



Crystal structures of eight- and ten-membered cyclic bisanisylphosphonothioyl disulfanes and comparison with their *P*-ferrocenyl analogues

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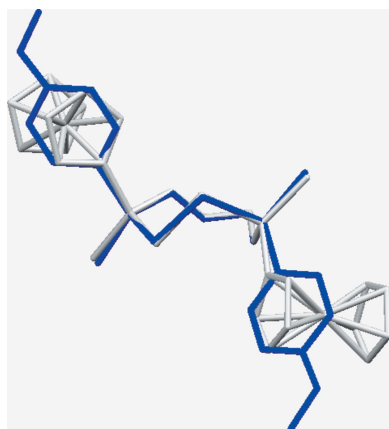
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Two new crystal structures of eight- and ten-membered cyclic bisanisylphosphonothioyl disulfanes, namely 2,5-bis(4-methoxyphenyl)-1,6,3,4,2λ⁵,5λ⁵-dioxadithiadiphosphocane-2,5-dithione, C₁₆H₁₈O₄P₂S₄, and 2,5-bis(4-methoxyphenyl)-1,6,3,4,2λ⁵,5λ⁵-dioxadithiadiphosphocane-2,5-dithione, C₁₈H₂₂O₄P₂S₄, have been determined and compared to structures of the ferrocenyl analogues. The eight-membered rings have similar conformations (TBC) but the ten-membered macrocycles are differently puckered. Structural parameters of the relevant SPSSPS motif have been analysed and are discussed in detail. Compound **1** was refined as an inversion twin and **2** was refined as a two-component rotational twin.

1. Chemical context

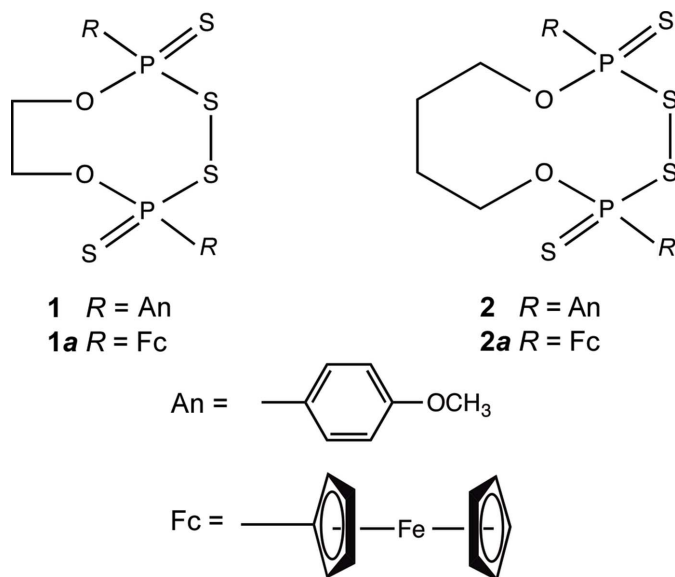
The most widely used sulfur-transfer agents for thionation of carbonyl compounds are the four-membered 2,4-dianisyl-1,3-dithiadiphosphetane disulfide dimer [AnP(μ-S)]₂ and the 2,4-diferrocenyl-1,3-dithiadiphosphetane disulfide dimer [FcP(μ-S)]₂, *i.e.* Lawesson reagent LR (Jesberger *et al.*, 2003) and ferrocenyl Lawesson reagent fLR (Foreman *et al.*, 1996). However, thiophosphine oxides (AnPSO or FcPSO) separating as cyclic trimers during thionation reactions are usually unwanted side-products. On the other hand, the corresponding alkoxyphosphinodithioic acids, *i.e.* An(RO)P(S)SH and Fc(RO)P(S)SH, obtained in a simple reaction between LR or fLR and alcohols, are of considerable interest because they form a plethora of structurally interesting chelate complexes with metal ions (van Zyl & Woollins, 2013).

