# research communications

## Crystal structures of 9-[bis(benzylsulfanyl)methyl]anthracene and of *cyclo*-dodecakis($\mu_2$-phenylmethanethiolato-$\kappa^2S$:$S$)hexapalladium(6 *Pd*—*Pd*)–anthracene-9,10-dione (1/1)

**Abhinav Raghuvanshi,**[a]‡ **Anna Krupp,**[b] **Lydie Viau,**[a] **Michael Knorr**[a]* and **Carsten Strohmann**[b]*

[a]Institut UTINAM UMR 6213 CNRS, Université Bourgogne Franche-Comté, 16, Route de Gray, 25030 Besançon, France, and [b]Anorganische Chemie, TU Dortmund University, Otto-Hahn-Str. 6, D-44227 Dortmund, Germany. *Correspondence e-mail: michael.knorr@univ-fcomte.fr, carsten.strohmann@tu-dortmund.de

The first title compound, $C_{29}H_{24}S_2$, **L1**, represents an example of an anthracene-based functionalized dithioether, which may be useful as a potential chelating or terminal ligand for coordination chemistry. This dithioacetal **L1** crystallizes in the monoclinic space group $P2_1/c$. The phenyl rings of the benzyl groups and that of the anthracene unit form dihedral angles of 49.21 (4) and 58.79 (5)° and the crystal structure displays short C–H⋯$\pi$ contacts. Surprisingly, when attempting to coordinate **L1** to [PdCl$_2$(PhCN)$_2$], instead of the targeted chelate complex [PdCl$_2$($\kappa^2$-**L1**)], a cleavage reaction leads to the formation of the centrosymmetric hexanuclear cyclic cluster of composition [Pd$_6$($\mu_2$-SCH$_2$Ph)$_{12}$] **Pd6**, or [Pd$_6$(C$_7$H$_7$S)$_{12}$]·C$_{14}$H$_8$O$_2$. This tiara-shaped hexamer crystallizing in the triclinic space group $P\bar{1}$ consists of six approximately square planar Pd(II)S$_4$ centers, which are interconnected through twelve $\mu_2$-bridging benzyl thiolate groups. The Pd⋯Pd contacts range from 3.0892 (2) to 3.1609 (2) Å and can be considered as weakly bonding. The unit cell of **Pd6** contains also a co-crystallized anthracene-9,10-dione molecule.

## 1. Chemical context

Acyclic and cyclic thioacetals with the –S–C($R$)(H)–S ($R$ = H, alkyl, aryl) unit can either be synthesized by nucleophilic substitution of geminal dihalides $X$–C($R$)(H)–$X$ by thiolates $R$S$^-$ (Murray *et al.*, 1981) or by reaction of aldehydes and ketones with thiols and dithiols (Shaterian *et al.*, 2011). Because of their soft nature, organosulfur compounds preferentially interact with late transition metals in lower oxidation states. A variety of complexes as well as coordination polymers (CPs) of varying dimensionality, ranging from zero-dimensional (molecular) to three-dimensional, have been synthesized using these types of dithioether ligands and structurally characterized (Knaust & Keller, 2003; Awaleh *et al.*, 2005, 2008). However, many factors including the structural characteristics of the organic ligands, temperature, solvent, molar ratio, *etc.*, greatly influence the formation of the resulting materials.

Over the last few years, we have been engaged in exploring the assembly of molecular cluster compounds and coordination polymers using thioether ligands RSCH$_2$SR (Peindy *et al.*, 2007; Knorr *et al.*, 2014; Schlachter *et al.*, 2020). Recently, we have also reported the synthesis of Cu$^I$ coordination complexes ligated with cyclic thioacetal ligands bearing various substituents (Raghuvanshi *et al.*, 2017, 2019;
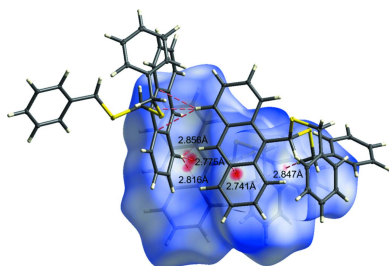
Schlachter *et al.*, 2018; Knauer *et al.*, 2020). Convenient synthetic protocols and interesting luminescent properties displayed by these complexes intrigued us to explore this field further.

Since the presence of an anthracene unit provides both rigidity as well as interesting luminescent properties to a given system, a large number of anthracene-based MOFs and CPs have been reported for various applications (for example: Hu *et al.*, 2020; Mohanty *et al.*, 2020; Quah *et al.*, 2016; Wang *et al.*, 2016). In most of these reports, either *N*- or *O*-donor substituents attached to the anthracene scaffold have been used as coordinating sites. In contrast, there are few reports where anthracene-based thioether ligands have been used for the construction of CPs. For example, a series of emissive molecular compounds and CPs have been assembled by reaction of 9,10-bis[(alkylthio)methyl]anthracenes with Ag$^I$ salts (Hu *et al.*, 2006). The synthesis of anthracene-based thioacetals with different –S*R* substituents including **L1** has been briefly reported (Goswami *et al.*, 2008 and Shaterian *et al.*, 2011). However, no spectroscopic characterization data have been communicated. Furthermore, no examples of structurally characterized anthracene-based thioacetals could be found within the Cambridge Structural Database. These disparities make this field interesting for further investigations.
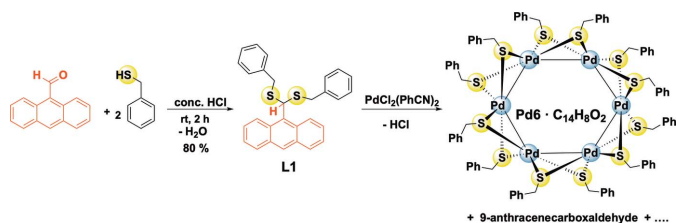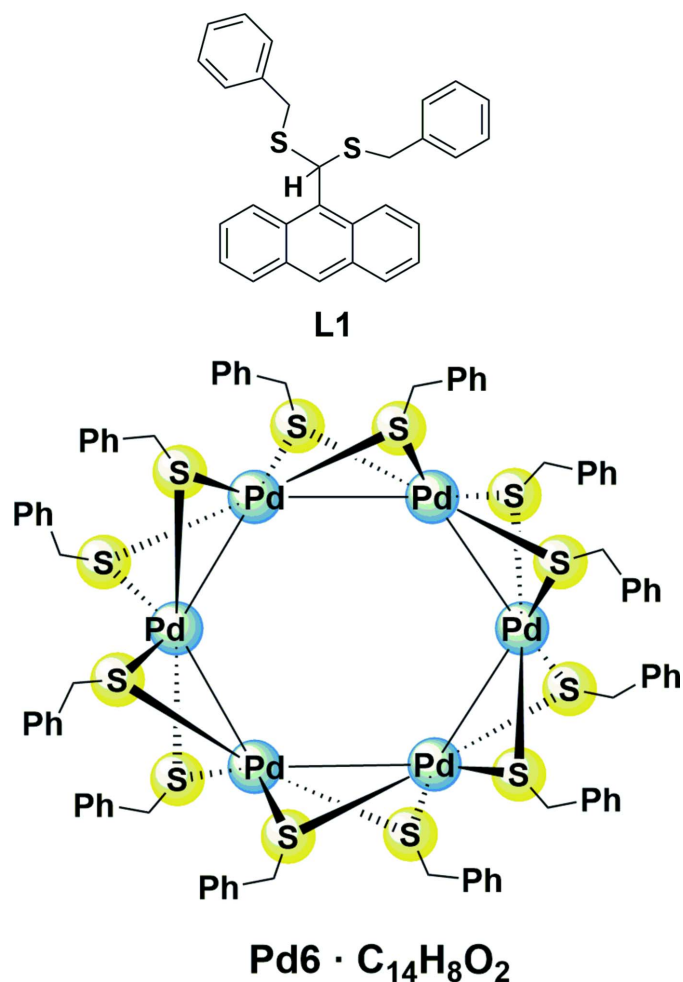


**Figure 1**
Synthesis scheme for **L1** and the cluster **Pd6·C$_{14}$H$_8$O$_2$**

In this context, we synthesized the anthracene thioacetal **L1** with the objective of using it as an *S*-donor ligand for the assembly of potentially luminescent coordination compounds. **L1** was prepared straightforwardly by the reaction of benzyl mercaptan and 9-anthracenecarboxaldehyde in the presence of an excess conc. HCl at room temperature (Fig. 1) and obtained in 80% yield as a yellow solid. Characteristic for its $^1$H NMR spectrum are two doublets at $\delta$ 3.55 and 3.79 ppm for the diastereotopic methylene protons and a singlet at $\delta$ 5.94 ppm for the methine proton. The complete spectroscopic data are reported in the *Synthesis and crystallization* section.

With this starting material in hand, we attempted to ligate **L1** to [PdCl$_2$(PhCN)$_2$], (Fig. 1). Although the coordination chemistry of [PdCl$_2$(S∩S)] compounds is dominated by chelate complexes in which open-chain dithioether or macrocyclic polythioether ligands form five- or six-membered rings such as [PdCl$_2$(1,2-bis(phenylthio)ethane-*S*,*S'*] (Rao *et al.*, 2015; Cambridge Structural Database refcode: CEYBUD01) or [PdCl$_2$(1,4,7-trithiacyclononane-*S*,*S'*)] (GATLES; Blake *et al.*, 1988), there is just one structurally characterized example of a chelate complex [PdCl$_2$(1,3,5,7-tetramethyl-2,4,6,8,9,10-hexathia-adamantane-*S*$^4$,*S*$^6$)], in which the thiamacrocycle forms a strained four-membered chelate ring (DOCNOY; Pickardt & Rautenberg, 1986). It has also been reported that upon treatment of PhSCH$_2$SPh with [*M*(MeCN)$_4$][ClO$_4$]$_2$, the strained chelate complexes [*M*(PhSCH$_2$SPh)$_4$](ClO$_4$)$_2$ (*M* = Pd, Pt) are formed (Murray *et al.*, 1981). However, to our surprise, the targeted compound [PdCl$_2$(anthracen-9-ylmethylene)bis(benzylsulfane)-*S*,*S'*)] was not formed according to the NMR data. Instead, a crystallographic study of a yellow–orange crystal revealed the formation of a cyclic hexanuclear thiolate-bridged cluster [Pd$_6$($\mu_2$-SCH$_2$Ph)$_{12}$], **Pd6**. It is well known that thioacetals can be cleaved by soft Hg$^{II}$ ions yielding aldehydes or other oxygenated products. One example is the Hg$^{II}$-promoted deprotection of 3,5-bis(dithioacetal)BODIPYs, in which cleavage of a dithioacetal function to aldehyde groups occurs (Madhu *et al.*, 2014). A mild quantitative AgNO$_3$-promoted cleavage of fluorenenylethanediyl-*S*,*S*-acetals with trichloro-isocyanuric acid yielding 9-fluorenone has also been reported (Olah *et al.*, 1980). We suppose that in our case PdCl$_2$ behaves similarly, acting as electrophilic agent. We have not examined the mechanistic aspects of this unexpected reaction in detail, but the fact that **Pd6** co-crystallizes with one molecule of anthracene-9,10-dione and smaller amounts of 9-anthraldehyde is in line with this hypothesis. It is noteworthy that this diketone has also been detected as one of the numerous



**L1**

**Pd6 · C$_{14}$H$_8$O$_2$**

| S1—C15 | 1.8309 (12) | S2—C15 | 1.8220 (12) |
|---|---|---|---|
| S1—C15—S2 | 110.93 (6) | | |

oxidation products stemming from the oxidation of (anthracen-9-ylmethyl)(benzyl)sulfane with ceric ammonium nitrate (Gopalakrishnan *et al.*, 2015).

Looking for a more rational manner to synthesize this tiara-like cluster, we attempted to prepare **Pd6** independently by reacting [PdCl$_2$(PhCN)$_2$] with 2.1 equivalents of benzyl mercaptan in CH$_2$Cl$_2$ solution. However, the isolation of **Pd6** was hampered by the co-crystallization of important amounts of the eight-membered cluster **Pd8** [Pd$_8$($\mu_2$-SCH$_2$Ph)$_{16}$], having a structure similar to that of [Pd$_8$($\mu_2$-SPr)$_{16}$] (Higgins *et al.*, 1988). Details of this reaction will be reported elsewhere.

## 2. Structural commentary

Compound **L1** crystallizes from the mixed solvents CH$_2$Cl$_2$/hexane in the monoclinic crystal system with $P2_1/c$ space group. The molecular structure of **L1** is presented in Fig. 2 and selected bond lengths and bond angles are given in Table 1. The C15—S1 and C15—S2 bond lengths of 1.8309 (12) and 1.8220 (12) Å are comparable with those of [BzSC(H)(C$_6$H$_4$NO$_2$-*p*)SBz] (SUNMAQ) [1.8262 (19) and 1.818 (2) Å; Binkowska *et al.*, 2009], but are elongated compared with those of bis(benzylsulfanyl)methane (TUQPAX) [1.7988 (13) and 1.8013 (13) Å; Yang *et al.*, 2010] and 2-[bis(benzylsulfanyl)methyl]-6-methoxyphenol
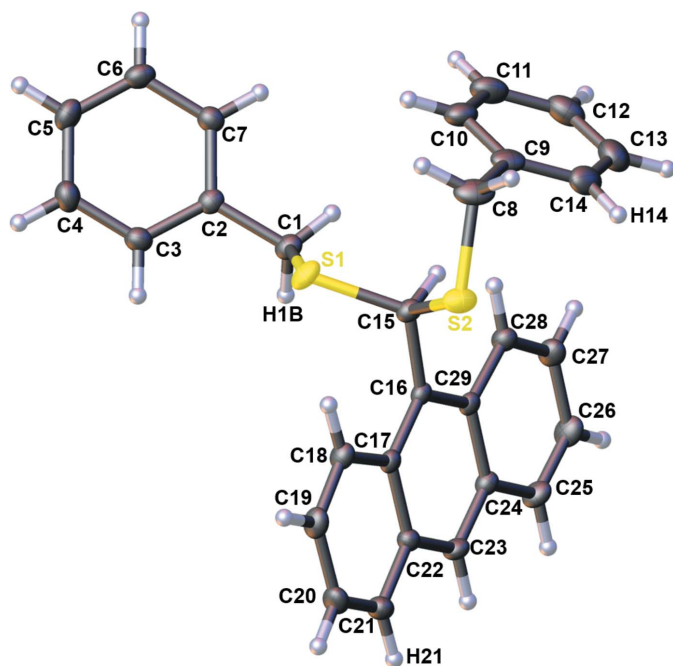
(IGOBOY) [1.8132 (12) and 1.8189 (12) Å; Raghuvanshi *et al.*, 2020). The angle S1—C15—S2 of 110.93 (6)° in **L1** is wider than those of 4-nitrophenylbis(benzylsulfanyl)methane [107.26 (6)°] and 2-[bis(benzylsulfanyl)methyl]-6-methoxy-phenol [107.76 (10)°], but considerably less than in [BzSCH$_2$SBz] [117.33 (7)°]. The phenyl rings of the benzyl groups and that of the anthracene unit form dihedral angles of 49.21 (4) and 58.79 (5)°.

