21—H21C	109.5	Pd1—Br3—Pd2	94.538 (13)

C2—P1—Se1—Br1	−42.39 (11)	Se1—P1—C2—C22	−164.30 (19)
C3—P1—Se1—Br1	77.19 (11)	C3—P1—C2—C21	−156.3 (2)
C1—P1—Se1—Br1	−161.98 (10)	C1—P1—C2—C21	74.7 (3)
C2—P1—C1—C11	80.0 (2)	Se1—P1—C2—C21	−36.6 (2)
C3—P1—C1—C11	−46.1 (3)	C2—P1—C3—C31	−174.0 (2)
Se1—P1—C1—C11	−163.1 (2)	C1—P1—C3—C31	−45.8 (3)
C2—P1—C1—C13	−158.8 (2)	Se1—P1—C3—C31	66.0 (2)
C3—P1—C1—C13	75.1 (2)	C2—P1—C3—C33	−50.5 (3)
Se1—P1—C1—C13	−41.9 (2)	C1—P1—C3—C33	77.8 (3)
C2—P1—C1—C12	−40.3 (2)	Se1—P1—C3—C33	−170.4 (2)
C3—P1—C1—C12	−166.4 (2)	C2—P1—C3—C32	67.3 (2)
Se1—P1—C1—C12	76.6 (2)	C1—P1—C3—C32	−164.4 (2)

C3—P1—C2—C22	76.0 (3)	Se1—P1—C3—C32	−52.6 (2)
C1—P1—C2—C22	−52.9 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*C*···Se1	0.98	3.12	3.640 (3)	115
C13—H13*B*···Se1	0.98	2.61	3.181 (3)	117
C21—H21*A*···Br3	0.98	3.14	3.822 (3)	128
C21—H21*B*···Se1	0.98	2.90	3.511 (3)	121
C21—H21*B*···Br1	0.98	2.80	3.574 (3)	137
C31—H31*B*···Se1	0.98	3.16	3.740 (3)	119
C32—H32*A*···Se1	0.98	2.97	3.526 (3)	117
C32—H32*A*···Br1	0.98	2.85	3.452 (3)	120
C12—H12*A*···Br2^{ii}	0.98	2.98	3.877 (3)	152
C12—H12*C*···Br3^{iii}	0.98	3.04	4.009 (3)	170
C2—H2···Br3^{i}	1.00	3.03	3.928 (3)	151
C32—H32*C*···Br2^{iv}	0.98	2.95	3.885 (3)	160

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2; (ii) *x*−1/2, *y*−1/2, *z*; (iii) −*x*+1/2, −*y*+3/2, −*z*; (iv) *x*, −*y*+2, *z*+1/2.

Bis[dimethyl(sulfanylidene)phosphinito-κ*Se*]\ bis(hydroxydiisopropylphosphine selenide-κ*Se*)palladium(II) (9)

Crystal data

$[Pd(C_6H_{14}OP)_2(C_6H_{15}OP)_2]$
M_r = 956.82
Monoclinic, $P2_1/n$
a = 7.56435 (6) Å
b = 10.09140 (9) Å
c = 24.13960 (19) Å
β = 92.7641 (8)°
V = 1840.55 (3) Å^3
Z = 2
F(000) = 952
D_x = 1.726 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 28620 reflections
θ = 2.2–30.8°
μ = 4.66 mm^{-1}
T = 100 K
Plate, dichroic orange yellow
0.10 × 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 (CrysAlisPro; Rigaku OD, 2012)
T_{min} = 0.650, T_{max} = 1.000
86102 measured reflections
5558 independent reflections
4939 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 30.9°, θ_{min} = 2.2°
h = −10→10
k = −14→14
l = −34→34