The reactions between Lawesson's reagent and diols/diphenols have been successfully involved in the preparation of bis(anisylphosphonodithioic) acid derivatives and among them the unique eight-, nine- and ten-membered cyclic bisanisylphosphonothioyl disulfanes (Przychodzeń, 2004). A high-yielding formation of these medium-sized cyclic disulfanes upon oxidation of bis(anisylphosphonodithioic) acid salts by iodine proceeding without oligomeric by-products may be attributed to their fixed structure, containing the most preferred a zigzag motif of the SPSSPS unit. Slightly modified procedures with respect to the original method have recently been applied for the synthesis of related cyclic bis(ferrocenylphosphonothioyl)disulfanes, *e.g.* eight-membered **1a** (Pillay *et al.*, 2015) and ten-membered **2a** (Hua *et al.*, 2017) and their crystal structures have been determined. Here we report



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crystal structures compounds **1** and **2**, containing anisyl groups instead of the ferrocenyl moiety.



2. Structural commentary

Views of molecular structures and atom-labelling scheme for **1** and **2** are given in Figs. 1 and 2, respectively. Compound **1** crystallizes in the $P4_32_12$ space group with a half-molecule in the asymmetric unit. It follows that the molecule obeys point group symmetry described by Schoenflies symbol C_2 (or symbol 2 in international notation). The related ferrocenyl compound **1a** crystallizes in space group $C2/c$ with non-typical three and half independent molecules in the asymmetric unit ($Z = 28$), which complicates comparisons.

Compound **2** forms a monoclinic crystalline phase obeying $P2_1/c$ space-group symmetry with one molecule in the asymmetric unit and $Z = 4$. The related ferrocenyl structure **2a** crystallizes in space group $P\bar{1}$ with one molecule in the asymmetric unit.

The anisyl groups as well as the ferrocenyl groups on the two phosphorus atoms are positioned in a *trans* arrangement,

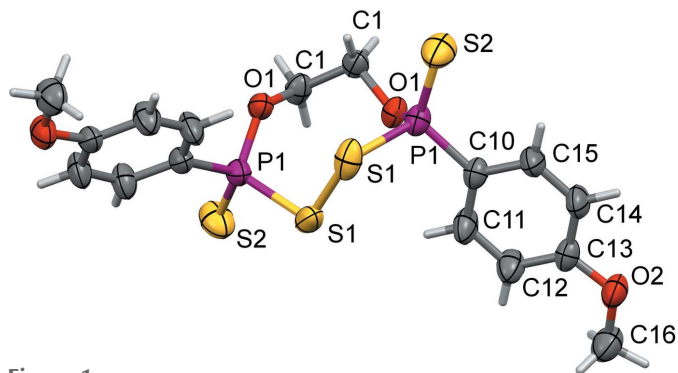


Figure 1
The molecular structure of **1**, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Symmetry-equivalent atoms are generated by the operation $(y + 1, x - 1, -z)$.

i.e. above and below the macrocycle ring plane for all compounds **1–2a**, which is also typical for all open-chain bisphosphorothioyl disulfanes studied previously (Gray *et al.*, 2004).

The S–S bond lengths have values of 2.068 (2) Å for **1**; 2.0697 (10), 2.0704 (10), 2.0685 (10), 2.0711 (15) Å for **1a**; 2.074 (3) Å for **2** and 2.0788 (9) Å for **2a**. They are longer than the typical S–S bond lengths for known diorganyl disulfanes RSSR [2.05 (3) Å]. The observed S–S bond elongation in **1–2a** may be correlated with the PSSP torsion angles (Knopik *et al.*, 1993). As expected, exocyclic P=S bond lengths (*ca* 1.92 Å) are shorter than the endocyclic P–S bonds (*ca* 2.10 Å).