The inorganic part of the crystal structure of the reaction product of **L1** with [PdCl$_2$(PhCN)$_2$] shown in Fig. 3 is very similar overall to the structures of a series of other structurally characterized tiara-like hexanuclear clusters bridged by aliphatic thiolate groups such as [Pd$_6$($\mu_2$-SPr)$_{12}$] (Kunchur, 1971; PDPRMC), [Pd$_6$($\mu_2$-SEt)$_{12}$] (Stash *et al.*, 2001; UCIXAF), [Pd$_6$($\mu_2$-SCH$_2$CH$_2$OH)$_{12}$] (Mahmudov *et al.*, 2013; XIPCUW), [Pd$_6$($\mu_2$-SBu)$_{12}$] (Stash *et al.*, 2009; LAFBUR) and [Pd$_6$($\mu_2$-SHexyl)$_{12}$] (Ananikov *et al.*, 2012; FAVQEA). Furthermore, the structure of the thiophenolate-spanned compound [Pd$_6$($\mu_2$-SPh)$_{12}$] has been reported (Stash *et al.*, 2009). However, within this series of metallacycles, the most reminiscent structure to our benzylic derivative [Pd$_6$($\mu_2$-SCH$_2$Ph)$_{12}$] is that of the phenylethanethiolate-decorated nanocluster [Pd$_6$($\mu_2$-SCH$_2$CH$_2$Ph)$_{12}$] (Chen *et al.*, 2017; HEGPAN).

The core of **Pd6** consists of three crystallographically different Pd$^{II}$ centers forming a centrosymmetric, almost planar, six-membered ring with Pd···Pd contacts ranging from 3.0892 (2) to 3.1609 (2) Å. The mean Pd···Pd separation of 3.1213 (2) Å is quite similar to that of the other derivatives and may be considered as weakly bonding (Stash *et al.*, 2009), being significantly shorter than the sum of the van der Waals



**Figure 2**
The molecular structure of **L1** with atom labelling and displacement ellipsoids drawn at the 50% probability level.
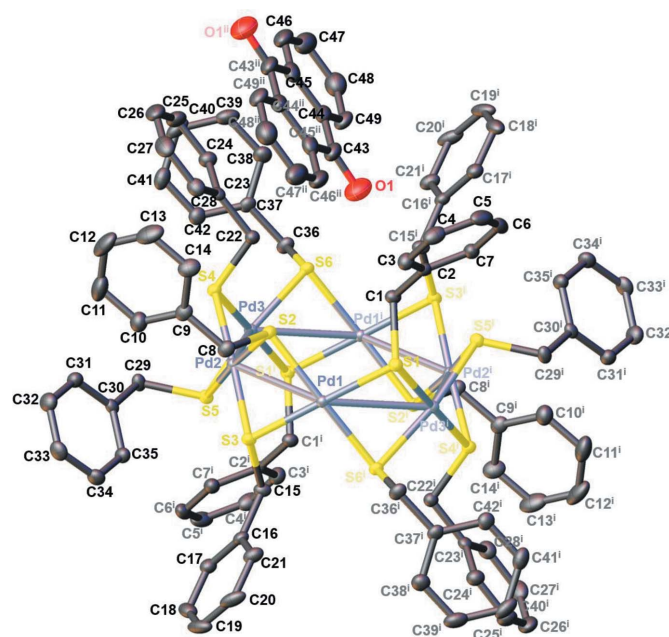


**Figure 3**
The molecular structure of **Pd6**·C$_{14}$H$_8$O$_2$ with the atom labelling and displacement ellipsoids drawn at the 50% probability level [symmetry codes: (i) −*x* + 1, −*y* + 1, −*z* + 1; (ii) −*x* + 1, −*y* + 1, −*z* + 2]. The H atoms are not shown for clarity.
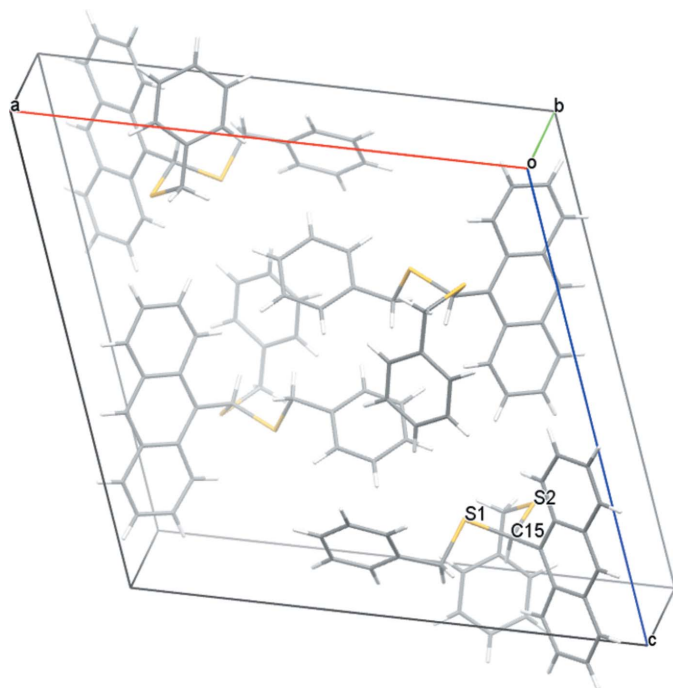
**Figure 4**
A view along the *b*-axis direction of the crystal packing of **L1**.

**Table 2**
Close contacts (Å, °) for **L1**.

*Cg* is the centroid of the C16/C17/C22–C24/C29 ring.

| $D—H\cdots A$ | $D—H$ | $H\cdots A$ | $D\cdots A$ | $D—H\cdots A$ |
|---|---|---|---|---|
| C21—H21···C16$^i$ | 0.951 (17) | 2.775 (17) | 3.7095 (17) | 167.6 (13) |
| C21—H21···C17$^i$ | 0.951 (17) | 2.856 (17) | 3.7737 (18) | 162.6 (13) |
| C21—H21···C29$^i$ | 0.951 (17) | 2.816 (17) | 3.6338 (17) | 144.7 (12) |
| C21—H21···Cg$^i$ | 0.951 (17) | 2.519 (18) | 3.4116 (14) | 156.3 (13) |
| C14—H14···C24$^{ii}$ | 0.976 (17) | 2.741 (18) | 3.5982 (19) | 146.9 (13) |
| C1—H1B···C9$^{iii}$ | 0.972 (16) | 2.847 (16) | 3.8023 (17) | 168.0 (12) |

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

central Pd$_6$ ring from both sides, conferring finally a tiara-like shape to the Pd$_6$S$_{12}$ scaffold.

Note that the crystal structure of anthracene-9,10-dione (also named 9,10-anthraquinone) has already been the object of several crystallographic studies and is therefore not commented herein (Fu & Brock, 1998; Slouf, 2002).

## 3. Supramolecular features

The crystal packing of dithioactal **L1** is shown in Fig. 4. Three different types of C—H···$\pi$ interactions are observed in the crystal structure (Fig. 5) where the H···$\pi$ distances range from 2.51 to 2.84 Å. The C21—H21···*Cg*(C16/C17/C22/C23/C24/C29 centroid) distance of 2.519 (18) Å, the C14—H14···C24 distance of 2.741 (18) Å and the C1—H1B···C9 distance of 2.847 (16) Å are short enough to be considered as weak intermolecular interactions (see Table 2). The closest C—H···S contact of 2.702 Å occurs between the aromatic H18 atom and S; however, the C18—H18···S1 angle of 123° suggests that this contribution has a neglectable impact on the conformation of **L1**.

A Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) for the further investigation of close contacts and intermolecular interactions was performed for **L1** using *Crystal-Explorer17* (Turner *et al.*, 2017). Figs. 6*a* and 7 illustrate the three-dimensional Hirshfeld surface mapped over $d_{norm}$ in the range from −1.11 to 1.36 (arbitrary units). The red spots on the surface indicate the close contacts to adjacent molecules. There are three areas of red spots which can be classified as C—H···$\pi$ interactions. The first and most important interaction is the C—H···$\pi$ contact of one of the phenyl-methanethiolate substituents to the anthracene scaffold of a
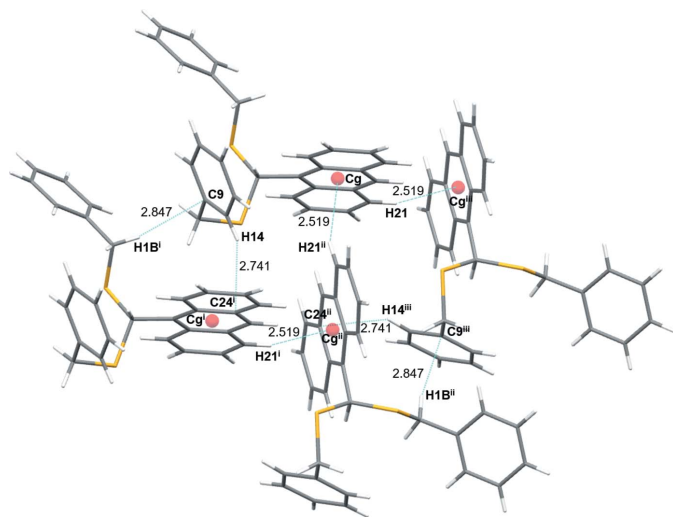


**Figure 5**
Intermolecular C—H···$\pi$ interactions occurring in **L1** generating a one-dimensional supramolecular ribbon [symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$; (iii) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$]
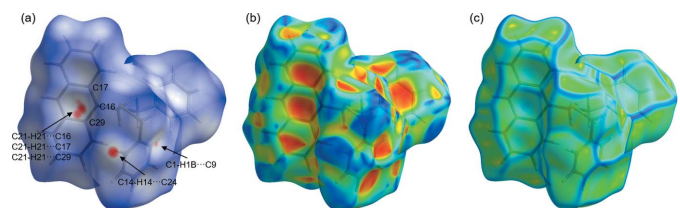


**Figure 6**
Hirshfeld surface mapped with (*a*) $d_{norm}$, (*b*) shape-index and (*c*) curvedness for **L1**.
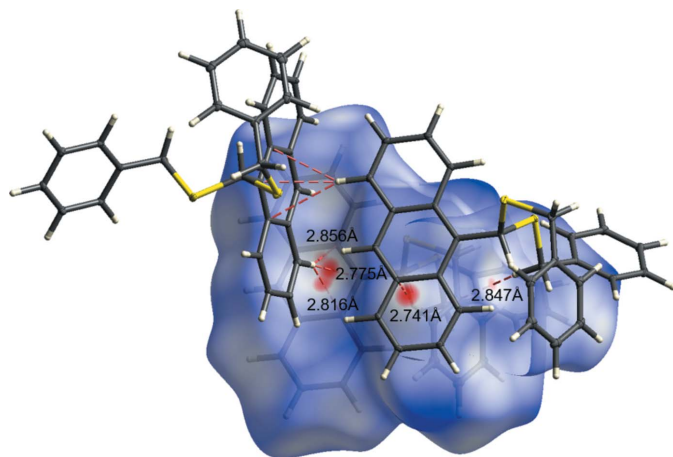
**Figure 7**
Hirshfeld surface analysis of **L1** showing close contacts in the crystal.

neighboring molecule (C14—H14···C24). Furthermore, there are significant interactions of the anthracene unit to an adjacent anthracene unit (C21—H21···C16/17/29). Then, there is also a weak C—H···$\pi$ contact of two phenylmethanethiolate substituents (C1—H1$B$···C9). The contributions of the different types of intermolecular interactions are shown in the two-dimensional fingerprint plots in Fig. 8. The weak van der Waals H···H contacts appear as the largest region with a 51.0% contribution. The C···H/H···C contacts exhibit a significant contribution at 40.4% and constitute a major contribution to the packing arrangement within the crystal structure. Fig. 6$b$ and 6$c$ illustrate the Hirshfeld surface mapped over the shape-index and the curvedness. The shape-index shows large red regions of concave curvature for the anthracene motif, whereas the C—H-donors shows opposite curvature.

