Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of (μ-1,4-dicarboxybutane-1,4-dicarboxylato)bis[bis(triphenylphosphane)silver(I)] dichloromethane trisolvate

Peter Frenzel, Marcus Korb and Heinrich Lang*

Technische Universität Chemnitz, Fakultät für Naturwissenschaften, Institut für Chemie, Anorganische Chemie, D-09107 Chemnitz. *Correspondence e-mail: heinrich.lang@chemie.tu-chemnitz.de

Received 19 November 2015
Accepted 14 January 2016

Edited by M. Zeller, Youngstown State University, USA

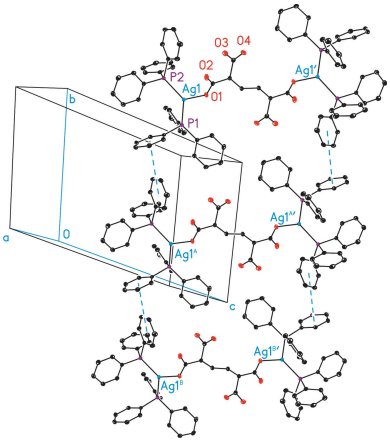
Keywords: crystal structure; silver; triphenylphosphane; tetracarboxylic acid; hydrogen bond; bridges

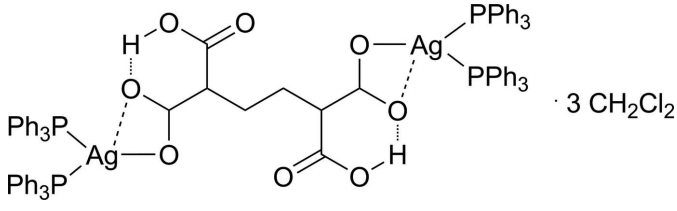
CCDC reference: 1447419
Supporting information: this article has supporting information at journals.iucr.org/e

The molecular structure of the tetrakis(triphenylphosphanyl)disilver salt of butane-1,1,4,4-tetracarboxylic acid, $[Ag_2(C_8H_8O_8)(C_{18}H_{15}P)_4]\cdot 3CH_2Cl_2$, crystallizes with one and a half molecules of dichloromethane in the asymmetric unit. The coordination complex exhibits an inversion centre through the central CH_2-CH_2 bond. The Ag^I atom has a distorted trigonal–planar P_2O coordination environment. The packing is characterized by intermolecular *T*-shaped π–π interactions between the phenyl rings of the PPh_3 substituents in neighbouring molecules, forming a ladder-type superstructure parallel to [010]. These ladders are arranged in layers parallel to (101). Intramolecular hydrogen bonds between the OH group and one O atom of the Ag-bonded carboxylate group results in an asymmetric bidendate coordination of the carboxylate moiety to the Ag^I ion.

1. Chemical context

Silver precursors [*e.g.* silver(I) carboxylates and silver(I) β-diketonates] exhibit a wide range of applications, for instance the formation of thin, metallic layers by means of CVD (Chemical Vapour Deposition) or CCVD (Combustion Chemical Vapour Deposition) (Struppert *et al.*, 2010; Jakob *et al.*, 2006; Schmidt *et al.*, 2005; Lang & Buschbeck, 2009; Lang, 2011; Lang & Dietrich, 2013; Chi & Lu, 2001), spin coating (Jakob *et al.*, 2010) or inkjet printing (Jahn *et al.*, 2010*a*,*b*; Gäbler *et al.*, 2016). The respective silver layers show closed and homogeneous silver films and therefore possess a good conductivity. In addition, silver carboxylates such as $[AgO_2CR]_n$ (n is the degree of aggregation) allow for the formation and stabilization of silver nanoparticles, which can, for example, be used for catalytic processes (Steffan *et al.*, 2009). They are also used in biological studies (Fourie *et al.*, 2012; Langner *et al.*, 2012).





A further application of silver carboxylate precursors includes their use for joining of bulk copper to produce metallic interconnects, for example in microelectronic applications (Oestreicher *et al.*, 2012, 2013).

We anticipate that a metal oxide layer will need to be removed during such a silver-facilitated copper-joining

process. Leaving some of the carboxyl groups of the silver precursor uncoordinated is expected to assist in this process. In the case of sparingly soluble silver carboxylates, the solubility in common organic solvents can be increased through addition of phosphanes, such as triphenyl phosphane. In this context, the title compound (I) was prepared by the reaction of the disilver salt of butane-1,1,4,4-tetracarboxyl acid (BTCA) with triphenylphosphane.

2. Structural commentary

The title compound (I) crystallizes in the triclinic space group $P\overline{1}$. The asymmetric unit contains a half molecule of butane-1,4-dicarboxy-1,4-dicarboxylate bonded to a bis(triphenylphosphanyl)silver moiety and 1.5 molecules of dichloromethane. The inversion centre to build up the whole disilver complex is located in the middle of the C4—C4′ bond (Fig. 1; (A): –x, –y + 1, –z + 2). The three molecules of dichloromethane are also located on or nearby inversion centres (Fig. 1; C2*S*, (B): –x + 1, –y + 1, –z; C1*S*, C1*SB*, –x + 1, –y, –z; see *Refinement* section for details).

The anionic $C_8H_8O_8$ moiety contains an intramolecular hydrogen bond between the O3 atom of the HO_2C-carboxy group and the O2 atom that is in interaction with Ag1 (Fig. 1, Table 1), resulting in a boat-type conformation including the C1, C2 and C3 atoms, due to a synperiplanar torsion of the C1—O2 and C3—O3 bonds by 6.3 (2)°. Within the H-bearing carboxylato substituent a distinction between the C,O single [1.321 (3) Å] and double [1.205 (3) Å] bonds can be observed. The C1 labeled carboxylato group exhibits an unsymmetrically bidendate binding to Ag1. Therefore, O1 is, with 2.3305 (17) Å, σ-bonded, whereas O2 exhibits a weaker interaction with an increased O2···Ag1 distance of 2.6872 (19) Å, probably due to the involvement in the hydrogen bond.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2	0.84	1.79	2.525 (3)	146
C1*S*—H1*S*1···O3^{i}	0.99	2.53	2.997 (4)	108
C1*SB*—H1*S*4···O3^{i}	0.99	2.46	3.04 (4)	117

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

The Ag1 atom exhibits a somewhat distorted trigonal–planar P_2O coordination environment, whereby the two phosphanes enclose an increased P—Ag—P angle of 128.56 (2)°, in contrast to the O1—Ag1—P angles of 117.69 (5) (P1) and 113.27 (5)° (P2). The weak interaction to the O2 atom occurs below this AgP_2 plane with an interaction to the CO_2 group of 67.38 (17)° with, however, two nearly equal C1—O1/O2 bond lengths of 1.251 (3) (O1) and 1.261 (3) Å (O2). Both phosphanes reveal an eclipsed conformation regarding the phenyl rings of 2.09 (10)°.

3. Supramolecular features

The packing of (I) consists of a layer-type structure parallel to (101), which is supported by weak *T*-shaped π–π interactions (Sinnokrot *et al.*, 2002) between the C5–C10 and the C35–C40 labeled phenyl rings with centroid–centroid distances of 4.8497 (16) Å [α = 77.40 (13)°] at both sides of the molecules, forming a ladder-type arrangement parallel to [010] (Fig. 2). These ladders are packed along (101) through the phenyl rings, however, without showing any further π–π or C—H interactions.