All phosphorus atoms in **1–2a** adopt a distorted tetrahedral geometry, where the C–P=S angles deviated the most (116.1–118.5°) from the ideal tetrahedral angle. This is obviously due to the steric effects of the anisyl and ferrocenyl substituents. On the other hand, it is worthy to note that the O–P–S bond angles in **1–2a** (107–108°) are not distorted, probably due to minimal conformational strain present in those medium-sized heterocycles. Moreover, both the P=S and aromatic anisyl groups in **1** are almost perfectly coplanar (unlike P=S and the cyclopentadienyl ring in **1a**), which provides energetically favorable conjugation [torsion angle S2–P1–C10–C15 = -3.8 (4)° in **1** vs 35.75 (3)° for the equivalent angle in a selected representative molecule with Fe7 in **1a**]. The other related independent torsion angles in **1a** are $-31.$ (3), -33.9 (3), -27.0 (3), -28.7 (3), 34.8 (3), 35.7 (3)°, for Fe1–Fe6, respectively.

It is well recognised that PSSP torsion is a characteristic feature of all disulfanes as a class of organic compounds. The structure of **1** is the most symmetric with the lowest PSSP torsion [-93.68 (8)°] and shows only a moderate deviation from a right angle. The PSSP torsion angles in **1a** [-101.19 (4), -100.06 (4), -101.47 (4) and 99.89 (4)°] are 6–8° wider than in **1**. Notably, ten-membered disulfanes have even wider PSSP torsion angles and the difference between them is smaller, -112.89 (11) and 114.9 (4)°, for **2** and **2a**, respectively.

Only non-classical hydrogen-bonding interactions of the type C–H...X (X = O or S) can be found in the structures of **1** and **2** (Tables 1 and 2).

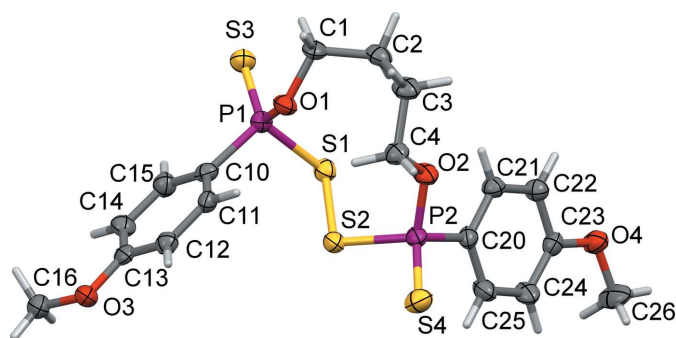


Figure 2
View of the asymmetric unit of **2**, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1–H1A \cdots O2 ⁱ	0.97	2.60	3.4843 (2)	151
C14–H14 \cdots O1 ⁱⁱ	0.93	2.55	3.4548 (2)	163

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{1}{4}$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{1}{4}$.

The transannular P \cdots P distances are very similar within the same ring size and increase, from 4.3331 (17) Å in **1** and 4.2625 (9), 4.2670 (9), 4.2652 (9) or 4.261 (1) Å (for different independent molecules in **1a**) for eight-membered rings, to 4.614 (2) in **2** and 4.604 (1) Å in **2a** for the ten-membered rings.

The conformation of the eight-membered macrocycles in **1** and **1a** was recognised by *PLATON* (Spek, 2009) as being closest to the TBC form (twist-boat chair; Evans & Boeyens, 1989; Wiberg, 2003), which is consistent with C_2 point symmetry. Fig. 3 shows the overlay of the two structures based on the best fit of the PSSP fragment. The conformation of **2** was not assigned to any border type by *PLATON*, but Fig. 4 shows the puckering in **2** and **2a** is distinctively different.

It is probably important to note that the intramolecular C4–H4B \cdots O1 hydrogen bond (Table 2) stabilizes the ten-membered ring of **2**.

3. Supramolecular features

The strongest intermolecular hydrogen-bonding interaction in **1** is between the anisyl *ortho*-hydrogen and macrocyclic O1 atoms and links the molecules into a diamondoid network.