Concerning the cluster **Pd6**, there are no particular directional intermolecular interactions in the packing warranting any discussion. The packing is shown in Fig. 9.
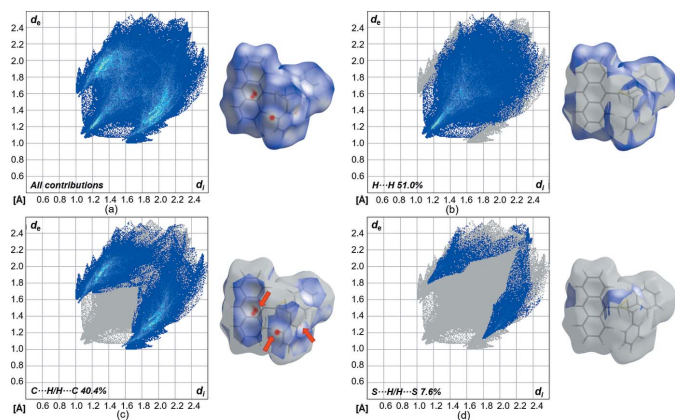


**Figure 8**
($a$) Two-dimensional fingerprint plots of **L1**, showing all contributions, and delineated ($b$)–($d$) showing the contributions of atoms within specific interacting pairs (blue areas).
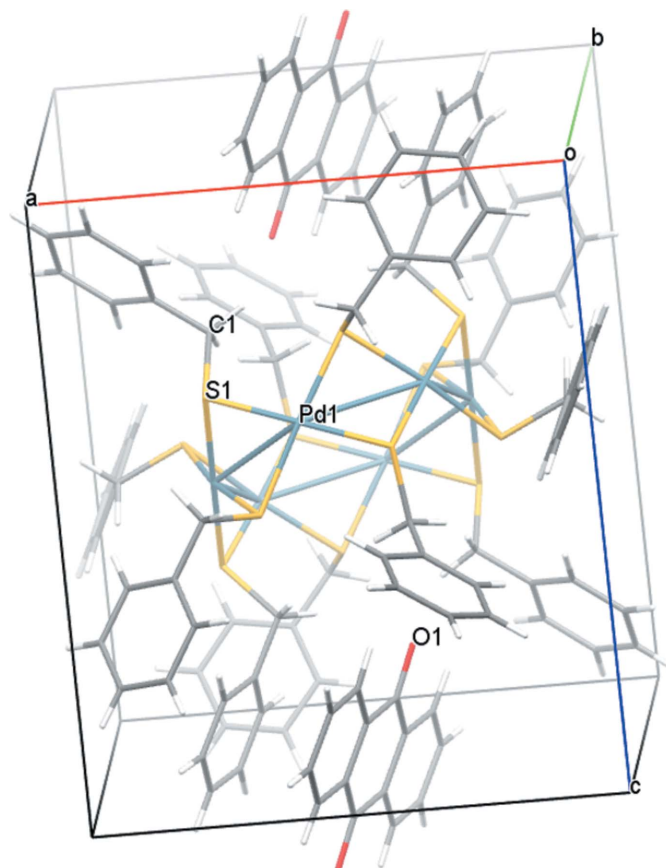


**Figure 9**
A view along the $b$-axis direction of the crystal packing of **Pd6**·$C_{14}H_8O_2$.

## 4. Database survey

A search of the Cambridge Structural Database (Groom *et al.*, 2016) for related anthracene-substituted dithioacetals did not reveal any structure hits. However, there are several examples of monothioethers attached on an anthracenyl scaffold and include {9-[(2-chloroethyl)thio]methyl}anthracene (CETMAN; Lewis *et al.*, 1976), 1,6-bis(9-anthryl)-2,5-dithia-hexane (LEYHIH; Schwarze *et al.*, 2007) and $S$-(9-anthr-yl)methyl-3,5-dinitrothiobenzoate (VEZLUI; Fowelin *et al.*, 2007). A search for the bis(benzylthio)methane motif $HC(SCH_2Ph)_2$ revealed only three similar structures, namely 2,6,10,14,19,24-hexa-$p$-benz-4,8,12,16,17,21,22,26-octathiatri-cyclo(9.5.5.5$^{3,9}$)hexacosaphane benzene clathrate (CUHLUM; Takemura *et al.*, 1984), 4-nitrophenyl-[bis(benzylthio)]-methane (SUNMAQ; Binkowska *et al.*, 2009) and 2-[bis-(benzylsulfanyl)methyl]-6-methoxyphenol (IGOBOY; Raghuvanshi *et al.*, 2020).

In contrast to mononuclear palladium complexes bearing terminal phenylmethanethiolate groups such as *trans*-[Pd(SCH$_2$Ph)$_2$(PMe$_3$)$_2$] (Lee *et al.* 2015; NOQZOK), [Pd(SCH$_2$Ph)$_2$(1,2-bis(diphenylphosphino)ethane)] (Su *et al.* 1997$a$,$b$; TERREN) and [Pd(SCH$_2$Ph)$_2$(1,3-bis(diphenyl-phosphino)propane)] (Su *et al.* 1997; SUTMOJ), those of phenylmethanethiolate-bridged di- and polynuclear Pd complexes are scare. The only crystallographically character-

**Table 3**
Experimental details.

| | L1 | Pd6 |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{29}H_{24}S_2$ | $[Pd_6(C_7H_7S)_{12}]\cdot C_{14}H_8O_2$ |
| $M_r$ | 436.60 | 2324.83 |
| Crystal system, space group | Monoclinic, $P2_1/c$ | Triclinic, $P\bar{1}$ |
| Temperature (K) | 123 | 100 |
| $a, b, c$ (Å) | 18.0842 (13), 7.5279 (5), 17.4975 (13) | 12.4037 (6), 13.2255 (6), 14.7347 (7) |
| $\alpha, \beta, \gamma$ (°) | 90, 108.439 (3), 90 | 109.842 (2), 91.616 (2), 91.191 (2) |
| $V$ (Å$^3$) | 2259.7 (3) | 2271.56 (19) |
| $Z$ | 4 | 1 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.25 | 1.49 |
| Crystal size (mm) | $0.95 \times 0.44 \times 0.30$ | $0.33 \times 0.24 \times 0.18$ |
| | | |
| Data collection | | |
| Diffractometer | Bruker D8 Venture | Bruker D8 Venture |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2016) | Multi-scan (*SADABS*; Bruker, 2016) |
| $T_{min}$, $T_{max}$ | 0.522, 0.563 | 0.300, 0.333 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 25688, 4994, 4423 | 109169, 10078, 9452 |
| $R_{int}$ | 0.025 | 0.028 |
| $(\sin \theta/\lambda)_{max}$ (Å$^{-1}$) | 0.641 | 0.644 |
| | | |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.032, 0.086, 1.05 | 0.019, 0.048, 1.10 |
| No. of reflections | 4994 | 10078 |
| No. of parameters | 295 | 532 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.24, −0.30 | 1.20, −0.79 |

Computer programs: *APEX2* (Bruker, 2018), *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009), *CrystalExplorer17* (Turner *et al.*, 2017), *publCIF* (Westrip, 2010) and *Mercury* (Macrae *et al.*, 2020).

ized hit is the tetranuclear cluster [Pd$_4$Se$_4$($\mu_2$-SCH$_2$Ph)$_2$-(bis(diphenylphosphino)methane)Cl$_2$] (Cao *et al.* 1998; JIXRAJ). The aforementioned [Pd$_6$($\mu_2$-SR)$_{12}$] clusters have found applications as precursors for the preparation of monodisperse PdS nanoparticles (Yang *et al.*, 2007), for the self-assembly of palladiumthiolate bilayers (Thomas *et al.*, 2001), as fluorescence materials (Chen *et al.*, 2017) and as electrocatalysts for H and O evolution reactions (Gao & Chen, 2017). Also noteworthy is the observation that individual [Pd$_6$($\mu_2$-SCH$_2$CH$_2$OH)$_{12}$] molecules are interconnected in the solid state by hydrogen bonds through the hydroxy groups of the thiolate ligands, thus generating an infinite three-dimensional supramolecular network (Mahmudov *et al.*, 2013). Concerning the influence of hydrogen-bonding interactions on nuclearity and structure for other tiara-like palladium complexes, see: Martin *et al.* (2018). Recently, a structurally related Pt$^{II}$ thiolate complex [Pt$_6$($\mu_2$-SC$_{12}$H$_{23}$)$_{12}$] has been prepared and probed as a macrocyclic host to include an Ag$^I$ ion as guest (Shichibu *et al.*, 2016).

## 5. Synthesis and crystallization

9-Anthracenecarboxaldehyde (206 mg, 1 mmol) and benzyl mercaptan (348 mg, 3 mmol) were suspended in conc. HCl (2 ml) and allowed to stir at room temperature. After 2 h, the reaction mixture was neutralized with aqueous NaHCO$_3$ solution and extracted with dichloromethane. The organic fraction was dried over Na$_2$SO$_4$, filtered and concentrated under reduced pressure. Purification by column chromatography using a hexane/dichloromethane solvent mixture as eluent gives a pale-yellow solid product in 80% yield (350 mg). Crystals suitable for single-crystal X-ray crystallography were grown by slow diffusion of hexane into a dichloromethane solution of **L1**, m.p. 438–440 K. $^1$H NMR (400 MHz, $\delta$ in ppm, CD$_2$Cl$_2$): 9.03 (*dd*, $J$ = 9.0 Hz, $J$ = 1.1 Hz, 1H, H$_{18}$), 8.39 (*s*, 1H, H$_{23}$), 8.00 (*dd*, $J$ = 8.5 Hz, $J$ = 1.1 Hz, 1H, H$_{21}$), 7.95 (*dd*, $J$ = 8.5 Hz, $J$ = 1.1 Hz, 1H, H$_{25}$), 7.55–7.47 (*m*, 2H, H$_{19}$, H$_{27}$), *ddd* ($J$ = 8.5 Hz, $J$ = 6.5 Hz, $J$ = 1.1 Hz, 1H, H$_3$), 7.28–7.22 (*m*, 6H, H$_{Ph}$ + H$_6$), 7.14–7.09 (*m*, 5H, H$_{Ph}$), 6.91 (*dd*, $J$ = 9.0 Hz, $J$ = 1.1 Hz, 1H, H$_{28}$), 5.94 (*s*, 1H, CHS$_2$), 3.79 (*d*, $J$ = 13.7 Hz, 2H, CH$_2$), 3.55 (*d*, $J$ = 13.7 Hz, 2H, CH$_2$). $^{13}$C{$^1$H} NMR (101 MHz, $\delta$ in ppm, CD$_2$Cl$_2$) 138.34 (C$_{16}$), 132.50 (C$_{17}$), 131.46 (Cq), 131.36 (Cq), 130.28 (Cq), 129.58 (CHAr), 129.56 (C$_{21}$), 129.47 (C$_{25}$), 129.13 (Cq), 128.96 (CHAr), 128.84 (C$_{23}$), 127.75 (C$_{18}$), 127.53 (CHAr), 126.63 (C$_{26}$), 125.61 (C$_{19}$), 125.12 (C$_{20}$), 124.91 (C$_{27}$), 122.99 (C$_{28}$), 45.02 (S$_2$CH), 37.89 (SCH$_2$). IR (ATR) cm$^{-1}$: 3050 and 3025 (C—H Ar), 2998, 2948 and 2906 (C—H aliphatic), 1589, 1519 (C=C), 696 (C—S).

**Reaction of L1 with PdCl$_2$(PhCN)$_2$**: **L1** (43 mg, 0.1 mmol) and PdCl$_2$(PhCN)$_2$ (38 mg, 0.1 mmol) were dissolved in 5 ml of dichloromethane and allowed to stir at room temperature for 30 minutes. During the reaction, a red solution was obtained, which was kept in refrigerator overnight yielding yellow crystals of 9-anthraldehyde along with yellow–orange co-crystals of the [Pd$_6$(SCH$_2$Ph)$_{12}$·anthracene-9,10-dione]

cluster, **Pd6**. $^1$H NMR (400 MHz, $\delta$ in ppm, $CD_2Cl_2$)): 8.92–6.86 (*m*, overlapping benzylic and anthracenyl H), 3.61 (*s*, $SCH_2$), 3.58 (*s*, $SCH_2$).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds, the H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and were refined using a riding model, with $U_{iso}$(H) = $1.2U_{eq}$(C). Hydrogen atoms H1*B*, H14 and H21 for **L1** were located in the difference-Fourier map and refined freely.

## References

Ananikov, V. P., Orlov, N. V., Zalesskiy, S. S., Beletskaya, I. P., Khrustalev, V. N., Morokuma, K. & Musaev, D. G. (2012). *J. Am. Chem. Soc.* **134**, 6637–6649.

Awaleh, M. O., Badia, A. & Brisse, F. (2005). *Acta Cryst.* E**61**, m1586–m1587.

Awaleh, M. O., Baril-Robert, F., Reber, C., Badia, A. & Brisse, F. (2008). *Inorg. Chem.* **47**, 2964–2974.

Binkowska, I., Ratajczak–Sitarz, M., Katrusiak, A. & Jarczewski, A. (2009). *J. Mol. Struct.* **928**, 54–58.

Blake, A. J., Holder, A. J., Roberts, Y. V. & Schröder, M. (1988). *Acta Cryst.* C**44**, 360–361.

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

Bruker (2016). *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2018). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cao, R., Su, W., Hong, M., Zhang, W., Lu, J. & Wong, W. (1998). *Chem. Commun.* pp. 2083–2084.

Chen, J., Pan, Y., Wang, Z. & Zhao, P. (2017). *Dalton Trans.* **46**, 12964–12970.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Fowelin, C., Schüpbach, B. & Terfort, A. (2007). *Eur. J. Org. Chem.* pp. 1013–1017.

Fu, Y. & Brock, C. P. (1998). *Acta Cryst.* B**54**, 308–315.

Gao, X. & Chen, W. (2017). *Chem. Commun.* **53**, 9733–9736.

Gopalakrishnan, R., Jacob, J. P., Moideen, S. F. T., Lalu, L. M., Unnikrishnan, P. A. & Prathapan, S. (2015). *Arkivoc*, **7**, 316–329.

Goswami, S. & Maity, A. C. (2008). *Tetrahedron Lett.* **49**, 3092–3096.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Higgins, J. D. & Suggs, J. W. (1988). *Inorg. Chim. Acta*, **145**, 247–252.

Hu, T.-L., Li, J.-R., Xie, Y.-B. & Bu, X.-H. (2006). *Cryst. Growth Des.* **6**, 648–655.

Hu, X.-L., Wang, K., Li, X., Pan, Q.-Q. & Su, Z.-M. (2020). *New J. Chem.* **44**, 12496–12502.

Knauer, L., Knorr, M., Viau, L. & Strohmann, C. (2020). *Acta Cryst.* E**76**, 38–41.

Knaust, J. M. & Keller, S. W. (2003). *CrystEngComm*, **5**, 459–465.

Knorr, M., Khatyr, A., Dini Aleo, A., El Yaagoubi, A., Strohmann, C., Kubicki, M. M., Rousselin, Y., Aly, S. M., Fortin, D., Lapprand, A. & Harvey, P. D. (2014). *Cryst. Growth Des.* **14**, 5373–5387.

Kunchur, N. R. (1971). *Acta Cryst.* B**27**, 2292.

Lee, S. G., Choi, K.-Y., Kim, Y.-J., Park, S. & Lee, S. W. (2015). *Polyhedron*, **85**, 880–887.