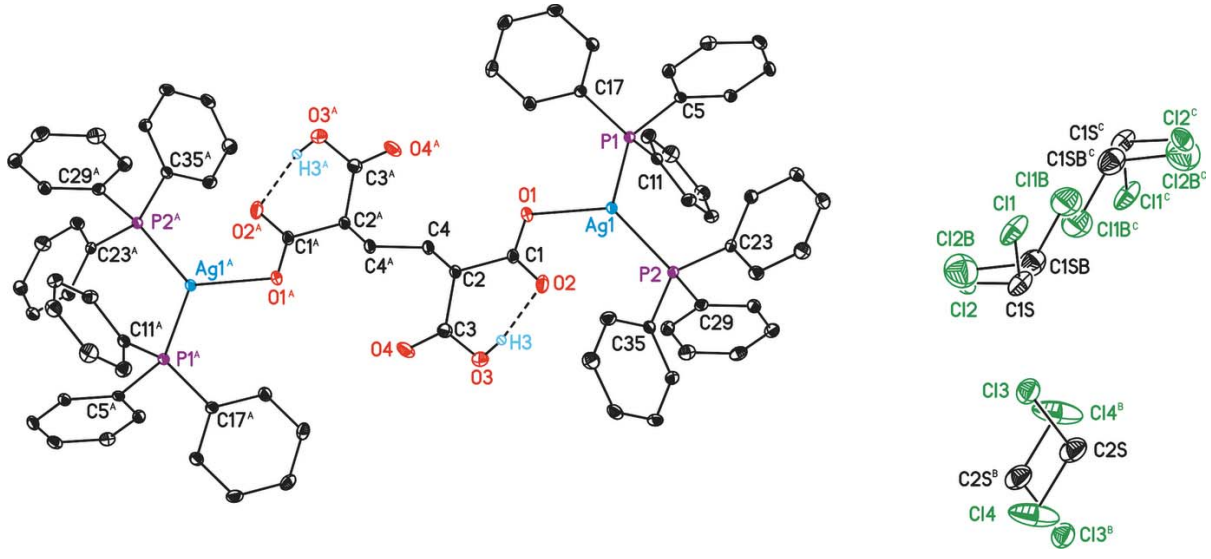


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level, including the intramolecular hydrogen bonds. All non-O-bonded H atoms and the labels of the *o*-, *m*- and *p*-phenyl C atoms have been omitted for clarity. [Symmetry codes: (A) $-x, -y+1, -z+2$; (B) $-x+1, -y+1, -z$; (C) $-x+1, -y, -z$.]

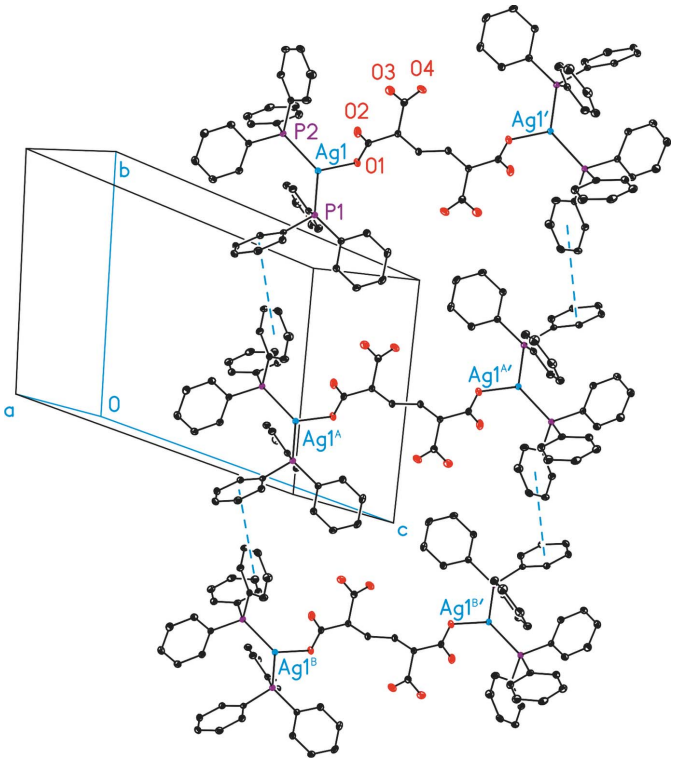


Figure 2
Intermolecular *T*-shaped π–π interactions (blue) between the centroids (D) of the C5–C10 and C35–C40 labeled phenyl rings in the crystal structure of (I). All H atoms and solvent molecules have been omitted for clarity. D—D = 4.8497 (16) Å; α = 77.40 (13) °. [Symmetry codes: (′) $-x$, $-y+1$, $-z+2$; (A) x, $\frac{1}{2}-y$, $z-\frac{1}{2}$; (B) x, y, $x-1$.]

One dichloromethane is stabilized by a non-classical hydrogen-bridge bond from C1*S*, as the hydrogen-bond donor, to the hydroxyl O3 atom (Table 1), which also acts as hydrogen-bridge bond donor in an intramolecular classical bridge bond (see *Structural commentary*).

Further intermolecular interactions involving hydrogen bonds or O···Ag interactions are not observed.

4. Database survey

In the CSD database (Groom & Allen, 2014; Version 5.36), only two acyclic silver tetracarboxylates with six-membered carbon backbones are reported. These are butane-1,2,3,4-tetracarboxylato silver compounds containing further nitrogen and oxygen donor ligands, coordinating the silver ions either in a tetrahedral or a *T*-shaped trigonal fashion (Sun *et al.*, 2010). Three aliphatic cyclohexane silver complexes with four to six carboxylate groups are also known. Within those, the silver ions are also coordinated by additional ligands such as ammonia and water and possess distorted tetrahedral coordination or *Y*-shaped coordination environments (Wang *et al.*, 2006, 2009). For six-membered unbranched acyclic silver dicarboxylates derived from adipic acid, more crystal structures are reported compared to the respective tetracarboxylato derivatives. Several coordination geometries for the silver atoms are reported such as *T*-shaped (Wu *et al.*, 1995), tetrahedral (Li *et al.*, 2011) or trigonal–planar environments (Liu *et al.*, 2009) containing nitrogen, oxygen or sulfur donor ligands. To the best of our knowledge, the title compound (I) is the only example of a silver tetracarboxylate consisting of a six-membered carbon backbone and containing a silver–phosphorus bond. In contrast to the title compound, which exists as a monomer presumably due to the steric shielding by triphenyl phosphane, all of the above-mentioned complexes exist as polymeric networks, formed by bridging through the different donor atoms of the ligands. For example, by using silver dicarboxylates frequently the formation of dimeric subunits can take place, which in turn results in the construction of polymeric systems (Wu *et al.*, 1995). Structures containing water molecules coordinating to the Ag^{I} ions result in the formation of a further polymeric hydrogen bridge-bond network, also including carboxylato moieties (Wang *et al.*, 2006, 2009).