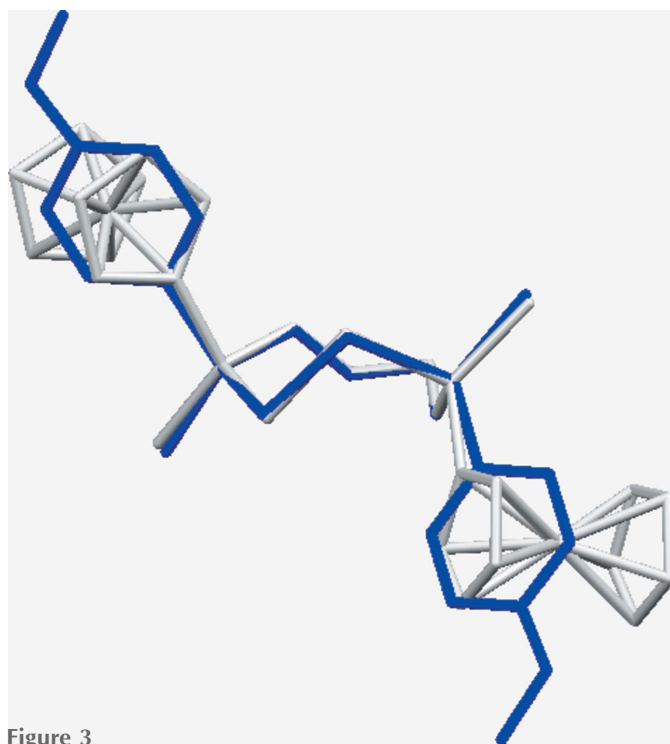


Figure 3
 Superimposition of eight-membered diphosphocanes **1** (blue) and **1a** (grey) based on the best PSSP fragment fit.

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

C_g is the centroid of the C20–C25 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1–H1B \cdots S3	0.99	2.81	3.3883 (2)	118
C4–H4B \cdots O1	0.99	2.48	3.1308 (2)	123
C4–H4B \cdots O4 ⁱ	0.99	2.56	3.2708 (2)	128
C11–H11 \cdots O4 ⁱ	0.95	2.62	3.4951 (3)	154
C24–H24 \cdots O3 ⁱⁱ	0.95	2.51	3.4240 (3)	162
C16–H16A \cdots Cg ⁱⁱⁱ	0.98	2.62	3.454 (8)	143

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

There are no ring-stacking interactions since the shortest centroid–centroid distance is 5.0965 (3) Å. The anisyl substituents may have inhibited this kind of interaction.

Intermolecular interactions in **2** are mainly based on the anisyl methoxyl CH₃O oxygen atoms O3 and O4 and the P=S sulfur atom S3 as acceptors. Hydrogen-bond donors are the anisyl *ortho*-hydrogen atoms or methylene hydrogen atoms. Moreover, some C–H \cdots π interactions may play some role in the system, e.g. C16–H16A \cdots ring(C20–C25), see Fig. 5. Again, the stacking interactions are weak since the closest intercentroid distance is equal to 4.9213 (4) Å.

4. Database survey

Bisphosphonothioyl disulfanes represent a rather rare class of compounds (CSD Version 5.28, updated to Nov. 2016; Groom *et al.*, 2016). Only three structures of cyclic bisphosphonothioyl disulfanes can be found in the database, HUGXAK, HUXEO and HUGXIS (ferrocenyl derivatives; Pillay *et al.*, 2015) and four more will be available there soon (Hua *et al.*, 2017). For structures of acyclic bisphosphonothioyl disulfanes