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.041
S = 1.07
5558 reflections
185 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0132P)^2 + 1.1714P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Pd1	0.500000	0.500000	0.000000	0.01153 (4)	
Se1	0.57000 (2)	0.38861 (2)	0.08922 (2)	0.01535 (4)	
Se2	0.41580 (2)	0.71744 (2)	0.03720 (2)	0.01366 (4)	
P1	0.43798 (6)	0.47524 (4)	0.15938 (2)	0.01456 (8)	
P2	0.65706 (5)	0.79940 (4)	0.07727 (2)	0.01312 (8)	
O1	0.49525 (18)	0.61413 (12)	0.17883 (5)	0.0217 (3)	
H01	0.569 (4)	0.653 (3)	0.1640 (15)	0.012 (10)*	0.5
O2	0.72157 (16)	0.73009 (12)	0.13081 (5)	0.0173 (2)	
H02	0.651 (5)	0.686 (4)	0.1445 (17)	0.026 (12)*	0.5
C1	0.4898 (2)	0.36446 (17)	0.21777 (7)	0.0185 (3)	
H1	0.444759	0.273758	0.208155	0.022*	
C2	0.1998 (2)	0.47669 (19)	0.14275 (8)	0.0233 (4)	
H2	0.184451	0.507699	0.103463	0.028*	
C3	0.8443 (2)	0.80181 (17)	0.03261 (7)	0.0183 (3)	
H3	0.944534	0.845492	0.054113	0.022*	
C4	0.6011 (2)	0.97035 (16)	0.09352 (7)	0.0165 (3)	
H4	0.560610	1.016268	0.058455	0.020*	
C11	0.6902 (2)	0.3574 (2)	0.22936 (8)	0.0276 (4)	
H11A	0.736533	0.446633	0.236850	0.041*	
H11B	0.746032	0.320366	0.196994	0.041*	
H11C	0.716396	0.300656	0.261670	0.041*	
C12	0.3978 (3)	0.4140 (2)	0.26924 (7)	0.0251 (4)	
H12A	0.444268	0.365769	0.302042	0.038*	
H12B	0.270059	0.398763	0.264315	0.038*	
H12C	0.420437	0.508956	0.274180	0.038*	
C21	0.1008 (3)	0.5759 (2)	0.17752 (10)	0.0403 (5)	
H21A	−0.020928	0.585362	0.162203	0.061*	
H21B	0.160572	0.661983	0.176856	0.061*	
H21C	0.099058	0.544047	0.215846	0.061*	
C22	0.1194 (2)	0.3383 (2)	0.14410 (8)	0.0287 (4)	
H22A	0.116893	0.307857	0.182601	0.043*	
H22B	0.191026	0.277268	0.122951	0.043*	
H22C	−0.001539	0.340790	0.127601	0.043*	
C31	0.8062 (2)	0.88557 (19)	−0.01933 (7)	0.0233 (4)	
H31A	0.911658	0.888141	−0.041347	0.035*	
H31B	0.774974	0.975846	−0.008477	0.035*	
H31C	0.707527	0.846427	−0.041469	0.035*	
C32	0.9062 (2)	0.66192 (19)	0.01842 (8)	0.0224 (4)	
H32A	0.817095	0.619247	−0.006420	0.034*	

H32B	0.922430	0.609846	0.052553	0.034*
H32C	1.018726	0.667126	0.000034	0.034*
C41	0.4514 (2)	0.97368 (18)	0.13373 (8)	0.0231 (4)
H41A	0.488956	0.927620	0.168046	0.035*
H41B	0.346770	0.929646	0.116767	0.035*
H41C	0.422435	1.065926	0.142169	0.035*
C42	0.7641 (2)	1.04303 (18)	0.11869 (8)	0.0248 (4)
H42A	0.731021	1.133533	0.128663	0.037*
H42B	0.856172	1.046052	0.091527	0.037*
H42C	0.809062	0.995924	0.151958	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.01300 (8)	0.01194 (8)	0.00948 (8)	−0.00030 (6)	−0.00123 (6)	0.00009 (6)
Se1	0.01935 (8)	0.01597 (8)	0.01067 (8)	0.00335 (6)	0.00021 (6)	0.00170 (6)
Se2	0.01356 (7)	0.01384 (8)	0.01338 (8)	0.00039 (6)	−0.00162 (6)	−0.00140 (6)
P1	0.01670 (19)	0.0150 (2)	0.01200 (19)	0.00073 (16)	0.00040 (15)	0.00122 (16)
P2	0.01415 (19)	0.01285 (19)	0.01223 (19)	−0.00036 (15)	−0.00065 (15)	−0.00012 (15)
O1	0.0318 (7)	0.0181 (6)	0.0155 (6)	−0.0043 (5)	0.0057 (5)	−0.0010 (5)
O2	0.0192 (6)	0.0159 (6)	0.0164 (6)	−0.0012 (5)	−0.0035 (5)	0.0032 (5)
C1	0.0223 (8)	0.0198 (8)	0.0134 (8)	0.0003 (7)	0.0018 (6)	0.0035 (6)
C2	0.0184 (8)	0.0296 (10)	0.0216 (9)	0.0059 (7)	−0.0011 (7)	−0.0001 (8)
C3	0.0144 (7)	0.0222 (9)	0.0183 (8)	−0.0026 (6)	−0.0003 (6)	−0.0024 (7)
C4	0.0209 (8)	0.0127 (8)	0.0157 (8)	0.0006 (6)	0.0002 (6)	0.0006 (6)
C11	0.0257 (9)	0.0415 (12)	0.0155 (9)	0.0067 (8)	−0.0017 (7)	0.0058 (8)
C12	0.0282 (9)	0.0324 (11)	0.0149 (8)	−0.0012 (8)	0.0048 (7)	0.0030 (8)
C21	0.0283 (11)	0.0401 (13)	0.0526 (14)	0.0160 (10)	0.0014 (10)	−0.0102 (11)
C22	0.0185 (9)	0.0387 (11)	0.0290 (10)	−0.0051 (8)	0.0022 (7)	−0.0067 (9)
C31	0.0249 (9)	0.0251 (9)	0.0203 (9)	−0.0038 (7)	0.0058 (7)	0.0011 (7)
C32	0.0153 (8)	0.0265 (9)	0.0255 (9)	0.0027 (7)	0.0005 (7)	−0.0061 (8)
C41	0.0270 (9)	0.0212 (9)	0.0215 (9)	0.0032 (7)	0.0055 (7)	−0.0036 (7)
C42	0.0278 (9)	0.0164 (9)	0.0299 (10)	−0.0041 (7)	−0.0030 (8)	−0.0042 (7)