5. Synthesis and crystallization

Synthesis of butane-1,4-dicarboxyl-1,4-dicarboxylatodisilver(I):

Potassium *tert*-butanolate (192 mg, 1.71 mmol) was added to a solution of butane-1,1,4,4-tetracarboxyl acid (200 mg, 0.854 mmol) in 5 mL of tetrahydrofuran. After stirring overnight at ambient temperature, the reaction mixture was filtered off and the residue was washed trice with tetrahydrofuran (10 mL each) and dried under vacuum (yield 243 mg). Subsequently, the obtained colorless solid (243 mg, 0.783 mmol) was dissolved in water (15 mL) and added dropwise to a solution of silver nitrate (267 mg, 1.57 mmol) in water (8 mL). After 12 h of stirring the colorless precipitate was filtered off and washed twice with water (10 mL each) and dried in a desiccator. The desired colorless butane-1,4-dicarboxyl-1,4-dicarboxylatodisilver(I) was obtained in a yield of 71%, based on butane-1,1,4,4-tetracarboxyl acid (271 mg, 0.608 mmol). Analysis calculated for $C_8H_8Ag_2O_8$ (447.88): C, 21.45; H, 1.80. Found: C, 21.49; H, 1.68. IR (KBr, cm^{-1}): ν = 2977 (*m*), 2903 (*m*), 2461 (*m*), 1660 (*s*), 1549 (*vs*), 1380 (*s*), 1257 (*s*), 1069 (*s*), 955 (*m*), 711 (*m*).

Synthesis of (μ-1,4-dicarboxybutane-1,4-dicarboxylato)bis[bis(triphenylphosphane)silver(I)]:

To a suspension of butane-1,4-dicarboxyl-1,4-dicarboxylatodisilver(I) (100 mg, 0.223 mmol) in 10 mL of tetrahydrofuran, PPh_3 (234 mg, 0.892 mmol) was added in a single portion at ambient temperature. After 12 h of stirring the reaction mixture was filtered through a pad of celite. After removal of all volatiles under reduced pressure, the title compound (I) was obtained as a colorless solid (275 mg, 0.184 mmol, 83% based on butane-1,4-dicarboxyl-1,4-dicarboxylatodisilver(I). Slow diffusion of pentane into a dichloromethane solution containing (I) at ambient temperature afforded colourless crystals of (I). M.p. 398 K (decomp.). ^{1}H NMR (500 MHz, $CDCl_3$, 298 K, p.p.m.): δ = 7.39–7.29 (*m*, 60H, C_6H_5), 2.93 (*m*, 2H, CH), 2.04 (*m*, 4H, CH_2). $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$, 298 K, p.p.m.): δ = 175.9 (*s*, C═O), 134.0 (*d*, $^2J_{PC}$ = 16.1 Hz, *o*-C_6H_5), 131.7 (*d*, $^1J_{PC}$ = 29.3 Hz, C^i-

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Ag_2(C_8H_8O_8)(C_{18}H_{15}P)_4]\cdot 3CH_2Cl_2$
M_r	1751.74
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	110
a, b, c (Å)	10.0279 (3), 12.9540 (4), 16.8190 (5)
α, β, γ (°)	112.306 (3), 96.080 (3), 103.601 (3)
V (Å^3)	1917.80 (11)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.86
Crystal size (mm)	0.3 × 0.3 × 0.2
Data collection	
Diffractometer	Oxford Gemini S
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min}, T_{max}	0.889, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21441, 8700, 7959
R_{int}	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.680
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.083, 1.06
No. of reflections	8700
No. of parameters	507
No. of restraints	27
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	2.28, −0.61

Computer programs: *CrysAlis CCD* (Oxford Diffraction, 2006), *CrysAlis RED* (Oxford Diffraction, 2006), *SHELXS2013* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *SHELXTL* (Sheldrick, 2008), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

C_6H_5), 130.6 (*s*, *p*-C_6H_5), 129.1 (*d*, $^3J_{PC}$ = 9.3 Hz, *m*-C_6H_5), 50.8 (*s*, CH), 29.6 (*s*, CH_2). $^{31}P\{^1H\}$ NMR (203 MHz, $CDCl_3$, 298 K, p.p.m.): δ = 10.3 (*s*). IR (KBr, cm^{-1}): ν = 3108 (*w*), 2993 (*w*), 1751 (*s*), 1494 (*vs*), 1106 (*s*), 752 (*vs*), 701 (*vs*).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bonded hydrogen atoms were placed in calculated positions and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C) and a C—H distance of 0.93 Å for aromatic (AFIX 43), 0.98 Å for methine (AFIX 13) and 0.97 Å for methylene H atoms (AFIX 23). The same applies for the O-bonded H atom; however, the torsion angle was derived from electron density (AFIX 147). The structure contains three molecules of dichloromethane as the solvent. Both crystallographically independent molecules consist of two moieties each. One molecule was refined as disordered over two positions (C1*S*; C1*SB*) with occupancies of 92.7 (2) and 7.3 (3)%, respectively. The less prevalent moiety of C1*SB* is located close to a crystallographic inversion centre and symmetry-related pairs are mutually exclusive. The second disordered molecule is located directly atop of another inversion centre with an occupancy of 0.5 (Fig. 1), with the inversion centre located near the C2*S* and Cl1*B* atoms. The less-occupied methylene chloride molecule was restrained to have a geometry similar to that of its major moiety counterpart by using the SAME command. U_{ij} components of ADPs for C1*S* C1*SB* Cl1*B* and Cl2*B* were restrained to be similar if closer than 1.7 Å (SIMU restraint, McArdle, 1995; Sheldrick, 2008).

Acknowledgements

MK thanks the Fonds der Chemischen Industrie for a PhD Chemiefonds fellowship. This work was performed within the Federal Cluster of Excellence EXC 1075 MERGE Technologies for Multifunctional Lightweight Structures and supported by the German Research Foundation (DFG). Financial support is gratefully acknowledged.