Lewis, M., Carrell, H. L., Glusker, J. P. & Sparks, R. A. (1976). *Acta Cryst.* B**32**, 2040–2044.

Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.

Madhu, S., Josimuddin, S. & Ravikanth, M. (2014). *New J. Chem.* **38**, 3770–3776.

Mahmudov, K. T., Hasanov, X. I., Maharramov, A. M., Azizova, A. N., Ragimov, K. Q., Askerov, R. K., Kopylovich, M. N., Ma, Z. & Pombeiro, A. J. L. (2013). *Inorg. Chem. Commun.* **29**, 37–39.

Martin, H. J., Pfeiffer, C. R., Davies, S. E., Davis, A. L., Lewis, W. & Champness, N. R. (2018). *ACS Omega*, **3**, 8769–8776.

Mohanty, A., Singh, U. P., Butcher, R. J., Das, N. & Roy, P. (2020). *CrystEngComm*, **22**, 4468–4477.

Murray, S. G., Levason, W. & Tuttlebee, H. E. (1981). *Inorg. Chim. Acta*, **51**, 185–189.

Olah, G. A., Narang, S. C. & Salem, G. F. (1980). *Synthesis*, pp. 659–660.

Peindy, H. N., Guyon, F., Khatyr, A., Knorr, M. & Strohmann, C. (2007). *Eur. J. Inorg. Chem.* pp. 1823–1828.

Pickardt, J. & Rautenberg, N. (1986). *Z. Naturforsch. Teil B*, **41**, 409–412.

Quah, H. S., Ng, L. T., Donnadieu, B., Tan, G. K. & Vittal, J. J. (2016). *Inorg. Chem.* **55**, 10851–10854.

Raghuvanshi, A., Dargallay, N. J., Knorr, M., Viau, L., Knauer, L. & Strohmann, C. (2017). *J. Inorg. Organomet. Polym.* **27**, 1501–1513.

Raghuvanshi, A., Knauer, L., Viau, L., Knorr, M. & Strohmann, C. (2020). *Acta Cryst.* E**76**, 484–487.

Raghuvanshi, A., Knorr, M., Knauer, L., Strohmann, C., Boullanger, S., Moutarlier, V. & Viau, L. (2019). *Inorg. Chem.* **58**, 5753–5775.

Rao, G. K., Kumar, A., Saleem, F., Singh, M. P., Kumar, S., Kumar, B., Mukherjee, G. & Singh, A. K. (2015). *Dalton Trans.* **44**, 6600–6612.

Schlachter, A., Lapprand, A., Fortin, D., Strohmann, C., Harvey, P. D. M. & Knorr, M. (2020). *Inorg. Chem.* **59**, 3686–3708.

Schlachter, A., Viau, L., Fortin, D., Knauer, L., Strohmann, C., Knorr, M. & Harvey, P. D. (2018). *Inorg. Chem.* **57**, 13564–13576.

Schwarze, T., Müller, H., Dosche, C., Klamroth, T., Mickler, W., Kelling, A., Löhmannsröben, H.-G., Saalfrank, P. & Holdt, H.-J. (2007). *Angew. Chem. Int. Ed.* **46**, 1671–1674.

Shaterian, H. R., Azizi, K. & Fahimi, N. (2011). *J. Sulfur Chem.* **32**, 85–91.

Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Shichibu, Y., Yoshida, K. & Konishi, K. (2016). *Inorg. Chem.* **55**, 9147–9149.

Slouf, M. (2002). *J. Mol. Struct.* **611**, 139–146.

Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm*, **11**, 19–32.

Stash, A. I., Levashova, V. V., Lebedev, S. A., Hoskov, Yu. G., Mal'kov, A. A. & Romm, I. P. (2009). *Russ. J. Coord. Chem.* **35**, 136–141.

Stash, A. I., Perepelkova, T. I., Noskov, Yu. G., Buslaeva, T. M. & Romm, I. P. (2001). *Russ. J. Coord. Chem.* **27**, 585–590.

Su, W., Cao, R., Hong, M., Zhou, Z., Xie, F., Liu, H. & Mak, T. C. W. (1997a). *Polyhedron*, **16**, 2531–2535.

Su, W., Hong, M., Cao, R. & Liu, H. (1997b). *Acta Cryst.* C**53**, 66–67.

Takemura, T., Kozawa, K., Uchida, T. & Mori, N. (1984). *Chem. Lett.* **13**, 1839–1842.

Thomas, P. J., Lavanya, A., Sabareesh, V. & Kulkarni, G. U. (2001). *J. Chem. Sci.* **113**, 611–619.

Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *CrystalExplorer17*. The University of Western Australia.

Wang, X., Gao, W.-Y., Luan, J., Wojtas, L. & Ma, S. (2016). *Chem. Commun.* **52**, 1971–1974.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Yang, H., Kim, T. H., Moon, S.-H. & Kim, J. (2010). *Acta Cryst.* E**66**, o1519.

Yang, Z., Klabunde, K. J. & Sorensen, C. M. (2007). *J. Phys. Chem. C*, **111**, 18143–18147.

# Crystal structures of 9-[bis(benzylsulfanyl)methyl]anthracene and of *cyclo*-dodecakis(*μ₂*-phenylmethanethiolato-*κ²S:S*)hexapalladium(6 *Pd*—*Pd*)–anthracene-9,10-dione (1/1)

**Abhinav Raghuvanshi, Anna Krupp, Lydie Viau, Michael Knorr and Carsten Strohmann**

**Computing details**

For both structures, data collection: *APEX2* (Bruker, 2018); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009), *CrystalExplorer17* (Turner *et al.*, 2017), *publCIF* (Westrip, 2010), *Mercury* (Macrae *et al.*, 2020).

**9-[Bis(benzylsulfanyl)methyl]anthracene (mo_b0159_0m)**

*Crystal data*

| | |
|---|---|
| $C_{29}H_{24}S_2$ | $F(000) = 920$ |
| $M_r = 436.60$ | $D_x = 1.283$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 18.0842$ (13) Å | Cell parameters from 9706 reflections |
| $b = 7.5279$ (5) Å | $\theta = 2.8$–27.1° |
| $c = 17.4975$ (13) Å | $\mu = 0.25$ mm$^{-1}$ |
| $\beta = 108.439$ (3)° | $T = 123$ K |
| $V = 2259.7$ (3) Å$^3$ | Block, yellow |
| $Z = 4$ | $0.95 \times 0.44 \times 0.30$ mm |

*Data collection*

| | |
|---|---|
| Bruker D8 Venture diffractometer | $T_{min} = 0.522$, $T_{max} = 0.563$ |
| | 25688 measured reflections |
| Radiation source: microfocus sealed X-ray tube, Incoatec I$\mu$s | 4994 independent reflections |
| | 4423 reflections with $I > 2\sigma(I)$ |
| HELIOS mirror optics monochromator | $R_{int} = 0.025$ |
| Detector resolution: 10.4167 pixels mm$^{-1}$ | $\theta_{max} = 27.1°$, $\theta_{min} = 2.8°$ |
| $\omega$ and $\varphi$ scans | $h = -23{\rightarrow}23$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2016) | $k = -9{\rightarrow}9$ |
| | $l = -22{\rightarrow}22$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | 295 parameters |
| Least-squares matrix: full | 0 restraints |
| $R[F^2 > 2\sigma(F^2)] = 0.032$ | Primary atom site location: iterative |
| $wR(F^2) = 0.086$ | Hydrogen site location: mixed |
| $S = 1.05$ | H atoms treated by a mixture of independent |
| 4994 reflections | and constrained refinement |

$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.9034P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.30$ 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)*

|  | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.68666 (2) | 0.64448 (5) | 0.70893 (2) | 0.02319 (9) |
| S2 | 0.80911 (2) | 0.92465 (4) | 0.72381 (2) | 0.02454 (9) |
| C1 | 0.62676 (7) | 0.52418 (17) | 0.62051 (8) | 0.0205 (2) |
| H1A | 0.6175 (9) | 0.602 (2) | 0.5751 (9) | 0.025* |
| C2 | 0.55092 (7) | 0.47989 (16) | 0.63477 (7) | 0.0185 (2) |
| C3 | 0.54888 (7) | 0.35800 (16) | 0.69428 (8) | 0.0221 (3) |
| H3 | 0.5950 | 0.2966 | 0.7235 | 0.027* |
| C4 | 0.47989 (8) | 0.32580 (18) | 0.71106 (8) | 0.0269 (3) |
| H4 | 0.4793 | 0.2443 | 0.7523 | 0.032* |
| C5 | 0.41191 (8) | 0.41281 (18) | 0.66757 (8) | 0.0270 (3) |
| H5 | 0.3648 | 0.3909 | 0.6790 | 0.032* |
| C6 | 0.41294 (7) | 0.53190 (18) | 0.60730 (8) | 0.0250 (3) |
| H6 | 0.3663 | 0.5899 | 0.5768 | 0.030* |
| C7 | 0.48232 (7) | 0.56614 (16) | 0.59167 (7) | 0.0213 (3) |
| H7 | 0.4829 | 0.6494 | 0.5511 | 0.026* |
| C8 | 0.73177 (9) | 1.08485 (18) | 0.67732 (9) | 0.0324 (3) |
| H8A | 0.6879 | 1.0657 | 0.6987 | 0.039* |
| H8B | 0.7519 | 1.2066 | 0.6922 | 0.039* |
| C9 | 0.70229 (8) | 1.06923 (16) | 0.58667 (9) | 0.0269 (3) |
| C10 | 0.62799 (8) | 1.00169 (18) | 0.54845 (9) | 0.0298 (3) |
| H10 | 0.5945 | 0.9749 | 0.5792 | 0.036* |
| C11 | 0.60257 (9) | 0.97328 (19) | 0.46564 (9) | 0.0350 (3) |
| H11 | 0.5521 | 0.9259 | 0.4402 | 0.042* |
| C12 | 0.65053 (10) | 1.0137 (2) | 0.42027 (9) | 0.0368 (3) |
| H12 | 0.6332 | 0.9937 | 0.3638 | 0.044* |
| C13 | 0.72407 (10) | 1.08361 (19) | 0.45753 (10) | 0.0378 (4) |
| H13 | 0.7568 | 1.1134 | 0.4264 | 0.045* |
| C14 | 0.74995 (9) | 1.11021 (18) | 0.54031 (10) | 0.0330 (3) |
| C15 | 0.76483 (7) | 0.72278 (16) | 0.67172 (7) | 0.0177 (2) |
| H15 | 0.7393 | 0.7570 | 0.6142 | 0.021* |
| C16 | 0.82754 (6) | 0.59003 (15) | 0.67177 (7) | 0.0151 (2) |
| C17 | 0.87649 (7) | 0.51631 (15) | 0.74463 (7) | 0.0160 (2) |
| C18 | 0.86799 (7) | 0.55321 (17) | 0.82190 (7) | 0.0203 (2) |
| H18 | 0.8266 | 0.6274 | 0.8251 | 0.024* |
| C19 | 0.91823 (8) | 0.48387 (18) | 0.89085 (7) | 0.0240 (3) |
| H19 | 0.9110 | 0.5098 | 0.9411 | 0.029* |

| | | | | |
|---|---|---|---|---|
| C20 | 0.98118 (8) | 0.37349 (18) | 0.88869 (8) | 0.0251 (3) |
| H20 | 1.0163 | 0.3281 | 0.9373 | 0.030* |
| C21 | 0.99092 (8) | 0.33320 (17) | 0.81703 (8) | 0.0231 (3) |
| C22 | 0.93948 (7) | 0.40166 (15) | 0.74321 (7) | 0.0178 (2) |
| C23 | 0.95162 (7) | 0.36306 (16) | 0.67019 (7) | 0.0196 (2) |
| H23 | 0.9933 | 0.2866 | 0.6697 | 0.024* |
| C24 | 0.90401 (7) | 0.43409 (15) | 0.59806 (7) | 0.0172 (2) |
| C25 | 0.91875 (7) | 0.39569 (17) | 0.52438 (8) | 0.0228 (3) |
| H25 | 0.9608 | 0.3194 | 0.5251 | 0.027* |
| C26 | 0.87412 (8) | 0.46550 (19) | 0.45322 (8) | 0.0257 (3) |
| H26 | 0.8849 | 0.4387 | 0.4048 | 0.031* |
| C27 | 0.81107 (8) | 0.57903 (18) | 0.45207 (7) | 0.0239 (3) |
| H27 | 0.7797 | 0.6278 | 0.4023 | 0.029* |
| C28 | 0.79469 (7) | 0.61934 (16) | 0.52090 (7) | 0.0203 (2) |
| H28 | 0.7520 | 0.6954 | 0.5180 | 0.024* |
| C29 | 0.84043 (6) | 0.54966 (15) | 0.59774 (7) | 0.0154 (2) |
| H1B | 0.6540 (9) | 0.417 (2) | 0.6137 (9) | 0.025 (4)* |
| H21 | 1.0321 (9) | 0.258 (2) | 0.8136 (9) | 0.033 (4)* |
| H14 | 0.8029 (10) | 1.152 (2) | 0.5669 (10) | 0.038 (5)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.01573 (15) | 0.03525 (18) | 0.02062 (16) | −0.00149 (12) | 0.00864 (12) | −0.00719 (12) |
| S2 | 0.02187 (17) | 0.02029 (16) | 0.02713 (17) | 0.00168 (11) | 0.00160 (13) | −0.00742 (12) |
| C1 | 0.0191 (6) | 0.0225 (6) | 0.0207 (6) | 0.0010 (5) | 0.0076 (5) | −0.0028 (5) |
| C2 | 0.0183 (6) | 0.0188 (5) | 0.0187 (6) | −0.0005 (4) | 0.0060 (5) | −0.0038 (4) |
| C3 | 0.0209 (6) | 0.0208 (6) | 0.0228 (6) | 0.0017 (5) | 0.0043 (5) | 0.0017 (5) |
| C4 | 0.0283 (7) | 0.0261 (6) | 0.0266 (6) | −0.0042 (5) | 0.0093 (5) | 0.0040 (5) |
| C5 | 0.0208 (6) | 0.0304 (7) | 0.0311 (7) | −0.0060 (5) | 0.0102 (5) | −0.0030 (6) |
| C6 | 0.0175 (6) | 0.0260 (6) | 0.0286 (7) | 0.0018 (5) | 0.0032 (5) | −0.0016 (5) |
| C7 | 0.0221 (6) | 0.0202 (6) | 0.0202 (6) | 0.0004 (5) | 0.0047 (5) | 0.0009 (5) |
| C8 | 0.0315 (7) | 0.0227 (6) | 0.0377 (8) | 0.0088 (6) | 0.0032 (6) | −0.0093 (6) |
| C9 | 0.0253 (7) | 0.0145 (6) | 0.0364 (7) | 0.0061 (5) | 0.0032 (6) | −0.0024 (5) |
| C10 | 0.0222 (6) | 0.0246 (6) | 0.0399 (8) | 0.0062 (5) | 0.0058 (6) | 0.0022 (6) |
| C11 | 0.0276 (7) | 0.0288 (7) | 0.0388 (8) | 0.0039 (6) | −0.0033 (6) | 0.0016 (6) |
| C12 | 0.0449 (9) | 0.0272 (7) | 0.0324 (8) | 0.0068 (6) | 0.0039 (7) | 0.0053 (6) |
| C13 | 0.0439 (9) | 0.0263 (7) | 0.0453 (9) | 0.0049 (6) | 0.0171 (7) | 0.0116 (6) |
| C14 | 0.0282 (7) | 0.0192 (6) | 0.0479 (9) | −0.0019 (5) | 0.0068 (7) | 0.0025 (6) |
| C15 | 0.0156 (5) | 0.0190 (5) | 0.0188 (5) | 0.0003 (4) | 0.0058 (4) | −0.0032 (4) |
| C16 | 0.0133 (5) | 0.0154 (5) | 0.0177 (5) | −0.0017 (4) | 0.0064 (4) | −0.0015 (4) |
| C17 | 0.0147 (5) | 0.0168 (5) | 0.0177 (6) | −0.0018 (4) | 0.0066 (4) | −0.0005 (4) |
| C18 | 0.0195 (6) | 0.0244 (6) | 0.0187 (6) | −0.0010 (5) | 0.0083 (5) | −0.0018 (5) |
| C19 | 0.0256 (6) | 0.0312 (7) | 0.0172 (6) | −0.0034 (5) | 0.0095 (5) | 0.0001 (5) |
| C20 | 0.0227 (6) | 0.0310 (7) | 0.0195 (6) | −0.0001 (5) | 0.0036 (5) | 0.0082 (5) |
| C21 | 0.0203 (6) | 0.0243 (6) | 0.0243 (6) | 0.0042 (5) | 0.0064 (5) | 0.0054 (5) |
| C22 | 0.0162 (6) | 0.0174 (5) | 0.0195 (6) | 0.0004 (4) | 0.0052 (5) | 0.0022 (4) |
| C23 | 0.0163 (6) | 0.0200 (6) | 0.0235 (6) | 0.0036 (4) | 0.0077 (5) | −0.0008 (5) |