Geometric parameters (Å, º)

Pd1—Se1	2.4642 (2)	C4—H4	1.0000
Pd1—Se1^{i}	2.4642 (2)	C11—H11A	0.9800
Pd1—Se2^{i}	2.4662 (2)	C11—H11B	0.9800
Pd1—Se2	2.4662 (2)	C11—H11C	0.9800
Se1—P1	2.1894 (5)	C12—H12A	0.9800
Se2—P2	2.1863 (4)	C12—H12B	0.9800
P1—O1	1.5340 (13)	C12—H12C	0.9800
P1—C1	1.8267 (17)	C21—H21A	0.9800
P1—C2	1.8272 (18)	C21—H21B	0.9800
P2—O2	1.5287 (12)	C21—H21C	0.9800
P2—C3	1.8213 (17)	C22—H22A	0.9800
P2—C4	1.8234 (17)	C22—H22B	0.9800

O1—H01	0.782 (18)	C22—H22C	0.9800
O2—H02	0.778 (19)	C31—H31A	0.9800
C1—C11	1.530 (2)	C31—H31B	0.9800
C1—C12	1.536 (2)	C31—H31C	0.9800
C1—H1	1.0000	C32—H32A	0.9800
C2—C22	1.524 (3)	C32—H32B	0.9800
C2—C21	1.526 (3)	C32—H32C	0.9800
C2—H2	1.0000	C41—H41A	0.9800
C3—C31	1.528 (2)	C41—H41B	0.9800
C3—C32	1.531 (2)	C41—H41C	0.9800
C3—H3	1.0000	C42—H42A	0.9800
C4—C41	1.527 (2)	C42—H42B	0.9800
C4—C42	1.534 (2)	C42—H42C	0.9800

Se1—Pd1—Se1^{i}	180.0	C1—C11—H11B	109.5
Se1—Pd1—Se2^{i}	82.184 (5)	H11A—C11—H11B	109.5
Se1^{i}—Pd1—Se2^{i}	97.816 (5)	C1—C11—H11C	109.5
Se1—Pd1—Se2	97.817 (5)	H11A—C11—H11C	109.5
Se1^{i}—Pd1—Se2	82.183 (5)	H11B—C11—H11C	109.5
Se2^{i}—Pd1—Se2	180.0	C1—C12—H12A	109.5
P1—Se1—Pd1	114.028 (13)	C1—C12—H12B	109.5
P2—Se2—Pd1	105.840 (13)	H12A—C12—H12B	109.5
O1—P1—C1	106.03 (8)	C1—C12—H12C	109.5
O1—P1—C2	108.72 (8)	H12A—C12—H12C	109.5
C1—P1—C2	110.31 (8)	H12B—C12—H12C	109.5
O1—P1—Se1	117.97 (5)	C2—C21—H21A	109.5
C1—P1—Se1	105.29 (6)	C2—C21—H21B	109.5
C2—P1—Se1	108.36 (6)	H21A—C21—H21B	109.5
O2—P2—C3	106.31 (7)	C2—C21—H21C	109.5
O2—P2—C4	108.60 (7)	H21A—C21—H21C	109.5
C3—P2—C4	108.05 (8)	H21B—C21—H21C	109.5
O2—P2—Se2	115.25 (5)	C2—C22—H22A	109.5
C3—P2—Se2	113.56 (6)	C2—C22—H22B	109.5
C4—P2—Se2	104.81 (6)	H22A—C22—H22B	109.5
P1—O1—H01	121 (3)	C2—C22—H22C	109.5
P2—O2—H02	115 (3)	H22A—C22—H22C	109.5
C11—C1—C12	110.52 (15)	H22B—C22—H22C	109.5
C11—C1—P1	110.08 (12)	C3—C31—H31A	109.5
C12—C1—P1	109.65 (12)	C3—C31—H31B	109.5
C11—C1—H1	108.8	H31A—C31—H31B	109.5
C12—C1—H1	108.8	C3—C31—H31C	109.5
P1—C1—H1	108.8	H31A—C31—H31C	109.5
C22—C2—C21	112.48 (17)	H31B—C31—H31C	109.5
C22—C2—P1	112.23 (13)	C3—C32—H32A	109.5
C21—C2—P1	112.91 (14)	C3—C32—H32B	109.5
C22—C2—H2	106.2	H32A—C32—H32B	109.5
C21—C2—H2	106.2	C3—C32—H32C	109.5
P1—C2—H2	106.2	H32A—C32—H32C	109.5