References

Chi, K. M. & Lu, Y. H. (2001). *Chem. Vap. Deposition*, **7**, 117–120.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Fourie, E., Erasmus, E., Swarts, J. C., Tuchscherer, A., Jakob, A., Lang, H., Joone, G. K. & Van Rensburg, C. E. (2012). *Anticancer Res.* **32**, 519–522.
Gäbler, C., Schliebe, C., Adner, D., Blaudeck, T. & Lang, H. (2016). *Inkjet Printing of Group-11 Metal Structures*, in *Comprehensive Guide for Nanocoatings Technology*. New York: Nova Science Publishers. In the press.
Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
Jahn, S. F., Blaudeck, T., Baumann, R. R., Jakob, A., Ecorchard, P., Rüffer, T., Lang, H. & Schmidt, P. (2010*a*). *Chem. Mater.* **22**, 3067–3071.
Jahn, S. F., Jakob, A., Blaudeck, T., Schmidt, P., Lang, H. & Baumann, R. R. (2010*b*). *Thin Solid Films*, **518**, 3218–3222.
Jakob, A., Rüffer, T., Schmidt, H., Djiele, P., Körbitz, K., Ecorchard, P., Haase, T., Kohse-Höinghaus, K., Frühauf, S., Wächtler, T., Schulz, S., Gessner, T. & Lang, H. (2010). *Eur. J. Inorg. Chem.* pp. 2975–2986.
Jakob, A., Schmidt, H., Djiele, P., Shen, Y. & Lang, H. (2006). *Microchim. Acta*, **156**, 77–81.
Lang, H. (2011). *Jordan. J. Chem.* **6**, 231–245.
Lang, H. & Buschbeck, R. (2009). *Deposition of metals and metal oxides by means of metal enolates*, in *The Chemistry of Metal Enolates*, edited by J. Zabicky, pp. 929–1017. Chichester: Wiley.
Lang, H. & Dietrich, S. (2013). *4.10 – Metals – Gas-Phase Deposition and Applications*, in *Comprehensive Inorganic Chemistry II (Second Edition)*, edited by J. Reedijk & K. Poeppelmeier, pp. 211–269. Amsterdam: Elsevier.
Langner, E. H. G., Swarts, J. C., Tuchscherer, A., Lang, H., Joone, G. K. & van Rensburg, C. E. J. (2012). *Anticancer Res.* **32**, 2697–2701.
Li, Y.-H., Sun, D., Luo, G.-G., Liu, F.-J., Hao, H.-J., Wen, Y.-M., Zhao, Y., Huang, R.-B. & Zheng, L.-S. (2011). *J. Mol. Struct.* **1000**, 85–91.
Liu, S., Zhang, X., Wang, H., Meng, C. & Mou, W. (2009). *Chin. J. Chem.* **27**, 722–726.
McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
Oestreicher, A., Röhrich, T. & Lerch, M. (2012). *J. Nanosci. Nanotechnol.* **12**, 9088–9093.
Oestreicher, A., Röhrich, T., Wilden, J., Lerch, M., Jakob, A. & Lang, H. (2013). *Appl. Surf. Sci.* **265**, 239–244.
Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction, Abingdon, England.
Schmidt, H., Jakob, A., Haase, T., Kohse-Höinghaus, K., Schulz, S. E., Wächtler, T., Gessner, T. & Lang, H. (2005). *Z. Anorg. Allg. Chem.* **631**, 2786–2791.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Sinnokrot, M. O., Valeev, E. F. & Sherrill, C. D. (2002). *J. Am. Chem. Soc.* **124**, 10887–10893.
Steffan, M., Jakob, A., Claus, P. & Lang, H. (2009). *Catal. Commun.* **10**, 437–441.
Struppert, T., Jakob, A., Heft, A., Grünler, B. & Lang, H. (2010). *Thin Solid Films*, **518**, 5741–5744.
Sun, D., Zhang, N., Xu, Q.-J., Huang, R.-B. & Zheng, L.-S. (2010). *J. Mol. Struct.* **975**, 17–22.
Wang, J., Hu, S. & Tong, M.-L. (2006). *Eur. J. Inorg. Chem.* **2006**, 2069–2077.
Wang, J., Ou, Y.-C., Shen, Y., Yun, L., Leng, J.-D., Lin, Z. & Tong, M.-L. (2009). *Cryst. Growth Des.* **9**, 2442–2450.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Wu, D.-D. & Mak, T. C. W. (1995). *J. Chem. Soc. Dalton Trans.* pp. 2671–2678.

supporting information

Acta Cryst. (2016). E**72**, 215-219 [doi:10.1107/S2056989016000797]

Crystal structure of (μ-1,4-dicarboxybutane-1,4-dicarboxylato)bis[bis(triphenylphosphane)silver(I)] dichloromethane trisolvate

Peter Frenzel, Marcus Korb and Heinrich Lang

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 200); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

(μ-1,4-Dicarboxybutane-1,4-dicarboxylato)bis[bis(triphenylphosphane)silver(I)] dichloromethane trisolvate

Crystal data

$[Ag_2(C_8H_8O_8)(C_{18}H_{15}P)_4]\cdot 3CH_2Cl_2$
M_r = 1751.74
Triclinic, $P\overline{1}$
a = 10.0279 (3) Å
b = 12.9540 (4) Å
c = 16.8190 (5) Å
α = 112.306 (3)°
β = 96.080 (3)°
γ = 103.601 (3)°
V = 1917.80 (11) Å^3
Z = 1
$F(000)$ = 892
D_x = 1.517 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 12088 reflections
θ = 3.4–28.8°
μ = 0.86 mm^{-1}
T = 110 K
Block, colorless
0.3 × 0.3 × 0.2 mm

Data collection

Oxford Gemini S diffractometer
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.889, T_{max} = 1.000
21441 measured reflections
8700 independent reflections
7959 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.9°, θ_{min} = 2.9°
h = −13→12
k = −17→17
l = −22→20
2 standard reflections every 50 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.083
S = 1.06
8700 reflections
507 parameters
27 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
$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 2.6912P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.28$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.1098 (3)	0.4419 (2)	0.82508 (15)	0.0190 (5)	
C2	0.0325 (2)	0.5106 (2)	0.88968 (15)	0.0174 (4)	
H2	−0.0699	0.4812	0.8615	0.021*	
C3	0.0822 (3)	0.6429 (2)	0.91901 (16)	0.0247 (5)	
C4	0.0521 (3)	0.4833 (2)	0.97147 (15)	0.0189 (5)	
H4A	0.0402	0.3987	0.9518	0.023*	
H4B	0.1489	0.5265	1.0070	0.023*	
C5	0.2398 (2)	−0.00432 (19)	0.61984 (14)	0.0158 (4)	
C6	0.1010 (3)	−0.0530 (2)	0.57113 (16)	0.0204 (5)	
H6	0.0276	−0.0297	0.5975	0.024*	
C7	0.0703 (3)	−0.1351 (2)	0.48448 (16)	0.0232 (5)	
H7	−0.0241	−0.1678	0.4518	0.028*	
C8	0.1774 (3)	−0.1698 (2)	0.44511 (16)	0.0243 (5)	
H8	0.1562	−0.2258	0.3857	0.029*	
C9	0.3145 (3)	−0.1225 (2)	0.49301 (16)	0.0231 (5)	
H9	0.3875	−0.1461	0.4663	0.028*	
C10	0.3465 (3)	−0.0403 (2)	0.58043 (16)	0.0193 (5)	
H10	0.4409	−0.0088	0.6131	0.023*	
C11	0.4578 (2)	0.1469 (2)	0.77394 (15)	0.0173 (4)	
C12	0.5538 (3)	0.2188 (2)	0.74783 (17)	0.0230 (5)	
H12	0.5203	0.2509	0.7103	0.028*	
C13	0.6972 (3)	0.2435 (2)	0.77632 (17)	0.0256 (5)	
H13	0.7616	0.2911	0.7575	0.031*	
C14	0.7467 (3)	0.1989 (2)	0.83205 (18)	0.0280 (6)	
H14	0.8450	0.2153	0.8512	0.034*	
C15	0.6527 (3)	0.1299 (3)	0.86010 (19)	0.0314 (6)	
H15	0.6869	0.1010	0.8997	0.038*	
C16	0.5089 (3)	0.1029 (2)	0.83059 (17)	0.0251 (5)	
H16	0.4451	0.0544	0.8491	0.030*	
C17	0.1720 (2)	0.0459 (2)	0.78922 (15)	0.0172 (4)	
C18	0.1697 (3)	−0.0662 (2)	0.78067 (16)	0.0222 (5)	
H18	0.2230	−0.1071	0.7440	0.027*	
C19	0.0898 (3)	−0.1177 (2)	0.82571 (17)	0.0269 (5)	
H19	0.0880	−0.1940	0.8196	0.032*	
C20	0.0124 (3)	−0.0581 (2)	0.87957 (17)	0.0266 (5)	
H20	−0.0423	−0.0938	0.9103	0.032*	