| | | | | | |
|---|---|---|---|---|---|
| C24 | 0.0158 (5) | 0.0178 (5) | 0.0189 (6) | −0.0011 (4) | 0.0068 (4) | −0.0024 (4) |
| C25 | 0.0201 (6) | 0.0287 (6) | 0.0220 (6) | 0.0026 (5) | 0.0101 (5) | −0.0045 (5) |
| C26 | 0.0259 (7) | 0.0357 (7) | 0.0178 (6) | −0.0006 (6) | 0.0102 (5) | −0.0041 (5) |
| C27 | 0.0224 (6) | 0.0312 (7) | 0.0171 (6) | 0.0000 (5) | 0.0047 (5) | 0.0021 (5) |
| C28 | 0.0176 (6) | 0.0228 (6) | 0.0202 (6) | 0.0014 (5) | 0.0057 (5) | 0.0010 (5) |
| C29 | 0.0137 (5) | 0.0155 (5) | 0.0175 (5) | −0.0024 (4) | 0.0056 (4) | −0.0015 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C1 | 1.8240 (13) | C13—C14 | 1.389 (2) |
| S1—C15 | 1.8309 (12) | C14—H14 | 0.976 (17) |
| S2—C8 | 1.8309 (14) | C15—H15 | 1.0000 |
| S2—C15 | 1.8220 (12) | C15—C16 | 1.5114 (15) |
| C1—H1A | 0.958 (16) | C16—C17 | 1.4153 (16) |
| C1—C2 | 1.5070 (17) | C16—C29 | 1.4197 (16) |
| C1—H1B | 0.971 (16) | C17—C18 | 1.4355 (16) |
| C2—C3 | 1.3971 (18) | C17—C22 | 1.4356 (16) |
| C2—C7 | 1.3920 (17) | C18—H18 | 0.9500 |
| C3—H3 | 0.9500 | C18—C19 | 1.3639 (18) |
| C3—C4 | 1.3905 (19) | C19—H19 | 0.9500 |
| C4—H4 | 0.9500 | C19—C20 | 1.4195 (19) |
| C4—C5 | 1.3884 (19) | C20—H20 | 0.9500 |
| C5—H5 | 0.9500 | C20—C21 | 1.3545 (19) |
| C5—C6 | 1.3887 (19) | C21—C22 | 1.4281 (17) |
| C6—H6 | 0.9500 | C21—H21 | 0.951 (17) |
| C6—C7 | 1.3902 (18) | C22—C23 | 1.3937 (17) |
| C7—H7 | 0.9500 | C23—H23 | 0.9500 |
| C8—H8A | 0.9900 | C23—C24 | 1.3902 (17) |
| C8—H8B | 0.9900 | C24—C25 | 1.4262 (16) |
| C8—C9 | 1.510 (2) | C24—C29 | 1.4405 (16) |
| C9—C10 | 1.3938 (19) | C25—H25 | 0.9500 |
| C9—C14 | 1.392 (2) | C25—C26 | 1.3573 (19) |
| C10—H10 | 0.9500 | C26—H26 | 0.9500 |
| C10—C11 | 1.391 (2) | C26—C27 | 1.4200 (19) |
| C11—H11 | 0.9500 | C27—H27 | 0.9500 |
| C11—C12 | 1.382 (2) | C27—C28 | 1.3623 (18) |
| C12—H12 | 0.9500 | C28—H28 | 0.9500 |
| C12—C13 | 1.386 (2) | C28—C29 | 1.4365 (16) |
| C13—H13 | 0.9500 | | |
| | | | |
| C1—S1—C15 | 100.16 (6) | C13—C14—H14 | 120.0 (10) |
| C15—S2—C8 | 100.02 (6) | S1—C15—H15 | 106.2 |
| S1—C1—H1A | 107.5 (9) | S1—C15—S2 | 110.93 (6) |
| S1—C1—H1B | 109.1 (9) | S2—C15—H15 | 106.2 |
| H1A—C1—H1B | 111.7 (13) | C16—C15—S1 | 116.78 (8) |
| C2—C1—S1 | 107.27 (8) | C16—C15—S2 | 109.89 (8) |
| C2—C1—H1A | 110.1 (9) | C16—C15—H15 | 106.2 |
| C2—C1—H1B | 111.0 (9) | C17—C16—C15 | 121.01 (10) |

| | | | |
|---|---|---|---|
| C3—C2—C1 | 120.58 (11) | C17—C16—C29 | 120.10 (10) |
| C7—C2—C1 | 120.60 (11) | C29—C16—C15 | 118.73 (10) |
| C7—C2—C3 | 118.75 (11) | C16—C17—C18 | 123.35 (11) |
| C2—C3—H3 | 119.7 | C16—C17—C22 | 119.59 (10) |
| C4—C3—C2 | 120.59 (12) | C18—C17—C22 | 117.04 (11) |
| C4—C3—H3 | 119.7 | C17—C18—H18 | 119.4 |
| C3—C4—H4 | 120.0 | C19—C18—C17 | 121.28 (12) |
| C5—C4—C3 | 120.01 (12) | C19—C18—H18 | 119.4 |
| C5—C4—H4 | 120.0 | C18—C19—H19 | 119.5 |
| C4—C5—H5 | 120.0 | C18—C19—C20 | 121.06 (12) |
| C4—C5—C6 | 119.91 (12) | C20—C19—H19 | 119.5 |
| C6—C5—H5 | 120.0 | C19—C20—H20 | 120.1 |
| C5—C6—H6 | 120.0 | C21—C20—C19 | 119.74 (12) |
| C5—C6—C7 | 119.92 (12) | C21—C20—H20 | 120.1 |
| C7—C6—H6 | 120.0 | C20—C21—C22 | 121.21 (12) |
| C2—C7—H7 | 119.6 | C20—C21—H21 | 121.7 (10) |
| C6—C7—C2 | 120.81 (12) | C22—C21—H21 | 117.1 (10) |
| C6—C7—H7 | 119.6 | C21—C22—C17 | 119.65 (11) |
| S2—C8—H8A | 109.1 | C23—C22—C17 | 119.81 (11) |
| S2—C8—H8B | 109.1 | C23—C22—C21 | 120.50 (11) |
| H8A—C8—H8B | 107.8 | C22—C23—H23 | 119.3 |
| C9—C8—S2 | 112.44 (9) | C24—C23—C22 | 121.35 (11) |
| C9—C8—H8A | 109.1 | C24—C23—H23 | 119.3 |
| C9—C8—H8B | 109.1 | C23—C24—C25 | 120.23 (11) |
| C10—C9—C8 | 119.94 (14) | C23—C24—C29 | 120.02 (11) |
| C14—C9—C8 | 121.12 (13) | C25—C24—C29 | 119.74 (11) |
| C14—C9—C10 | 118.80 (14) | C24—C25—H25 | 119.2 |
| C9—C10—H10 | 119.8 | C26—C25—C24 | 121.59 (12) |
| C11—C10—C9 | 120.45 (14) | C26—C25—H25 | 119.2 |
| C11—C10—H10 | 119.8 | C25—C26—H26 | 120.4 |
| C10—C11—H11 | 119.9 | C25—C26—C27 | 119.19 (11) |
| C12—C11—C10 | 120.24 (14) | C27—C26—H26 | 120.4 |
| C12—C11—H11 | 119.9 | C26—C27—H27 | 119.3 |
| C11—C12—H12 | 120.1 | C28—C27—C26 | 121.32 (12) |
| C11—C12—C13 | 119.75 (15) | C28—C27—H27 | 119.3 |
| C13—C12—H12 | 120.1 | C27—C28—H28 | 119.2 |
| C12—C13—H13 | 119.9 | C27—C28—C29 | 121.61 (11) |
| C12—C13—C14 | 120.13 (15) | C29—C28—H28 | 119.2 |
| C14—C13—H13 | 119.9 | C16—C29—C24 | 119.14 (10) |
| C9—C14—H14 | 119.3 (10) | C16—C29—C28 | 124.31 (11) |
| C13—C14—C9 | 120.61 (14) | C28—C29—C24 | 116.54 (10) |
| | | | |
| S1—C1—C2—C3 | −67.96 (13) | C15—C16—C29—C24 | 175.17 (10) |
| S1—C1—C2—C7 | 108.83 (11) | C15—C16—C29—C28 | −3.66 (17) |
| S1—C15—C16—C17 | −63.16 (13) | C16—C17—C18—C19 | −177.73 (12) |
| S1—C15—C16—C29 | 121.33 (10) | C16—C17—C22—C21 | 177.36 (11) |
| S2—C8—C9—C10 | 109.50 (13) | C16—C17—C22—C23 | −0.39 (17) |
| S2—C8—C9—C14 | −66.21 (15) | C17—C16—C29—C24 | −0.38 (16) |

| | | | |
|---|---|---|---|
| S2—C15—C16—C17 | 64.29 (12) | C17—C16—C29—C28 | −179.21 (11) |
| S2—C15—C16—C29 | −111.22 (10) | C17—C18—C19—C20 | 0.44 (19) |
| C1—S1—C15—S2 | 153.75 (6) | C17—C22—C23—C24 | 0.29 (18) |
| C1—S1—C15—C16 | −79.31 (9) | C18—C17—C22—C21 | −1.20 (17) |
| C1—C2—C3—C4 | 175.75 (11) | C18—C17—C22—C23 | −178.95 (11) |
| C1—C2—C7—C6 | −176.90 (11) | C18—C19—C20—C21 | −1.3 (2) |
| C2—C3—C4—C5 | 1.1 (2) | C19—C20—C21—C22 | 0.8 (2) |
| C3—C2—C7—C6 | −0.05 (18) | C20—C21—C22—C17 | 0.42 (19) |
| C3—C4—C5—C6 | 0.0 (2) | C20—C21—C22—C23 | 178.16 (12) |
| C4—C5—C6—C7 | −1.2 (2) | C21—C22—C23—C24 | −177.45 (11) |
| C5—C6—C7—C2 | 1.19 (19) | C22—C17—C18—C19 | 0.77 (17) |
| C7—C2—C3—C4 | −1.10 (19) | C22—C23—C24—C25 | 178.59 (11) |
| C8—S2—C15—S1 | −73.63 (8) | C22—C23—C24—C29 | −0.23 (18) |
| C8—S2—C15—C16 | 155.73 (9) | C23—C24—C25—C26 | −178.85 (12) |
| C8—C9—C10—C11 | −174.81 (12) | C23—C24—C29—C16 | 0.27 (17) |
| C8—C9—C14—C13 | 175.49 (13) | C23—C24—C29—C28 | 179.19 (11) |
| C9—C10—C11—C12 | −0.7 (2) | C24—C25—C26—C27 | −0.2 (2) |
| C10—C9—C14—C13 | −0.3 (2) | C25—C24—C29—C16 | −178.55 (11) |
| C10—C11—C12—C13 | −0.3 (2) | C25—C24—C29—C28 | 0.37 (16) |
| C11—C12—C13—C14 | 1.0 (2) | C25—C26—C27—C28 | 0.1 (2) |
| C12—C13—C14—C9 | −0.8 (2) | C26—C27—C28—C29 | 0.2 (2) |
| C14—C9—C10—C11 | 1.00 (19) | C27—C28—C29—C16 | 178.40 (12) |
| C15—S1—C1—C2 | −169.19 (8) | C27—C28—C29—C24 | −0.46 (17) |
| C15—S2—C8—C9 | −47.89 (12) | C29—C16—C17—C18 | 178.91 (11) |
| C15—C16—C17—C18 | 3.46 (17) | C29—C16—C17—C22 | 0.44 (16) |
| C15—C16—C17—C22 | −175.01 (10) | C29—C24—C25—C26 | −0.04 (19) |