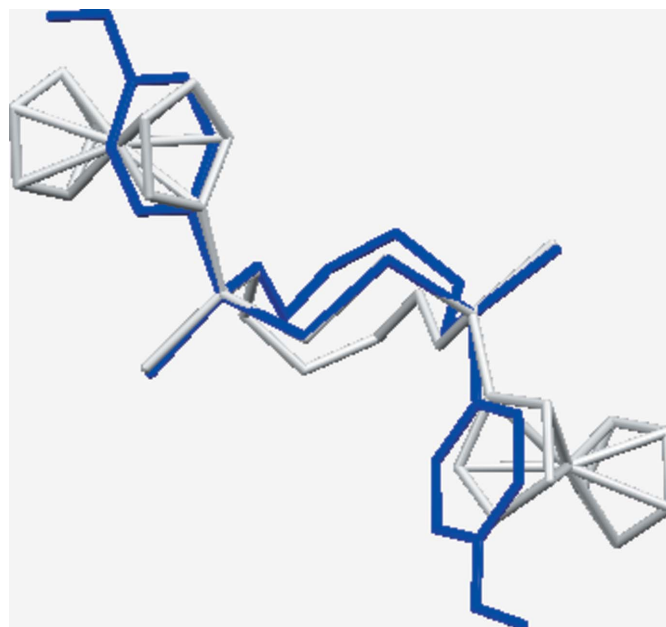


Figure 4
 Overlay of ten-membered diphosphocanes **2** (blue) and **2a** (grey) based on the best PSSP fragment fit.

Table 3
Experimental details.

	1	2
Crystal data		
Chemical formula	C ₁₆ H ₁₈ O ₄ P ₂ S ₄	C ₁₈ H ₂₂ O ₄ P ₂ S ₄
<i>M_r</i>	464.48	492.53
Crystal system, space group	Tetragonal, <i>P</i> 4 ₃ 2 ₁ 2	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	296	120
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.2415 (3), 7.2415 (3), 39.516 (2)	9.4262 (6), 13.3761 (8), 17.7998 (13)
α , β , γ (°)	90, 90, 90	90, 90.068 (7), 90
<i>V</i> (Å ³)	2072.2 (2)	2244.3 (3)
<i>Z</i>	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.63	0.59
Crystal size (mm)	0.44 × 0.42 × 0.03	0.21 × 0.20 × 0.14
Data collection		
Diffractometer	Oxford Diffraction KM-4 CCD	Oxford Diffraction KM-4 CCD
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2011)	Analytical [<i>CrysAlis PRO</i> (Agilent, 2011) based on expressions derived by Clark & Reid (1995)]
<i>T_{min}</i> – <i>T_{max}</i>	0.689, 0.98	0.893, 0.929
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	14211, 2019, 1839	9496, 4047, 3309
<i>R_{int}</i>	0.036	0.051
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.617	0.606
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.038, 0.092, 1.09	0.082, 0.241, 1.05
No. of reflections	2019	4047
No. of parameters	120	256
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.33, –0.22	2.27, –0.84
Absolute structure	Refined as an inversion twin	–
Absolute structure parameter	0.45 (17)	–

Computer programs: *CrysAlis PRO* (Agilent, 2011), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2008), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

see: FATTEA, FATTIE, FATVEC (Gray *et al.*, 2004), YESDIY (Łopusiński *et al.*, 1991), SIZHUF (Przychodzeń & Chojnacki, 2008) and WAYMEO (Knopik *et al.*, 1993).

5. Synthesis and crystallization

Eight- and ten-membered cyclic bisanisylphosphonothioyl disulfanes **1** and **2** were prepared using previously reported procedure (Przychodzeń, 2004). Compound **1** was fully spectroscopically characterized in that paper. Disulfane **2** is quite new, so all available spectroscopic data are given below. Both **1**