C21	0.0144 (3)	0.0534 (2)	0.88891 (17)	0.0256 (5)	
H21	−0.0382	0.0942	0.9263	0.031*	
C22	0.0939 (3)	0.1055 (2)	0.84336 (16)	0.0215 (5)	
H22	0.0948	0.1816	0.8493	0.026*	
C23	0.2912 (2)	0.2770 (2)	0.50087 (15)	0.0162 (4)	
C24	0.2945 (3)	0.1622 (2)	0.47850 (16)	0.0207 (5)	
H24	0.3061	0.1354	0.5234	0.025*	
C25	0.2812 (3)	0.0869 (2)	0.39126 (17)	0.0239 (5)	
H25	0.2846	0.0094	0.3768	0.029*	
C26	0.2629 (3)	0.1250 (2)	0.32544 (17)	0.0259 (5)	
H26	0.2542	0.0739	0.2658	0.031*	
C27	0.2574 (3)	0.2385 (2)	0.34706 (17)	0.0276 (6)	
H27	0.2436	0.2644	0.3019	0.033*	
C28	0.2719 (3)	0.3141 (2)	0.43393 (16)	0.0220 (5)	
H28	0.2686	0.3917	0.4480	0.026*	
C29	0.5028 (2)	0.4324 (2)	0.65811 (15)	0.0175 (4)	
C30	0.5974 (3)	0.4021 (2)	0.60549 (16)	0.0212 (5)	
H30	0.5636	0.3484	0.5454	0.025*	
C31	0.7417 (3)	0.4499 (2)	0.64032 (18)	0.0245 (5)	
H31	0.8060	0.4283	0.6041	0.029*	
C32	0.7910 (3)	0.5285 (2)	0.72730 (18)	0.0269 (5)	
H32	0.8893	0.5623	0.7507	0.032*	
C33	0.6974 (3)	0.5585 (2)	0.78088 (17)	0.0285 (6)	
H33	0.7319	0.6123	0.8409	0.034*	
C34	0.5535 (3)	0.5101 (2)	0.74688 (16)	0.0240 (5)	
H34	0.4896	0.5297	0.7838	0.029*	
C35	0.2611 (2)	0.4971 (2)	0.61527 (14)	0.0171 (4)	
C36	0.1241 (3)	0.4982 (2)	0.61812 (18)	0.0252 (5)	
H36	0.0602	0.4370	0.6253	0.030*	
C37	0.0796 (3)	0.5889 (2)	0.6105 (2)	0.0303 (6)	
H37	−0.0151	0.5885	0.6114	0.036*	
C38	0.1724 (3)	0.6791 (2)	0.60168 (16)	0.0253 (5)	
H38	0.1413	0.7401	0.5955	0.030*	
C39	0.3101 (3)	0.6805 (2)	0.60184 (17)	0.0248 (5)	
H39	0.3749	0.7438	0.5977	0.030*	
C40	0.3546 (3)	0.5896 (2)	0.60809 (17)	0.0223 (5)	
H40	0.4495	0.5906	0.6075	0.027*	
P1	0.27220 (6)	0.11490 (5)	0.72910 (4)	0.01518 (12)	
P2	0.31285 (6)	0.37213 (5)	0.61693 (4)	0.01519 (12)	
Ag1	0.22109 (2)	0.27730 (2)	0.70903 (2)	0.01622 (6)	
O1	0.04100 (19)	0.34138 (15)	0.76927 (11)	0.0239 (4)	
O2	0.23936 (19)	0.48772 (17)	0.83418 (12)	0.0301 (4)	
O3	0.2146 (2)	0.68777 (18)	0.91847 (15)	0.0409 (5)	
H3	0.2542	0.6350	0.9052	0.061*	
O4	0.0070 (2)	0.70357 (16)	0.94320 (13)	0.0333 (4)	
C1S	0.4951 (4)	0.1726 (4)	0.0721 (3)	0.0525 (11)	0.928 (2)
H1S1	0.5689	0.1417	0.0457	0.063*	0.928 (2)
H1S2	0.4983	0.2447	0.0638	0.063*	0.928 (2)