*Hydrogen-bond geometry (Å, º)*

*Cg* is the centroid of the C16/C17/C22–C24/C29 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C21—H21···C16[i] | 0.951 (17) | 2.775 (17) | 3.7095 (17) | 167.6 (13) |
| C21—H21···C17[i] | 0.951 (17) | 2.856 (17) | 3.7737 (18) | 162.6 (13) |
| C21—H21···C29[i] | 0.951 (17) | 2.816 (17) | 3.6338 (17) | 144.7 (12) |
| C21—H21···*Cg*[i] | 0.951 (17) | 2.519 (18) | 3.4116 (14) | 156.3 (13) |
| C14—H14···C24[ii] | 0.976 (17) | 2.741 (18) | 3.5982 (19) | 146.9 (13) |
| C1—H1*B*···C9[iii] | 0.972 (16) | 2.847 (16) | 3.8023 (17) | 168.0 (12) |

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

*cyclo*-Dodecakis(*µ*$_2$-phenylmethanethiolato-*κ*$^2$*S*:*S*)hexapalladium(6 *Pd*—*Pd*)–anthracene-9,10-dione (1/1) (mo_b0283_0m)

*Crystal data*

| | |
|---|---|
| [Pd$_6$(C$_7$H$_7$S)$_{12}$]·C$_{14}$H$_8$O$_2$ | *c* = 14.7347 (7) Å |
| *M*$_r$ = 2324.83 | *α* = 109.842 (2)° |
| Triclinic, *P*1̄ | *β* = 91.616 (2)° |
| *a* = 12.4037 (6) Å | *γ* = 91.191 (2)° |
| *b* = 13.2255 (6) Å | *V* = 2271.56 (19) Å$^3$ |

Z = 1
F(000) = 1164
$D_x$ = 1.699 Mg m$^{-3}$
Mo Kα radiation, λ = 0.71073 Å
Cell parameters from 9790 reflections

θ = 2.4–27.2°
μ = 1.49 mm$^{-1}$
T = 100 K
Block, yellow
0.33 × 0.24 × 0.18 mm

*Data collection*

Bruker D8 Venture
 diffractometer
Radiation source: microfocus sealed X-ray tube,
 Incoatec Iμs
HELIOS mirror optics monochromator
Detector resolution: 10.4167 pixels mm$^{-1}$
φ and ω scans
Absorption correction: multi-scan
 (SADABS; Bruker, 2016)

$T_{min}$ = 0.300, $T_{max}$ = 0.333
109169 measured reflections
10078 independent reflections
9452 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028
$\theta_{max}$ = 27.3°, $\theta_{min}$ = 2.3°
h = −15→15
k = −16→16
l = −18→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.048
S = 1.10
10078 reflections
532 parameters
0 restraints
Primary atom site location: dual

Hydrogen site location: inferred from
 neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0198P)^2 + 2.3315P]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 1.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.79 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  | 0.44100 (2)  |   | 0.57757 (2)  | 0.01264 (3)         |
| Pd2  | 0.67651 (2)  |   | 0.61284 (2)  | 0.01273 (4)         |
| Pd3  | 0.74009 (2)  |   | 0.53617 (2)  | 0.01294 (4)         |
| S1   | 0.27073 (4)  |   | 0.62062 (3)  | 0.01475 (8)         |
| S2   | 0.53960 (4)  |   | 0.71742 (3)  | 0.01514 (8)         |
| S3   | 0.61070 (4)  |   | 0.53538 (3)  | 0.01522 (9)         |
| S4   | 0.74917 (4)  |   | 0.69054 (3)  | 0.01469 (8)         |
| S5   | 0.79906 (4)  |   | 0.49857 (3)  | 0.01519 (9)         |
| S6   | 0.65044 (4)  |   | 0.56887 (3)  | 0.01522 (9)         |
| C1   | 0.28536 (15) |   | 0.71060 (13) | 0.0183 (4)          |
| H1A  | 0.2914       |   | 0.6773       | 0.022*              |
| H1B  | 0.3521       |   | 0.7508       | 0.022*              |
| C2   | 0.18968 (15) |   | 0.77377 (13) | 0.0183 (4)          |
| C3   | 0.18149 (16) |   | 0.84843 (14) | 0.0232 (4)          |
| H3   | 0.2364       |   | 0.8586       | 0.028*              |

| | | | |
|---|---|---|---|
| C4 | 0.09401 (19) | 0.90784 (15) | 0.0306 (5) |
| H4 | 0.0897 | 0.9589 | 0.037* |
| C5 | 0.01266 (18) | 0.89271 (17) | 0.0344 (5) |
| H5 | −0.0477 | 0.9330 | 0.041* |
| C6 | 0.01964 (17) | 0.81929 (17) | 0.0307 (5) |
| H6 | −0.0359 | 0.8090 | 0.037* |
| C7 | 0.10780 (16) | 0.75997 (15) | 0.0229 (4) |
| H7 | 0.1121 | 0.7096 | 0.027* |
| C8 | 0.57745 (16) | 0.77333 (14) | 0.0200 (4) |
| H8A | 0.6094 | 0.7244 | 0.024* |
| H8B | 0.5121 | 0.7966 | 0.024* |
| C9 | 0.65716 (16) | 0.85634 (14) | 0.0204 (4) |
| C10 | 0.76764 (18) | 0.84224 (17) | 0.0292 (5) |
| H10 | 0.7929 | 0.7797 | 0.035* |
| C11 | 0.8405 (2) | 0.9192 (2) | 0.0423 (6) |
| H11 | 0.9157 | 0.9094 | 0.051* |
| C12 | 0.8046 (2) | 1.0104 (2) | 0.0457 (7) |
| H12 | 0.8550 | 1.0630 | 0.055* |
| C13 | 0.6952 (2) | 1.02487 (17) | 0.0410 (6) |
| H13 | 0.6705 | 1.0875 | 0.049* |
| C14 | 0.6216 (2) | 0.94840 (15) | 0.0286 (5) |
| H14 | 0.5465 | 0.9586 | 0.034* |
| C15 | 0.62522 (16) | 0.40406 (13) | 0.0186 (4) |
| H15A | 0.6840 | 0.3891 | 0.022* |
| H15B | 0.5575 | 0.3726 | 0.022* |
| C16 | 0.65046 (15) | 0.36623 (13) | 0.0173 (4) |
| C17 | 0.74129 (16) | 0.31192 (14) | 0.0217 (4) |
| H17 | 0.7869 | 0.2981 | 0.026* |
| C18 | 0.76570 (19) | 0.27772 (15) | 0.0297 (5) |
| H18 | 0.8279 | 0.2408 | 0.036* |
| C19 | 0.6997 (2) | 0.29739 (15) | 0.0346 (6) |
| H19 | 0.7169 | 0.2751 | 0.042* |
| C20 | 0.6084 (2) | 0.34971 (15) | 0.0317 (5) |
| H20 | 0.5623 | 0.3621 | 0.038* |
| C21 | 0.58351 (18) | 0.38428 (14) | 0.0236 (4) |
| H21 | 0.5207 | 0.4204 | 0.028* |
| C22 | 0.65300 (15) | 0.77154 (13) | 0.0173 (4) |
| H22A | 0.5908 | 0.7803 | 0.021* |
| H22B | 0.6257 | 0.7433 | 0.021* |
| C23 | 0.71036 (15) | 0.86724 (13) | 0.0170 (4) |
| C24 | 0.76401 (17) | 0.88386 (15) | 0.0238 (4) |
| H24 | 0.7644 | 0.8345 | 0.029* |
| C25 | 0.81688 (19) | 0.97204 (17) | 0.0337 (5) |
| H25 | 0.8541 | 0.9827 | 0.040* |
| C26 | 0.8154 (2) | 1.04440 (16) | 0.0379 (6) |
| H26 | 0.8503 | 1.1053 | 0.046* |
| C27 | 0.7634 (2) | 1.02844 (16) | 0.0351 (5) |
| H27 | 0.7632 | 1.0781 | 0.042* |

| | | | |
|---|---|---|---|
| C28 | 0.71125 (17) | 0.93983 (15) | 0.0250 (4) |
| H28 | 0.6760 | 0.9289 | 0.030* |
| C29 | 0.93066 (15) | 0.55928 (15) | 0.0193 (4) |
| H29A | 0.9376 | 0.6184 | 0.023* |
| H29B | 0.9887 | 0.5161 | 0.023* |
| C30 | 0.94446 (14) | 0.58605 (13) | 0.0161 (3) |
| C31 | 0.99352 (16) | 0.67410 (14) | 0.0219 (4) |
| H31 | 1.0152 | 0.7183 | 0.026* |
| C32 | 1.01116 (18) | 0.69817 (15) | 0.0271 (4) |
| H32 | 1.0455 | 0.7585 | 0.033* |
| C33 | 0.97909 (17) | 0.63512 (16) | 0.0249 (4) |
| H33 | 0.9920 | 0.6516 | 0.030* |
| C34 | 0.92801 (17) | 0.54779 (15) | 0.0237 (4) |
| H34 | 0.9045 | 0.5046 | 0.028* |
| C35 | 0.91107 (16) | 0.52327 (14) | 0.0206 (4) |
| H35 | 0.8763 | 0.4630 | 0.025* |
| C36 | 0.74754 (16) | 0.55010 (14) | 0.0204 (4) |
| H36A | 0.7085 | 0.5319 | 0.025* |
| H36B | 0.7954 | 0.4971 | 0.025* |
| C37 | 0.81360 (15) | 0.64207 (14) | 0.0185 (4) |
| C38 | 0.78166 (18) | 0.70989 (15) | 0.0257 (4) |
| H38 | 0.7179 | 0.6978 | 0.031* |
| C39 | 0.8426 (2) | 0.79530 (16) | 0.0317 (5) |
| H39 | 0.8201 | 0.8413 | 0.038* |
| C40 | 0.93562 (19) | 0.81318 (16) | 0.0319 (5) |
| H40 | 0.9777 | 0.8710 | 0.038* |
| C41 | 0.96730 (17) | 0.74663 (16) | 0.0287 (5) |
| H41 | 1.0310 | 0.7590 | 0.034* |
| C42 | 0.90628 (16) | 0.66178 (15) | 0.0225 (4) |
| H42 | 0.9281 | 0.6168 | 0.027* |
| O1 | 0.40792 (18) | 0.82446 (14) | 0.0565 (5) |
| C43 | 0.44804 (19) | 0.90584 (17) | 0.0368 (6) |
| C44 | 0.50130 (18) | 0.92599 (16) | 0.0339 (5) |
| C45 | 0.55287 (18) | 1.01771 (17) | 0.0345 (5) |
| C46 | 0.6016 (2) | 1.03389 (19) | 0.0461 (7) |
| H46 | 0.6360 | 1.0951 | 0.055* |
| C47 | 0.6028 (2) | 0.9611 (2) | 0.0513 (7) |
| H47 | 0.6372 | 0.9730 | 0.062* |
| C48 | 0.5518 (2) | 0.8727 (2) | 0.0460 (6) |
| H48 | 0.5495 | 0.8238 | 0.055* |
| C49 | 0.50413 (19) | 0.85410 (16) | 0.0323 (5) |
| H49 | 0.4733 | 0.7918 | 0.039* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Pd1 | 0.01360 (7) | 0.01177 (6) | 0.01328 (6) | 0.00124 (5) | 0.00019 (5) | 0.00521 (5) |
| Pd2 | 0.01367 (7) | 0.01141 (6) | 0.01350 (6) | 0.00108 (5) | −0.00006 (5) | 0.00477 (5) |