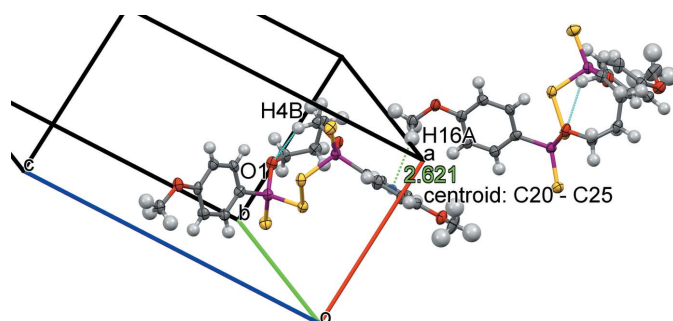


Figure 5
C–H... π interaction and internal C–H...O hydrogen bonding in the ten-membered ring of **2**.

and **2** gave good quality colourless crystals after crystallization from ethyl acetate–cyclohexane (1:2 *v/v*) solvent system.

2,5-Bis(4-methoxyphenyl)-1,6,3,4,2,5-dioxadithiadiphosphocane 2,5-dithione, **1**

M.p. 441–443 K.

2,5-Bis(4-methoxyphenyl)-1,6,3,4,2,5-dioxadithiadiphosphocane 2,5-dithione, **2**

Yield: 65%, m.p. 415–417 K.

¹H NMR (CDCl₃): 2.20 (*m*, 2H, OCH₂CH₂), 2.25 (*m*, 2H, OCH₂CH₂), 3.89 (*s*, 6H, OCH₃), 4.37 (*dddd*, ³*J*_{HH} = 11.6 Hz, ²*J*_{HH} = 10.4 Hz, ³*J*_{HP} = 5.4 Hz, ³*J*_{HH} = 2.2 Hz, 2H, OCH_AH_B), 4.89 (*ddt*, ²*J*_{HH} = 10.4 Hz, ³*J*_{HP} = 9.3 Hz, ³*J*_{HH} = 3.5 Hz, 2H, OCH_AH_B), 7.01 (*dd*, ³*J*_{HH} = 8.8 Hz, ⁴*J*_{HP} = 3.9 Hz, 4H, H_{meta}), 7.87 (*dd*, ³*J*_{HP} = 14.2 Hz, ³*J*_{HH} = 8.8 Hz, 4H, H_{ortho}).

¹³C NMR: 27.21 (*d*, *J* = 6.9 Hz), 55.46 (*s*), 67.08 (*d*, *J* = 6.3 Hz), 114.03 (*d*, *J* = 17.5 Hz), 125.41 (*d*, *J* = 134 Hz), 132.89 (*d*, *J* = 14.5 Hz), 163.09 (*s*).

³¹P{¹H} NMR (CDCl₃): 89.19 (³*J*_{PP} = 4 Hz)

MS calculated for C₁₈H₂₂O₄P₂S₄: 492.0. Found: 492.9 [*M*+H]⁺.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Structure **1** was refined as an

inversion twin with contribution of the second domain equal to 0.45 (17). This explains the ambiguous Flack parameter and is not surprising since we started from achiral substrates. Structure **2** was refined as a two-component rotational twin with twin law: $\{\bar{1} 0 0, 0 \bar{1} 0, 0 0 1\}$ and BASF = 0.767 (3). Relatively high residual electron-density peaks in **2** ($Q1-Q3$ ca $2e \text{ \AA}^{-3}$), which are close to sulfur atoms (0.58 Å from S4, 0.49 Å from S2, 0.49 Å from S1), may stem from conformational flexibility of the ring. Note: the structure of **1** was determined at room temperature (due to a failure of our CryoStream unit) not at 120 K as for **2** but we believe it did not influence the qualitative conclusions drawn from the results.

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supporting information

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Crystal structures of eight- and ten-membered cyclic bisanisylphosphonothioyl disulfanes and comparison with their *P*-ferrocenyl analogues

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Computing details

For both structures, data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2008). Software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010) for (1); *WinGX* (Farrugia, 2012) for (2).