Cl1	0.33022 (9)	0.06889 (12)	0.01685 (7)	0.0647 (4)	0.928 (2)
Cl2	0.53101 (12)	0.20717 (8)	0.18454 (6)	0.0474 (3)	0.928 (2)
C1SB	0.525 (5)	0.125 (3)	0.069 (3)	0.069 (7)	0.072 (2)
H1S3	0.6161	0.1301	0.1016	0.083*	0.072 (2)
H1S4	0.5460	0.1770	0.0381	0.083*	0.072 (2)
Cl1B	0.451 (2)	−0.0184 (18)	−0.0110 (13)	0.088 (6)	0.072 (2)
Cl2B	0.429 (3)	0.183 (2)	0.1455 (16)	0.112 (7)	0.072 (2)
C2S	0.5061 (9)	0.4570 (8)	−0.0394 (6)	0.060 (2)	0.5
H2S1	0.4788	0.4427	−0.1019	0.072*	0.5
H2S2	0.5940	0.4361	−0.0319	0.072*	0.5
Cl3	0.3762 (2)	0.3690 (2)	−0.01470 (16)	0.0548 (5)	0.5
Cl4	0.5373 (5)	0.6054 (2)	0.0271 (3)	0.1237 (17)	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0254 (12)	0.0211 (12)	0.0169 (11)	0.0116 (9)	0.0102 (9)	0.0105 (9)
C2	0.0230 (11)	0.0167 (11)	0.0149 (10)	0.0077 (9)	0.0082 (9)	0.0070 (9)
C3	0.0380 (14)	0.0213 (13)	0.0158 (11)	0.0076 (11)	0.0087 (10)	0.0087 (10)
C4	0.0236 (11)	0.0206 (12)	0.0174 (11)	0.0112 (9)	0.0094 (9)	0.0092 (9)
C5	0.0237 (11)	0.0118 (10)	0.0156 (10)	0.0066 (9)	0.0074 (9)	0.0080 (8)
C6	0.0230 (12)	0.0206 (12)	0.0214 (11)	0.0085 (9)	0.0081 (9)	0.0109 (10)
C7	0.0250 (12)	0.0215 (12)	0.0210 (12)	0.0037 (10)	0.0011 (9)	0.0097 (10)
C8	0.0388 (14)	0.0169 (12)	0.0179 (11)	0.0099 (10)	0.0077 (10)	0.0067 (9)
C9	0.0336 (13)	0.0204 (12)	0.0227 (12)	0.0149 (10)	0.0149 (10)	0.0105 (10)
C10	0.0239 (12)	0.0166 (11)	0.0216 (11)	0.0089 (9)	0.0086 (9)	0.0098 (9)
C11	0.0211 (11)	0.0149 (11)	0.0165 (10)	0.0072 (9)	0.0057 (9)	0.0056 (9)
C12	0.0258 (12)	0.0201 (12)	0.0247 (12)	0.0061 (10)	0.0051 (10)	0.0117 (10)
C13	0.0241 (12)	0.0200 (12)	0.0304 (13)	0.0032 (10)	0.0086 (10)	0.0092 (10)
C14	0.0210 (12)	0.0267 (14)	0.0312 (14)	0.0076 (10)	0.0020 (10)	0.0074 (11)
C15	0.0312 (14)	0.0380 (16)	0.0314 (14)	0.0139 (12)	0.0034 (11)	0.0200 (13)
C16	0.0264 (13)	0.0275 (13)	0.0270 (13)	0.0088 (10)	0.0069 (10)	0.0164 (11)
C17	0.0218 (11)	0.0156 (11)	0.0163 (10)	0.0055 (9)	0.0061 (9)	0.0084 (9)
C18	0.0327 (13)	0.0187 (12)	0.0195 (11)	0.0104 (10)	0.0103 (10)	0.0096 (9)
C19	0.0390 (15)	0.0187 (12)	0.0241 (12)	0.0046 (11)	0.0091 (11)	0.0120 (10)
C20	0.0293 (13)	0.0307 (14)	0.0224 (12)	0.0039 (11)	0.0087 (10)	0.0160 (11)
C21	0.0269 (13)	0.0335 (14)	0.0223 (12)	0.0117 (11)	0.0117 (10)	0.0148 (11)
C22	0.0258 (12)	0.0214 (12)	0.0227 (12)	0.0116 (10)	0.0094 (10)	0.0110 (10)
C23	0.0171 (10)	0.0168 (11)	0.0170 (10)	0.0056 (8)	0.0077 (8)	0.0080 (9)
C24	0.0273 (12)	0.0197 (12)	0.0211 (11)	0.0100 (10)	0.0094 (9)	0.0120 (10)
C25	0.0299 (13)	0.0182 (12)	0.0257 (12)	0.0095 (10)	0.0107 (10)	0.0087 (10)
C26	0.0306 (13)	0.0236 (13)	0.0195 (12)	0.0069 (10)	0.0071 (10)	0.0052 (10)
C27	0.0398 (15)	0.0277 (14)	0.0190 (12)	0.0108 (11)	0.0060 (11)	0.0134 (10)
C28	0.0286 (13)	0.0197 (12)	0.0216 (12)	0.0094 (10)	0.0064 (10)	0.0112 (10)
C29	0.0198 (11)	0.0173 (11)	0.0217 (11)	0.0075 (9)	0.0062 (9)	0.0131 (9)
C30	0.0235 (12)	0.0199 (12)	0.0240 (12)	0.0098 (10)	0.0081 (10)	0.0106 (10)
C31	0.0212 (12)	0.0256 (13)	0.0338 (14)	0.0100 (10)	0.0094 (10)	0.0173 (11)
C32	0.0223 (12)	0.0292 (14)	0.0342 (14)	0.0052 (10)	0.0022 (10)	0.0209 (12)

C33	0.0331 (14)	0.0281 (14)	0.0207 (12)	0.0033 (11)	0.0002 (10)	0.0116 (11)
C34	0.0286 (13)	0.0253 (13)	0.0215 (12)	0.0087 (10)	0.0088 (10)	0.0121 (10)
C35	0.0247 (12)	0.0156 (11)	0.0150 (10)	0.0091 (9)	0.0077 (9)	0.0080 (9)
C36	0.0249 (12)	0.0216 (13)	0.0360 (14)	0.0094 (10)	0.0107 (11)	0.0167 (11)
C37	0.0270 (13)	0.0295 (14)	0.0432 (16)	0.0162 (11)	0.0109 (12)	0.0190 (12)
C38	0.0389 (14)	0.0198 (12)	0.0222 (12)	0.0156 (11)	0.0084 (11)	0.0096 (10)
C39	0.0366 (14)	0.0165 (12)	0.0266 (13)	0.0087 (10)	0.0117 (11)	0.0127 (10)
C40	0.0264 (12)	0.0196 (12)	0.0271 (12)	0.0095 (10)	0.0121 (10)	0.0132 (10)
P1	0.0194 (3)	0.0141 (3)	0.0162 (3)	0.0073 (2)	0.0073 (2)	0.0084 (2)
P2	0.0195 (3)	0.0148 (3)	0.0162 (3)	0.0079 (2)	0.0082 (2)	0.0089 (2)
Ag1	0.02215 (9)	0.01473 (9)	0.01672 (9)	0.00874 (7)	0.00930 (6)	0.00856 (7)
O1	0.0280 (9)	0.0196 (9)	0.0237 (9)	0.0090 (7)	0.0139 (7)	0.0053 (7)
O2	0.0226 (9)	0.0352 (11)	0.0260 (9)	0.0061 (8)	0.0114 (7)	0.0060 (8)
O3	0.0440 (12)	0.0228 (10)	0.0449 (12)	−0.0002 (9)	0.0212 (10)	0.0058 (9)
O4	0.0535 (13)	0.0207 (10)	0.0303 (10)	0.0192 (9)	0.0126 (9)	0.0100 (8)
C1S	0.040 (2)	0.069 (3)	0.046 (2)	−0.0016 (19)	0.0037 (16)	0.034 (2)
Cl1	0.0314 (5)	0.1057 (9)	0.0486 (6)	−0.0137 (5)	−0.0060 (4)	0.0476 (6)
Cl2	0.0634 (6)	0.0420 (5)	0.0313 (4)	0.0193 (4)	0.0035 (4)	0.0091 (4)
C1SB	0.063 (9)	0.077 (9)	0.062 (9)	0.007 (9)	0.008 (8)	0.033 (9)
Cl1B	0.124 (15)	0.080 (11)	0.075 (10)	0.032 (12)	0.019 (11)	0.047 (9)
Cl2B	0.101 (11)	0.108 (11)	0.106 (11)	0.021 (10)	0.014 (10)	0.029 (10)
C2S	0.044 (4)	0.063 (6)	0.082 (6)	0.011 (4)	0.023 (4)	0.041 (5)
Cl3	0.0507 (11)	0.0616 (14)	0.0619 (12)	0.0195 (10)	0.0221 (10)	0.0321 (11)
Cl4	0.165 (4)	0.0433 (15)	0.109 (3)	−0.009 (2)	−0.068 (3)	0.0238 (16)

Geometric parameters (Å, º)