| | | | | | | |
|------|--------------|--------------|--------------|---------------|----------------|---------------|
| Pd3 | 0.01424 (7) | 0.01067 (6) | 0.01404 (7) | 0.00100 (5) | 0.00019 (5) | 0.00438 (5) |
| S1 | 0.0155 (2) | 0.0137 (2) | 0.0150 (2) | 0.00133 (16) | 0.00174 (16) | 0.00482 (16) |
| S2 | 0.0157 (2) | 0.0155 (2) | 0.0145 (2) | 0.00187 (16) | 0.00014 (16) | 0.00552 (16) |
| S3 | 0.0163 (2) | 0.0148 (2) | 0.0164 (2) | 0.00159 (16) | 0.00165 (16) | 0.00749 (16) |
| S4 | 0.0163 (2) | 0.0140 (2) | 0.0142 (2) | 0.00107 (16) | −0.00068 (16) | 0.00557 (16) |
| S5 | 0.0169 (2) | 0.01204 (19) | 0.0169 (2) | 0.00067 (16) | 0.00173 (16) | 0.00513 (16) |
| S6 | 0.0175 (2) | 0.0136 (2) | 0.0157 (2) | 0.00024 (16) | −0.00150 (16) | 0.00665 (16) |
| C1 | 0.0190 (9) | 0.0196 (9) | 0.0184 (9) | 0.0021 (7) | 0.0018 (7) | 0.0092 (7) |
| C2 | 0.0168 (9) | 0.0238 (9) | 0.0170 (9) | 0.0006 (7) | −0.0003 (7) | 0.0106 (7) |
| C3 | 0.0221 (10) | 0.0253 (10) | 0.0210 (10) | 0.0006 (8) | −0.0003 (8) | 0.0064 (8) |
| C4 | 0.0321 (12) | 0.0398 (13) | 0.0195 (10) | 0.0128 (10) | 0.0042 (8) | 0.0085 (9) |
| C5 | 0.0225 (11) | 0.0615 (16) | 0.0314 (12) | 0.0134 (10) | 0.0113 (9) | 0.0301 (12) |
| C6 | 0.0178 (10) | 0.0461 (14) | 0.0401 (13) | −0.0043 (9) | −0.0012 (9) | 0.0306 (11) |
| C7 | 0.0221 (10) | 0.0254 (10) | 0.0246 (10) | −0.0031 (8) | −0.0029 (8) | 0.0135 (8) |
| C8 | 0.0243 (10) | 0.0144 (9) | 0.0191 (9) | 0.0036 (7) | −0.0010 (7) | 0.0031 (7) |
| C9 | 0.0262 (10) | 0.0123 (8) | 0.0198 (9) | 0.0027 (7) | −0.0018 (8) | 0.0017 (7) |
| C10 | 0.0268 (11) | 0.0276 (11) | 0.0319 (11) | −0.0027 (9) | −0.0033 (9) | 0.0089 (9) |
| C11 | 0.0315 (13) | 0.0372 (13) | 0.0557 (16) | −0.0077 (10) | −0.0188 (12) | 0.0148 (12) |
| C12 | 0.0607 (18) | 0.0309 (13) | 0.0402 (14) | −0.0060 (12) | −0.0312 (13) | 0.0081 (11) |
| C13 | 0.0710 (19) | 0.0274 (12) | 0.0195 (11) | 0.0039 (12) | −0.0085 (11) | 0.0020 (9) |
| C14 | 0.0393 (12) | 0.0232 (10) | 0.0203 (10) | 0.0076 (9) | 0.0028 (9) | 0.0029 (8) |
| C15 | 0.0225 (9) | 0.0179 (9) | 0.0170 (9) | 0.0036 (7) | 0.0036 (7) | 0.0076 (7) |
| C16 | 0.0202 (9) | 0.0180 (9) | 0.0146 (8) | 0.0011 (7) | −0.0029 (7) | 0.0067 (7) |
| C17 | 0.0230 (10) | 0.0251 (10) | 0.0180 (9) | 0.0002 (8) | −0.0010 (7) | 0.0090 (8) |
| C18 | 0.0390 (12) | 0.0334 (12) | 0.0173 (9) | −0.0142 (10) | −0.0016 (9) | 0.0104 (9) |
| C19 | 0.0666 (17) | 0.0205 (10) | 0.0180 (10) | −0.0128 (10) | −0.0086 (10) | 0.0099 (8) |
| C20 | 0.0575 (15) | 0.0172 (10) | 0.0192 (10) | 0.0096 (10) | −0.0094 (10) | 0.0051 (8) |
| C21 | 0.0314 (11) | 0.0207 (10) | 0.0183 (9) | 0.0063 (8) | −0.0022 (8) | 0.0062 (8) |
| C22 | 0.0169 (9) | 0.0187 (9) | 0.0179 (9) | 0.0002 (7) | 0.0000 (7) | 0.0082 (7) |
| C23 | 0.0165 (9) | 0.0203 (9) | 0.0156 (8) | −0.0013 (7) | 0.0032 (7) | 0.0077 (7) |
| C24 | 0.0254 (10) | 0.0256 (10) | 0.0224 (10) | 0.0019 (8) | 0.0007 (8) | 0.0108 (8) |
| C25 | 0.0325 (12) | 0.0444 (14) | 0.0322 (12) | −0.0009 (10) | −0.0055 (9) | 0.0243 (11) |
| C26 | 0.0393 (13) | 0.0556 (16) | 0.0218 (11) | −0.0131 (12) | −0.0089 (9) | 0.0186 (11) |
| C27 | 0.0419 (13) | 0.0369 (13) | 0.0187 (10) | −0.0124 (10) | 0.0015 (9) | 0.0000 (9) |
| C28 | 0.0284 (11) | 0.0220 (10) | 0.0223 (10) | −0.0013 (8) | 0.0041 (8) | 0.0042 (8) |
| C29 | 0.0146 (9) | 0.0156 (9) | 0.0286 (10) | 0.0010 (7) | 0.0001 (7) | 0.0089 (8) |
| C30 | 0.0127 (8) | 0.0150 (8) | 0.0221 (9) | 0.0014 (7) | 0.0036 (7) | 0.0079 (7) |
| C31 | 0.0222 (10) | 0.0226 (10) | 0.0234 (10) | −0.0014 (8) | −0.0026 (8) | 0.0113 (8) |
| C32 | 0.0318 (11) | 0.0228 (10) | 0.0233 (10) | −0.0062 (8) | −0.0042 (8) | 0.0042 (8) |
| C33 | 0.0271 (10) | 0.0141 (9) | 0.0324 (11) | −0.0021 (8) | 0.0020 (8) | 0.0066 (8) |
| C34 | 0.0259 (10) | 0.0201 (10) | 0.0294 (10) | −0.0001 (8) | −0.0004 (8) | 0.0141 (8) |
| C35 | 0.0231 (10) | 0.0181 (9) | 0.0212 (9) | −0.0033 (7) | −0.0032 (7) | 0.0081 (8) |
| C36 | 0.0252 (10) | 0.0139 (9) | 0.0214 (9) | 0.0035 (7) | −0.0040 (8) | 0.0051 (7) |
| C37 | 0.0203 (9) | 0.0158 (9) | 0.0197 (9) | 0.0054 (7) | −0.0010 (7) | 0.0062 (7) |
| C38 | 0.0268 (11) | 0.0240 (10) | 0.0296 (11) | 0.0023 (8) | −0.0008 (8) | 0.0136 (9) |
| C39 | 0.0426 (13) | 0.0329 (12) | 0.0248 (11) | 0.0110 (10) | 0.0012 (9) | 0.0161 (9) |
| C40 | 0.0358 (12) | 0.0333 (12) | 0.0226 (10) | 0.0122 (10) | −0.0092 (9) | 0.0046 (9) |
| C41 | 0.0213 (10) | 0.0270 (11) | 0.0325 (11) | 0.0030 (8) | −0.0057 (8) | 0.0036 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C42 | 0.0207 (10) | 0.0217 (10) | 0.0250 (10) | 0.0042 (8) | 0.0008 (8) | 0.0075 (8) |
| O1 | 0.0614 (13) | 0.0745 (15) | 0.0344 (10) | −0.0014 (11) | −0.0080 (9) | 0.0205 (10) |
| C43 | 0.0286 (12) | 0.0586 (16) | 0.0255 (11) | −0.0101 (11) | −0.0076 (9) | 0.0186 (11) |
| C44 | 0.0256 (11) | 0.0528 (15) | 0.0261 (11) | −0.0107 (10) | −0.0017 (9) | 0.0181 (10) |
| C45 | 0.0242 (11) | 0.0483 (14) | 0.0310 (12) | −0.0096 (10) | 0.0036 (9) | 0.0140 (11) |
| C46 | 0.0350 (13) | 0.080 (2) | 0.0313 (13) | −0.0133 (13) | −0.0103 (10) | 0.0308 (14) |
| C47 | 0.0313 (14) | 0.088 (2) | 0.0444 (16) | 0.0005 (14) | 0.0021 (11) | 0.0351 (16) |
| C48 | 0.0423 (15) | 0.0525 (16) | 0.0411 (14) | −0.0083 (12) | 0.0014 (12) | 0.0136 (13) |
| C49 | 0.0310 (12) | 0.0455 (14) | 0.0190 (10) | −0.0032 (10) | −0.0067 (9) | 0.0099 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Pd1—Pd2 | 3.1609 (2) | C19—H19 | 0.9500 |
| Pd1—Pd3[i] | 3.1139 (2) | C19—C20 | 1.381 (4) |
| Pd1—S1 | 2.3231 (5) | C20—H20 | 0.9500 |
| Pd1—S2 | 2.3374 (5) | C20—C21 | 1.384 (3) |
| Pd1—S3 | 2.3281 (5) | C21—H21 | 0.9500 |
| Pd1—S6[i] | 2.3264 (5) | C22—H22A | 0.9900 |
| Pd2—Pd3 | 3.0892 (2) | C22—H22B | 0.9900 |
| Pd2—S2 | 2.3342 (5) | C22—C23 | 1.504 (2) |
| Pd2—S3 | 2.3154 (4) | C23—C24 | 1.394 (3) |
| Pd2—S4 | 2.3250 (4) | C23—C28 | 1.387 (3) |
| Pd2—S5 | 2.3277 (5) | C24—H24 | 0.9500 |
| Pd3—Pd1[i] | 3.1139 (2) | C24—C25 | 1.387 (3) |
| Pd3—S1[i] | 2.3367 (5) | C25—H25 | 0.9500 |
| Pd3—S4 | 2.3230 (5) | C25—C26 | 1.382 (4) |
| Pd3—S5 | 2.3197 (4) | C26—H26 | 0.9500 |
| Pd3—S6 | 2.3264 (5) | C26—C27 | 1.380 (4) |
| S1—Pd3[i] | 2.3367 (5) | C27—H27 | 0.9500 |
| S1—C1 | 1.8402 (18) | C27—C28 | 1.390 (3) |
| S2—C8 | 1.8440 (19) | C28—H28 | 0.9500 |
| S3—C15 | 1.8378 (19) | C29—H29A | 0.9900 |
| S4—C22 | 1.8411 (19) | C29—H29B | 0.9900 |
| S5—C29 | 1.8365 (19) | C29—C30 | 1.507 (2) |
| S6—Pd1[i] | 2.3264 (5) | C30—C31 | 1.385 (3) |
| S6—C36 | 1.8434 (19) | C30—C35 | 1.396 (3) |
| C1—H1A | 0.9900 | C31—H31 | 0.9500 |
| C1—H1B | 0.9900 | C31—C32 | 1.391 (3) |
| C1—C2 | 1.501 (3) | C32—H32 | 0.9500 |
| C2—C3 | 1.398 (3) | C32—C33 | 1.383 (3) |
| C2—C7 | 1.392 (3) | C33—H33 | 0.9500 |
| C3—H3 | 0.9500 | C33—C34 | 1.384 (3) |
| C3—C4 | 1.386 (3) | C34—H34 | 0.9500 |
| C4—H4 | 0.9500 | C34—C35 | 1.385 (3) |
| C4—C5 | 1.390 (4) | C35—H35 | 0.9500 |
| C5—H5 | 0.9500 | C36—H36A | 0.9900 |
| C5—C6 | 1.376 (4) | C36—H36B | 0.9900 |
| C6—H6 | 0.9500 | C36—C37 | 1.505 (3) |

| | | | |
|---|---|---|---|
| C6—C7 | 1.392 (3) | C37—C38 | 1.393 (3) |
| C7—H7 | 0.9500 | C37—C42 | 1.389 (3) |
| C8—H8A | 0.9900 | C38—H38 | 0.9500 |
| C8—H8B | 0.9900 | C38—C39 | 1.394 (3) |
| C8—C9 | 1.501 (3) | C39—H39 | 0.9500 |
| C9—C10 | 1.394 (3) | C39—C40 | 1.382 (4) |
| C9—C14 | 1.396 (3) | C40—H40 | 0.9500 |
| C10—H10 | 0.9500 | C40—C41 | 1.383 (3) |
| C10—C11 | 1.384 (3) | C41—H41 | 0.9500 |
| C11—H11 | 0.9500 | C41—C42 | 1.389 (3) |
| C11—C12 | 1.384 (4) | C42—H42 | 0.9500 |
| C12—H12 | 0.9500 | O1—C43 | 1.240 (3) |
| C12—C13 | 1.382 (4) | C43—C44 | 1.470 (4) |
| C13—H13 | 0.9500 | C43—C45ⁱⁱ | 1.473 (4) |
| C13—C14 | 1.384 (3) | C44—C45 | 1.425 (3) |
| C14—H14 | 0.9500 | C44—C49 | 1.383 (3) |
| C15—H15A | 0.9900 | C45—C43ⁱⁱ | 1.473 (4) |
| C15—H15B | 0.9900 | C45—C46 | 1.354 (4) |
| C15—C16 | 1.501 (2) | C46—H46 | 0.9500 |
| C16—C17 | 1.393 (3) | C46—C47 | 1.436 (5) |
| C16—C21 | 1.394 (3) | C47—H47 | 0.9500 |
| C17—H17 | 0.9500 | C47—C48 | 1.387 (4) |
| C17—C18 | 1.391 (3) | C48—H48 | 0.9500 |
| C18—H18 | 0.9500 | C48—C49 | 1.389 (4) |
| C18—C19 | 1.383 (4) | C49—H49 | 0.9500 |
| | | | |
| Pd3ⁱ—Pd1—Pd2 | 122.696 (6) | C16—C15—S3 | 109.37 (13) |
| S1—Pd1—Pd2 | 133.948 (12) | C16—C15—H15A | 109.8 |
| S1—Pd1—Pd3ⁱ | 48.255 (11) | C16—C15—H15B | 109.8 |
| S1—Pd1—S2 | 99.152 (16) | C17—C16—C15 | 120.28 (17) |
| S1—Pd1—S3 | 178.778 (16) | C17—C16—C21 | 118.90 (18) |
| S1—Pd1—S6ⁱ | 81.998 (16) | C21—C16—C15 | 120.82 (18) |
| S2—Pd1—Pd2 | 47.378 (11) | C16—C17—H17 | 119.8 |
| S2—Pd1—Pd3ⁱ | 129.132 (12) | C18—C17—C16 | 120.5 (2) |
| S3—Pd1—Pd2 | 46.933 (11) | C18—C17—H17 | 119.8 |
| S3—Pd1—Pd3ⁱ | 132.454 (13) | C17—C18—H18 | 119.9 |
| S3—Pd1—S2 | 81.033 (16) | C19—C18—C17 | 120.1 (2) |
| S6ⁱ—Pd1—Pd2 | 128.215 (13) | C19—C18—H18 | 119.9 |
| S6ⁱ—Pd1—Pd3ⁱ | 47.991 (11) | C18—C19—H19 | 120.2 |
| S6ⁱ—Pd1—S2 | 174.139 (16) | C20—C19—C18 | 119.59 (19) |
| S6ⁱ—Pd1—S3 | 97.940 (16) | C20—C19—H19 | 120.2 |
| Pd3—Pd2—Pd1 | 121.425 (6) | C19—C20—H20 | 119.6 |
| S2—Pd2—Pd1 | 47.463 (11) | C19—C20—C21 | 120.8 (2) |
| S2—Pd2—Pd3 | 128.222 (12) | C21—C20—H20 | 119.6 |
| S3—Pd2—Pd1 | 47.270 (12) | C16—C21—H21 | 119.9 |
| S3—Pd2—Pd3 | 131.984 (12) | C20—C21—C16 | 120.1 (2) |
| S3—Pd2—S2 | 81.367 (16) | C20—C21—H21 | 119.9 |
| S3—Pd2—S4 | 177.813 (17) | S4—C22—H22A | 110.0 |