C31—C3—C32	111.96 (15)	H32B—C32—H32C	109.5
C31—C3—P2	111.82 (12)	C4—C41—H41A	109.5
C32—C3—P2	112.02 (12)	C4—C41—H41B	109.5
C31—C3—H3	106.9	H41A—C41—H41B	109.5
C32—C3—H3	106.9	C4—C41—H41C	109.5
P2—C3—H3	106.9	H41A—C41—H41C	109.5
C41—C4—C42	110.11 (15)	H41B—C41—H41C	109.5
C41—C4—P2	110.15 (12)	C4—C42—H42A	109.5
C42—C4—P2	110.32 (12)	C4—C42—H42B	109.5
C41—C4—H4	108.7	H42A—C42—H42B	109.5
C42—C4—H4	108.7	C4—C42—H42C	109.5
P2—C4—H4	108.7	H42A—C42—H42C	109.5
C1—C11—H11A	109.5	H42B—C42—H42C	109.5

Se2^{i}—Pd1—Se1—P1	−156.179 (15)	C1—P1—C2—C22	−42.75 (16)
Se2—Pd1—Se1—P1	23.820 (15)	Se1—P1—C2—C22	72.03 (14)
Se1—Pd1—Se2—P2	65.789 (13)	O1—P1—C2—C21	−30.21 (17)
Se1^{i}—Pd1—Se2—P2	−114.210 (13)	C1—P1—C2—C21	85.66 (17)
Pd1—Se1—P1—O1	−68.57 (6)	Se1—P1—C2—C21	−159.57 (14)
Pd1—Se1—P1—C1	173.44 (6)	O2—P2—C3—C31	−172.02 (12)
Pd1—Se1—P1—C2	55.41 (7)	C4—P2—C3—C31	−55.61 (14)
Pd1—Se2—P2—O2	−70.31 (6)	Se2—P2—C3—C31	60.20 (13)
Pd1—Se2—P2—C3	52.68 (6)	O2—P2—C3—C32	61.36 (13)
Pd1—Se2—P2—C4	170.38 (6)	C4—P2—C3—C32	177.78 (12)
O1—P1—C1—C11	−65.74 (14)	Se2—P2—C3—C32	−66.41 (13)
C2—P1—C1—C11	176.72 (13)	O2—P2—C4—C41	−61.77 (13)
Se1—P1—C1—C11	60.02 (13)	C3—P2—C4—C41	−176.69 (12)
O1—P1—C1—C12	56.06 (14)	Se2—P2—C4—C41	61.91 (12)
C2—P1—C1—C12	−61.48 (15)	O2—P2—C4—C42	60.00 (14)
Se1—P1—C1—C12	−178.17 (11)	C3—P2—C4—C42	−54.92 (14)
O1—P1—C2—C22	−158.62 (13)	Se2—P2—C4—C42	−176.32 (11)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H01···O2	0.78 (2)	1.63 (2)	2.4156 (17)	178 (4)
O2—H02···O1	0.78 (2)	1.64 (2)	2.4156 (17)	172 (5)
C11—H11*B*···Se1	0.98	2.95	3.4748 (18)	115
C21—H21*A*···O2^{ii}	0.98	2.52	3.407 (2)	150
C32—H32*C*···Se2^{iii}	0.98	3.13	3.8999 (17)	136
C41—H41*B*···Se2	0.98	2.94	3.4825 (18)	116
C42—H42*A*···Se1^{iv}	0.98	2.98	3.8370 (19)	146
C32—H32*A*···Pd1	0.98	2.69	3.4897 (18)	139

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