2,5-Bis(4-methoxyphenyl)-1,6,3,4,2λ⁵,5λ⁵-dioxadithiadiphosphocane-2,5-dithione (1)

Crystal data

C₁₆H₁₈O₄P₂S₄

M_r = 464.48

Tetragonal, *P*4₃2₁2

Hall symbol: P 4nw 2abw

a = 7.2415 (3) Å

c = 39.516 (2) Å

V = 2072.2 (2) Å³

Z = 4

F(000) = 960

D_x = 1.489 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 6759 reflections

θ = 2.1–32.4°

μ = 0.63 mm⁻¹

T = 296 K

Plate, colourless

0.44 × 0.42 × 0.03 mm

Data collection

Oxford Diffraction KM-4 CCD
diffractometer

Detector resolution: 8.1883 pixels mm⁻¹

ω scans, 0.40 deg width

Absorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2011)

T_{min} = 0.689, *T_{max}* = 0.98

14211 measured reflections

2019 independent reflections

1839 reflections with *I* > 2σ(*I*)

R_{int} = 0.036

θ_{max} = 26.0°, θ_{min} = 2.9°

h = -8→8

k = -8→8

l = -48→38

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.038

wR(*F*²) = 0.092

S = 1.09

2019 reflections

120 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0514*P*)² + 0.5163*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Refined as an inversion twin
 Absolute structure parameter: 0.45 (17)

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.

Refinement. Refined as a 2-component inversion twin.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.93887 (12)	0.21832 (13)	0.04117 (2)	0.0424 (2)
S1	0.84270 (13)	0.04364 (17)	0.00256 (2)	0.0584 (3)
S2	0.79007 (17)	0.43697 (17)	0.03709 (3)	0.0715 (4)
O1	1.1556 (3)	0.2344 (3)	0.03669 (5)	0.0424 (6)
O2	0.8974 (4)	-0.1523 (4)	0.17469 (6)	0.0552 (7)
C1	1.2424 (5)	0.3553 (5)	0.01202 (8)	0.0450 (8)
H1A	1.3209	0.4439	0.0235	0.054*
H1B	1.1485	0.4228	-0.0004	0.054*
C10	0.9277 (5)	0.1007 (5)	0.08087 (8)	0.0412 (7)
C11	1.0238 (5)	-0.0610 (6)	0.08670 (9)	0.0576 (10)
H11	1.0949	-0.1113	0.0694	0.069*
C12	1.0170 (6)	-0.1496 (6)	0.11749 (9)	0.0584 (10)
H12	1.082	-0.2589	0.1207	0.07*
C13	0.9144 (5)	-0.0768 (5)	0.14352 (7)	0.0434 (8)
C14	0.8206 (6)	0.0864 (6)	0.13813 (9)	0.0555 (10)
H14	0.7529	0.1384	0.1557	0.067*
C15	0.8252 (6)	0.1736 (5)	0.10720 (9)	0.0526 (9)
H15	0.759	0.2823	0.104	0.063*
C16	1.0028 (6)	-0.3130 (6)	0.18232 (11)	0.0672 (12)
H16A	0.9613	-0.4138	0.1685	0.101*
H16B	0.9871	-0.3442	0.2058	0.101*
H16C	1.1309	-0.2894	0.1779	0.101*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0373 (4)	0.0559 (5)	0.0340 (4)	0.0025 (4)	0.0018 (3)	0.0025 (4)
S1	0.0423 (5)	0.0932 (8)	0.0396 (5)	-0.0164 (5)	-0.0034 (4)	-0.0029 (5)
S2	0.0711 (7)	0.0755 (7)	0.0678 (7)	0.0285 (6)	0.0106 (5)	0.0156 (6)
O1	0.0403 (12)	0.0552 (14)	0.0318 (11)	-0.0057 (11)	-0.0003 (9)	0.0010 (10)
O2	0.0598 (16)	0.0694 (17)	0.0363 (13)	0.0070 (13)	0.0086 (11)	0.0069 (11)
C1	0.054 (2)	0.0427 (18)	0.0381 (17)	-0.0144 (16)	0.0004 (15)	-0.0036 (14)
C10	0.0358 (16)	0.054 (2)	0.0336 (15)	0.0004 (15)	0.0034 (14)	0.0006 (14)
C11	0.056 (2)	0.076 (3)	0.0400 (18)	0.026 (2)	0.0145 (16)	0.0035 (18)
C12	0.062 (2)	0.069 (2)	0.0443 (19)	0.0260 (19)	0.0073 (17)	0.0058 (17)