| | | | |
|---|---|---|---|
| S3—Pd2—S5 | 98.089 (16) | S4—C22—H22B | 110.0 |
| S4—Pd2—Pd1 | 134.849 (12) | H22A—C22—H22B | 108.4 |
| S4—Pd2—Pd3 | 48.318 (11) | C23—C22—S4 | 108.39 (12) |
| S4—Pd2—S2 | 99.980 (16) | C23—C22—H22A | 110.0 |
| S4—Pd2—S5 | 80.756 (16) | C23—C22—H22B | 110.0 |
| S5—Pd2—Pd1 | 128.271 (13) | C24—C23—C22 | 120.23 (17) |
| S5—Pd2—Pd3 | 48.229 (11) | C28—C23—C22 | 120.67 (17) |
| S5—Pd2—S2 | 173.880 (17) | C28—C23—C24 | 119.10 (18) |
| Pd2—Pd3—Pd1ⁱ | 115.879 (6) | C23—C24—H24 | 119.8 |
| S1ⁱ—Pd3—Pd1ⁱ | 47.884 (11) | C25—C24—C23 | 120.4 (2) |
| S1ⁱ—Pd3—Pd2 | 130.174 (12) | C25—C24—H24 | 119.8 |
| S4—Pd3—Pd1ⁱ | 132.157 (12) | C24—C25—H25 | 120.0 |
| S4—Pd3—Pd2 | 48.373 (11) | C26—C25—C24 | 119.9 (2) |
| S4—Pd3—S1ⁱ | 178.547 (16) | C26—C25—H25 | 120.0 |
| S4—Pd3—S6 | 99.246 (16) | C25—C26—H26 | 119.9 |
| S5—Pd3—Pd1ⁱ | 125.043 (13) | C27—C26—C25 | 120.1 (2) |
| S5—Pd3—Pd2 | 48.449 (12) | C27—C26—H26 | 119.9 |
| S5—Pd3—S1ⁱ | 97.903 (16) | C26—C27—H27 | 120.0 |
| S5—Pd3—S4 | 80.964 (16) | C26—C27—C28 | 120.1 (2) |
| S5—Pd3—S6 | 169.831 (17) | C28—C27—H27 | 120.0 |
| S6—Pd3—Pd1ⁱ | 47.990 (11) | C23—C28—C27 | 120.3 (2) |
| S6—Pd3—Pd2 | 124.764 (13) | C23—C28—H28 | 119.8 |
| S6—Pd3—S1ⁱ | 81.707 (16) | C27—C28—H28 | 119.8 |
| Pd1—S1—Pd3ⁱ | 83.862 (15) | S5—C29—H29A | 109.2 |
| C1—S1—Pd1 | 109.07 (6) | S5—C29—H29B | 109.2 |
| C1—S1—Pd3ⁱ | 111.18 (6) | H29A—C29—H29B | 107.9 |
| Pd2—S2—Pd1 | 85.159 (15) | C30—C29—S5 | 111.89 (13) |
| C8—S2—Pd1 | 103.40 (6) | C30—C29—H29A | 109.2 |
| C8—S2—Pd2 | 106.26 (7) | C30—C29—H29B | 109.2 |
| Pd2—S3—Pd1 | 85.797 (15) | C31—C30—C29 | 119.56 (17) |
| C15—S3—Pd1 | 110.88 (7) | C31—C30—C35 | 118.78 (17) |
| C15—S3—Pd2 | 111.96 (6) | C35—C30—C29 | 121.64 (17) |
| Pd3—S4—Pd2 | 83.308 (15) | C30—C31—H31 | 119.8 |
| C22—S4—Pd2 | 111.99 (6) | C30—C31—C32 | 120.31 (18) |
| C22—S4—Pd3 | 112.72 (6) | C32—C31—H31 | 119.8 |
| Pd3—S5—Pd2 | 83.321 (15) | C31—C32—H32 | 119.7 |
| C29—S5—Pd2 | 103.36 (6) | C33—C32—C31 | 120.59 (19) |
| C29—S5—Pd3 | 109.93 (6) | C33—C32—H32 | 119.7 |
| Pd1ⁱ—S6—Pd3 | 84.020 (15) | C32—C33—H33 | 120.3 |
| C36—S6—Pd1ⁱ | 108.12 (6) | C32—C33—C34 | 119.45 (18) |
| C36—S6—Pd3 | 106.45 (7) | C34—C33—H33 | 120.3 |
| S1—C1—H1A | 109.6 | C33—C34—H34 | 119.9 |
| S1—C1—H1B | 109.6 | C33—C34—C35 | 120.10 (18) |
| H1A—C1—H1B | 108.1 | C35—C34—H34 | 119.9 |
| C2—C1—S1 | 110.14 (13) | C30—C35—H35 | 119.6 |
| C2—C1—H1A | 109.6 | C34—C35—C30 | 120.75 (18) |
| C2—C1—H1B | 109.6 | C34—C35—H35 | 119.6 |
| C3—C2—C1 | 120.30 (17) | S6—C36—H36A | 109.9 |

| | | | |
|---|---|---|---|
| C7—C2—C1 | 121.12 (18) | S6—C36—H36B | 109.9 |
| C7—C2—C3 | 118.57 (18) | H36A—C36—H36B | 108.3 |
| C2—C3—H3 | 119.7 | C37—C36—S6 | 108.93 (13) |
| C4—C3—C2 | 120.7 (2) | C37—C36—H36A | 109.9 |
| C4—C3—H3 | 119.7 | C37—C36—H36B | 109.9 |
| C3—C4—H4 | 120.0 | C38—C37—C36 | 120.57 (18) |
| C3—C4—C5 | 120.0 (2) | C42—C37—C36 | 120.61 (17) |
| C5—C4—H4 | 120.0 | C42—C37—C38 | 118.81 (18) |
| C4—C5—H5 | 120.1 | C37—C38—H38 | 119.8 |
| C6—C5—C4 | 119.9 (2) | C37—C38—C39 | 120.4 (2) |
| C6—C5—H5 | 120.1 | C39—C38—H38 | 119.8 |
| C5—C6—H6 | 119.9 | C38—C39—H39 | 119.9 |
| C5—C6—C7 | 120.3 (2) | C40—C39—C38 | 120.1 (2) |
| C7—C6—H6 | 119.9 | C40—C39—H39 | 119.9 |
| C2—C7—C6 | 120.6 (2) | C39—C40—H40 | 120.1 |
| C2—C7—H7 | 119.7 | C39—C40—C41 | 119.8 (2) |
| C6—C7—H7 | 119.7 | C41—C40—H40 | 120.1 |
| S2—C8—H8A | 109.5 | C40—C41—H41 | 119.9 |
| S2—C8—H8B | 109.5 | C40—C41—C42 | 120.2 (2) |
| H8A—C8—H8B | 108.0 | C42—C41—H41 | 119.9 |
| C9—C8—S2 | 110.92 (13) | C37—C42—H42 | 119.7 |
| C9—C8—H8A | 109.5 | C41—C42—C37 | 120.6 (2) |
| C9—C8—H8B | 109.5 | C41—C42—H42 | 119.7 |
| C10—C9—C8 | 120.45 (18) | O1—C43—C44 | 120.5 (3) |
| C10—C9—C14 | 119.15 (19) | O1—C43—C45ⁱⁱ | 120.3 (3) |
| C14—C9—C8 | 120.40 (19) | C44—C43—C45ⁱⁱ | 119.2 (2) |
| C9—C10—H10 | 120.0 | C45—C44—C43 | 120.8 (2) |
| C11—C10—C9 | 120.1 (2) | C49—C44—C43 | 119.5 (2) |
| C11—C10—H10 | 120.0 | C49—C44—C45 | 119.6 (2) |
| C10—C11—H11 | 119.8 | C44—C45—C43ⁱⁱ | 120.0 (2) |
| C10—C11—C12 | 120.5 (3) | C46—C45—C43ⁱⁱ | 120.6 (2) |
| C12—C11—H11 | 119.8 | C46—C45—C44 | 119.4 (2) |
| C11—C12—H12 | 120.1 | C45—C46—H46 | 119.1 |
| C13—C12—C11 | 119.8 (2) | C45—C46—C47 | 121.9 (2) |
| C13—C12—H12 | 120.1 | C47—C46—H46 | 119.1 |
| C12—C13—H13 | 119.9 | C46—C47—H47 | 121.4 |
| C12—C13—C14 | 120.2 (2) | C48—C47—C46 | 117.2 (3) |
| C14—C13—H13 | 119.9 | C48—C47—H47 | 121.4 |
| C9—C14—H14 | 119.9 | C47—C48—H48 | 119.2 |
| C13—C14—C9 | 120.3 (2) | C47—C48—C49 | 121.6 (3) |
| C13—C14—H14 | 119.9 | C49—C48—H48 | 119.2 |
| S3—C15—H15A | 109.8 | C44—C49—C48 | 120.2 (2) |
| S3—C15—H15B | 109.8 | C44—C49—H49 | 119.9 |
| H15A—C15—H15B | 108.2 | C48—C49—H49 | 119.9 |
| | | | |
| Pd1—S1—C1—C2 | 152.50 (12) | C17—C18—C19—C20 | −1.1 (3) |
| Pd1—S2—C8—C9 | −170.66 (13) | C18—C19—C20—C21 | 1.3 (3) |
| Pd1—S3—C15—C16 | 121.75 (12) | C19—C20—C21—C16 | −0.2 (3) |

| | | | |
|---|---|---|---|
| Pd1<sup>i</sup>—S6—C36—C37 | 175.62 (12) | C21—C16—C17—C18 | 1.1 (3) |
| Pd2—S2—C8—C9 | −81.85 (14) | C22—C23—C24—C25 | −179.93 (19) |
| Pd2—S3—C15—C16 | −144.29 (11) | C22—C23—C28—C27 | 179.26 (19) |
| Pd2—S4—C22—C23 | −132.34 (11) | C23—C24—C25—C26 | 0.8 (3) |
| Pd2—S5—C29—C30 | −60.03 (14) | C24—C23—C28—C27 | −1.1 (3) |
| Pd3<sup>i</sup>—S1—C1—C2 | −116.78 (12) | C24—C25—C26—C27 | −1.4 (4) |
| Pd3—S4—C22—C23 | 135.78 (11) | C25—C26—C27—C28 | 0.7 (4) |
| Pd3—S5—C29—C30 | −147.68 (12) | C26—C27—C28—C23 | 0.5 (3) |
| Pd3—S6—C36—C37 | 86.64 (13) | C28—C23—C24—C25 | 0.5 (3) |
| S1—C1—C2—C3 | −78.1 (2) | C29—C30—C31—C32 | 177.18 (19) |
| S1—C1—C2—C7 | 102.25 (18) | C29—C30—C35—C34 | −177.65 (18) |
| S2—C8—C9—C10 | 93.6 (2) | C30—C31—C32—C33 | 0.6 (3) |
| S2—C8—C9—C14 | −85.6 (2) | C31—C30—C35—C34 | 0.9 (3) |
| S3—C15—C16—C17 | 125.44 (16) | C31—C32—C33—C34 | 0.7 (3) |
| S3—C15—C16—C21 | −54.9 (2) | C32—C33—C34—C35 | −1.2 (3) |
| S4—C22—C23—C24 | −89.47 (19) | C33—C34—C35—C30 | 0.4 (3) |
| S4—C22—C23—C28 | 90.15 (19) | C35—C30—C31—C32 | −1.4 (3) |
| S5—C29—C30—C31 | 139.77 (16) | C36—C37—C38—C39 | −179.55 (19) |
| S5—C29—C30—C35 | −41.7 (2) | C36—C37—C42—C41 | −179.97 (18) |
| S6—C36—C37—C38 | 92.23 (19) | C37—C38—C39—C40 | −0.2 (3) |
| S6—C36—C37—C42 | −86.4 (2) | C38—C37—C42—C41 | 1.3 (3) |
| C1—C2—C3—C4 | −179.22 (18) | C38—C39—C40—C41 | 0.9 (3) |
| C1—C2—C7—C6 | 179.76 (18) | C39—C40—C41—C42 | −0.4 (3) |
| C2—C3—C4—C5 | −0.8 (3) | C40—C41—C42—C37 | −0.7 (3) |
| C3—C2—C7—C6 | 0.1 (3) | C42—C37—C38—C39 | −0.9 (3) |
| C3—C4—C5—C6 | 0.6 (3) | O1—C43—C44—C45 | 176.5 (2) |
| C4—C5—C6—C7 | −0.1 (3) | O1—C43—C44—C49 | −1.6 (4) |
| C5—C6—C7—C2 | −0.3 (3) | C43—C44—C45—C43<sup>ii</sup> | 0.8 (4) |
| C7—C2—C3—C4 | 0.5 (3) | C43—C44—C45—C46 | −179.7 (2) |
| C8—C9—C10—C11 | 179.8 (2) | C43—C44—C49—C48 | −178.4 (2) |
| C8—C9—C14—C13 | −179.77 (19) | C43<sup>ii</sup>—C45—C46—C47 | 179.3 (2) |
| C9—C10—C11—C12 | 0.5 (4) | C44—C45—C46—C47 | −0.1 (4) |
| C10—C9—C14—C13 | 1.0 (3) | C45<sup>ii</sup>—C43—C44—C45 | −0.8 (4) |
| C10—C11—C12—C13 | 0.0 (4) | C45<sup>ii</sup>—C43—C44—C49 | −178.9 (2) |
| C11—C12—C13—C14 | 0.0 (4) | C45—C44—C49—C48 | 3.5 (4) |
| C12—C13—C14—C9 | −0.6 (3) | C45—C46—C47—C48 | 0.0 (4) |
| C14—C9—C10—C11 | −1.0 (3) | C46—C47—C48—C49 | 1.8 (4) |
| C15—C16—C17—C18 | −179.22 (18) | C47—C48—C49—C44 | −3.6 (4) |
| C15—C16—C21—C20 | 179.37 (18) | C49—C44—C45—C43<sup>ii</sup> | 178.9 (2) |
| C16—C17—C18—C19 | −0.1 (3) | C49—C44—C45—C46 | −1.7 (3) |
| C17—C16—C21—C20 | −0.9 (3) | | |

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