C1—O1	1.251 (3)	C24—C25	1.390 (3)
C1—O2	1.261 (3)	C24—H24	0.9500
C1—C2	1.531 (3)	C25—C26	1.385 (4)
C2—C3	1.527 (3)	C25—H25	0.9500
C2—C4	1.550 (3)	C26—C27	1.390 (4)
C2—H2	1.0000	C26—H26	0.9500
C3—O4	1.205 (3)	C27—C28	1.385 (4)
C3—O3	1.321 (3)	C27—H27	0.9500
C4—C4^{i}	1.520 (4)	C28—H28	0.9500
C4—H4A	0.9900	C29—C30	1.388 (3)
C4—H4B	0.9900	C29—C34	1.399 (3)
C5—C10	1.397 (3)	C29—P2	1.825 (2)
C5—C6	1.400 (3)	C30—C31	1.395 (3)
C5—P1	1.831 (2)	C30—H30	0.9500
C6—C7	1.387 (3)	C31—C32	1.379 (4)
C6—H6	0.9500	C31—H31	0.9500
C7—C8	1.395 (4)	C32—C33	1.389 (4)
C7—H7	0.9500	C32—H32	0.9500
C8—C9	1.382 (4)	C33—C34	1.389 (4)
C8—H8	0.9500	C33—H33	0.9500
C9—C10	1.396 (3)	C34—H34	0.9500

C9—H9	0.9500	C35—C36	1.384 (3)
C10—H10	0.9500	C35—C40	1.391 (3)
C11—C16	1.393 (3)	C35—P2	1.821 (2)
C11—C12	1.402 (3)	C36—C37	1.394 (4)
C11—P1	1.819 (2)	C36—H36	0.9500
C12—C13	1.386 (4)	C37—C38	1.380 (4)
C12—H12	0.9500	C37—H37	0.9500
C13—C14	1.381 (4)	C38—C39	1.376 (4)
C13—H13	0.9500	C38—H38	0.9500
C14—C15	1.389 (4)	C39—C40	1.389 (3)
C14—H14	0.9500	C39—H39	0.9500
C15—C16	1.389 (4)	C40—H40	0.9500
C15—H15	0.9500	P1—Ag1	2.4109 (6)
C16—H16	0.9500	P2—Ag1	2.4433 (6)
C17—C22	1.393 (3)	Ag1—O1	2.3305 (17)
C17—C18	1.398 (3)	O3—H3	0.8400
C17—P1	1.819 (2)	C1S—Cl2	1.744 (4)
C18—C19	1.387 (3)	C1S—Cl1	1.756 (4)
C18—H18	0.9500	C1S—H1S1	0.9900
C19—C20	1.385 (4)	C1S—H1S2	0.9900
C19—H19	0.9500	C1SB—Cl2B	1.73 (2)
C20—C21	1.387 (4)	C1SB—Cl1B	1.75 (2)
C20—H20	0.9500	C1SB—H1S3	0.9900
C21—C22	1.395 (3)	C1SB—H1S4	0.9900
C21—H21	0.9500	C2S—Cl3	1.716 (8)
C22—H22	0.9500	C2S—Cl4	1.748 (10)
C23—C28	1.395 (3)	C2S—H2S1	0.9900
C23—C24	1.398 (3)	C2S—H2S2	0.9900
C23—P2	1.826 (2)		

O1—C1—O2	124.4 (2)	C25—C26—C27	119.7 (2)
O1—C1—C2	117.4 (2)	C25—C26—H26	120.2
O2—C1—C2	118.1 (2)	C27—C26—H26	120.2
C3—C2—C1	115.15 (19)	C28—C27—C26	120.5 (2)
C3—C2—C4	109.22 (19)	C28—C27—H27	119.7
C1—C2—C4	106.96 (18)	C26—C27—H27	119.7
C3—C2—H2	108.4	C27—C28—C23	120.3 (2)
C1—C2—H2	108.4	C27—C28—H28	119.8
C4—C2—H2	108.4	C23—C28—H28	119.8
O4—C3—O3	121.6 (2)	C30—C29—C34	119.4 (2)
O4—C3—C2	122.5 (2)	C30—C29—P2	122.57 (19)
O3—C3—C2	115.8 (2)	C34—C29—P2	118.00 (18)
C4^{i}—C4—C2	112.2 (2)	C29—C30—C31	120.4 (2)
C4^{i}—C4—H4A	109.2	C29—C30—H30	119.8
C2—C4—H4A	109.2	C31—C30—H30	119.8
C4^{i}—C4—H4B	109.2	C32—C31—C30	119.9 (2)
C2—C4—H4B	109.2	C32—C31—H31	120.0
H4A—C4—H4B	107.9	C30—C31—H31	120.0

C10—C5—C6	119.2 (2)	C31—C32—C33	120.2 (2)
C10—C5—P1	123.58 (18)	C31—C32—H32	119.9
C6—C5—P1	116.88 (17)	C33—C32—H32	119.9
C7—C6—C5	120.2 (2)	C32—C33—C34	120.1 (2)
C7—C6—H6	119.9	C32—C33—H33	119.9
C5—C6—H6	119.9	C34—C33—H33	119.9
C6—C7—C8	120.3 (2)	C33—C34—C29	119.9 (2)
C6—C7—H7	119.8	C33—C34—H34	120.0
C8—C7—H7	119.8	C29—C34—H34	120.0
C9—C8—C7	119.7 (2)	C36—C35—C40	119.1 (2)
C9—C8—H8	120.2	C36—C35—P2	119.20 (18)
C7—C8—H8	120.2	C40—C35—P2	121.65 (18)
C8—C9—C10	120.5 (2)	C35—C36—C37	120.1 (2)
C8—C9—H9	119.8	C35—C36—H36	119.9
C10—C9—H9	119.8	C37—C36—H36	119.9
C9—C10—C5	120.0 (2)	C38—C37—C36	120.3 (2)
C9—C10—H10	120.0	C38—C37—H37	119.8
C5—C10—H10	120.0	C36—C37—H37	119.8
C16—C11—C12	118.9 (2)	C39—C38—C37	119.8 (2)
C16—C11—P1	124.05 (18)	C39—C38—H38	120.1
C12—C11—P1	117.01 (18)	C37—C38—H38	120.1
C13—C12—C11	120.5 (2)	C38—C39—C40	120.2 (2)
C13—C12—H12	119.8	C38—C39—H39	119.9
C11—C12—H12	119.8	C40—C39—H39	119.9
C14—C13—C12	120.1 (2)	C39—C40—C35	120.4 (2)
C14—C13—H13	119.9	C39—C40—H40	119.8
C12—C13—H13	119.9	C35—C40—H40	119.8
C13—C14—C15	119.9 (2)	C11—P1—C17	107.82 (11)
C13—C14—H14	120.0	C11—P1—C5	103.98 (11)
C15—C14—H14	120.0	C17—P1—C5	103.48 (10)
C14—C15—C16	120.4 (2)	C11—P1—Ag1	112.08 (8)
C14—C15—H15	119.8	C17—P1—Ag1	120.40 (8)
C16—C15—H15	119.8	C5—P1—Ag1	107.53 (7)
C15—C16—C11	120.2 (2)	C35—P2—C29	102.98 (11)
C15—C16—H16	119.9	C35—P2—C23	104.23 (10)
C11—C16—H16	119.9	C29—P2—C23	104.33 (11)
C22—C17—C18	119.6 (2)	C35—P2—Ag1	120.17 (7)
C22—C17—P1	119.02 (17)	C29—P2—Ag1	106.98 (7)
C18—C17—P1	121.32 (18)	C23—P2—Ag1	116.33 (7)
C19—C18—C17	120.1 (2)	O1—Ag1—P1	117.69 (5)
C19—C18—H18	120.0	O1—Ag1—P2	113.27 (5)
C17—C18—H18	120.0	P1—Ag1—P2	128.56 (2)
C20—C19—C18	120.1 (2)	C1—O1—Ag1	100.12 (14)
C20—C19—H19	120.0	C3—O3—H3	109.5
C18—C19—H19	120.0	Cl2—C1S—Cl1	112.6 (2)
C19—C20—C21	120.3 (2)	Cl2—C1S—H1S1	109.1
C19—C20—H20	119.8	Cl1—C1S—H1S1	109.1
C21—C20—H20	119.8	Cl2—C1S—H1S2	109.1

C20—C21—C22	119.9 (2)	Cl1—C1S—H1S2	109.1
C20—C21—H21	120.0	H1S1—C1S—H1S2	107.8
C22—C21—H21	120.0	Cl2B—C1SB—Cl1B	118 (2)
C17—C22—C21	119.9 (2)	Cl2B—C1SB—H1S3	107.8
C17—C22—H22	120.0	Cl1B—C1SB—H1S3	107.8
C21—C22—H22	120.0	Cl2B—C1SB—H1S4	107.8
C28—C23—C24	118.8 (2)	Cl1B—C1SB—H1S4	107.8
C28—C23—P2	122.78 (18)	H1S3—C1SB—H1S4	107.1
C24—C23—P2	118.41 (17)	Cl3—C2S—Cl4	112.3 (5)
C25—C24—C23	120.7 (2)	Cl3—C2S—H2S1	109.1
C25—C24—H24	119.7	Cl4—C2S—H2S1	109.1
C23—C24—H24	119.7	Cl3—C2S—H2S2	109.1
C26—C25—C24	120.0 (2)	Cl4—C2S—H2S2	109.1
C26—C25—H25	120.0	H2S1—C2S—H2S2	107.9
C24—C25—H25	120.0		

O1—C1—C2—C3	−147.4 (2)	C30—C29—C34—C33	1.8 (4)
O2—C1—C2—C3	35.8 (3)	P2—C29—C34—C33	−179.70 (19)
O1—C1—C2—C4	91.0 (2)	C40—C35—C36—C37	2.4 (4)
O2—C1—C2—C4	−85.7 (3)	P2—C35—C36—C37	−175.4 (2)
C1—C2—C3—O4	153.9 (2)	C35—C36—C37—C38	−1.2 (4)
C4—C2—C3—O4	−85.8 (3)	C36—C37—C38—C39	−1.1 (4)
C1—C2—C3—O3	−27.7 (3)	C37—C38—C39—C40	2.0 (4)
C4—C2—C3—O3	92.6 (2)	C38—C39—C40—C35	−0.8 (4)
C3—C2—C4—C4^{i}	71.3 (3)	C36—C35—C40—C39	−1.5 (4)
C1—C2—C4—C4^{i}	−163.5 (2)	P2—C35—C40—C39	176.25 (19)
C10—C5—C6—C7	−0.6 (3)	C16—C11—P1—C17	12.2 (2)
P1—C5—C6—C7	173.30 (18)	C12—C11—P1—C17	−168.95 (18)
C5—C6—C7—C8	0.0 (4)	C16—C11—P1—C5	−97.2 (2)
C6—C7—C8—C9	0.3 (4)	C12—C11—P1—C5	81.6 (2)
C7—C8—C9—C10	0.0 (4)	C16—C11—P1—Ag1	146.97 (19)
C8—C9—C10—C5	−0.7 (3)	C12—C11—P1—Ag1	−34.2 (2)
C6—C5—C10—C9	1.0 (3)	C22—C17—P1—C11	112.8 (2)
P1—C5—C10—C9	−172.51 (18)	C18—C17—P1—C11	−68.5 (2)
C16—C11—C12—C13	1.5 (4)	C22—C17—P1—C5	−137.45 (19)
P1—C11—C12—C13	−177.4 (2)	C18—C17—P1—C5	41.2 (2)
C11—C12—C13—C14	−1.1 (4)	C22—C17—P1—Ag1	−17.5 (2)
C12—C13—C14—C15	−0.5 (4)	C18—C17—P1—Ag1	161.23 (17)
C13—C14—C15—C16	1.7 (4)	C10—C5—P1—C11	−10.3 (2)
C14—C15—C16—C11	−1.3 (4)	C6—C5—P1—C11	176.06 (17)
C12—C11—C16—C15	−0.3 (4)	C10—C5—P1—C17	−122.87 (19)
P1—C11—C16—C15	178.5 (2)	C6—C5—P1—C17	63.48 (19)
C22—C17—C18—C19	0.2 (4)	C10—C5—P1—Ag1	108.71 (18)
P1—C17—C18—C19	−178.5 (2)	C6—C5—P1—Ag1	−64.94 (18)
C17—C18—C19—C20	−0.3 (4)	C36—C35—P2—C29	−157.1 (2)
C18—C19—C20—C21	−0.1 (4)	C40—C35—P2—C29	25.2 (2)
C19—C20—C21—C22	0.5 (4)	C36—C35—P2—C23	94.2 (2)
C18—C17—C22—C21	0.2 (4)	C40—C35—P2—C23	−83.5 (2)

P1—C17—C22—C21	178.93 (19)	C36—C35—P2—Ag1	−38.4 (2)
C20—C21—C22—C17	−0.5 (4)	C40—C35—P2—Ag1	143.93 (17)
C28—C23—C24—C25	1.0 (4)	C30—C29—P2—C35	−110.1 (2)
P2—C23—C24—C25	−178.78 (19)	C34—C29—P2—C35	71.4 (2)
C23—C24—C25—C26	−0.6 (4)	C30—C29—P2—C23	−1.5 (2)
C24—C25—C26—C27	−0.3 (4)	C34—C29—P2—C23	−179.95 (18)
C25—C26—C27—C28	0.9 (4)	C30—C29—P2—Ag1	122.33 (18)
C26—C27—C28—C23	−0.5 (4)	C34—C29—P2—Ag1	−56.13 (19)
C24—C23—C28—C27	−0.5 (4)	C28—C23—P2—C35	13.2 (2)
P2—C23—C28—C27	179.3 (2)	C24—C23—P2—C35	−167.02 (19)
C34—C29—C30—C31	−1.0 (3)	C28—C23—P2—C29	−94.5 (2)
P2—C29—C30—C31	−179.43 (18)	C24—C23—P2—C29	85.3 (2)
C29—C30—C31—C32	−0.5 (4)	C28—C23—P2—Ag1	147.93 (18)
C30—C31—C32—C33	1.3 (4)	C24—C23—P2—Ag1	−32.3 (2)
C31—C32—C33—C34	−0.5 (4)	O2—C1—O1—Ag1	4.6 (3)
C32—C33—C34—C29	−1.1 (4)	C2—C1—O1—Ag1	−171.92 (16)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2	0.84	1.79	2.525 (3)	146
C1*S*—H1*S*1···O3^{ii}	0.99	2.53	2.997 (4)	108
C1*SB*—H1*S*4···O3^{ii}	0.99	2.46	3.04 (4)	117

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