C13	0.0363 (18)	0.061 (2)	0.0329 (15)	-0.0035 (15)	0.0030 (13)	0.0010 (15)
C14	0.066 (3)	0.061 (2)	0.0402 (18)	0.0157 (19)	0.0169 (16)	-0.0040 (17)
C15	0.065 (3)	0.050 (2)	0.0424 (19)	0.0145 (18)	0.0131 (17)	-0.0021 (16)
C16	0.073 (3)	0.078 (3)	0.051 (2)	0.011 (2)	0.0064 (19)	0.016 (2)

Geometric parameters (Å, °)

P1—O1	1.584 (2)	C10—C15	1.383 (5)
P1—C10	1.787 (3)	C11—C12	1.376 (5)
P1—S2	1.9220 (14)	C11—H11	0.93
P1—S1	2.1006 (13)	C12—C13	1.374 (5)
S1—S1 ⁱ	2.068 (2)	C12—H12	0.93
O1—C1	1.453 (4)	C13—C14	1.380 (5)
O2—C13	1.353 (4)	C14—C15	1.376 (5)
O2—C16	1.424 (5)	C14—H14	0.93
C1—C1 ⁱ	1.496 (7)	C15—H15	0.93
C1—H1A	0.97	C16—H16A	0.96
C1—H1B	0.97	C16—H16B	0.96
C10—C11	1.382 (5)	C16—H16C	0.96
O1—P1—C10	100.26 (14)	C10—C11—H11	119.2
O1—P1—S2	119.05 (11)	C13—C12—C11	120.2 (4)
C10—P1—S2	116.15 (13)	C13—C12—H12	119.9
O1—P1—S1	106.97 (9)	C11—C12—H12	119.9
C10—P1—S1	109.61 (12)	O2—C13—C12	125.1 (3)
S2—P1—S1	104.44 (6)	O2—C13—C14	116.2 (3)
S1 ⁱ —S1—P1	105.17 (6)	C12—C13—C14	118.6 (3)
C1—O1—P1	123.2 (2)	C15—C14—C13	121.2 (3)
C13—O2—C16	118.3 (3)	C15—C14—H14	119.4
O1—C1—C1 ⁱ	109.5 (3)	C13—C14—H14	119.4
O1—C1—H1A	109.8	C14—C15—C10	120.4 (3)
C1 ⁱ —C1—H1A	109.8	C14—C15—H15	119.8
O1—C1—H1B	109.8	C10—C15—H15	119.8
C1 ⁱ —C1—H1B	109.8	O2—C16—H16A	109.5
H1A—C1—H1B	108.2	O2—C16—H16B	109.5
C11—C10—C15	117.9 (3)	H16A—C16—H16B	109.5
C11—C10—P1	121.8 (3)	O2—C16—H16C	109.5
C15—C10—P1	120.2 (3)	H16A—C16—H16C	109.5
C12—C11—C10	121.6 (3)	H16B—C16—H16C	109.5
C12—C11—H11	119.2		
C10—P1—O1—C1	-165.8 (2)	P1—C10—C11—C12	179.0 (3)
S2—P1—O1—C1	-38.0 (3)	C10—C11—C12—C13	-0.6 (7)
S1—P1—O1—C1	79.8 (2)	C16—O2—C13—C12	4.4 (6)
P1—O1—C1—C1 ⁱ	-119.6 (3)	C16—O2—C13—C14	-175.3 (4)
O1—P1—C10—C11	-52.2 (3)	C11—C12—C13—O2	179.8 (4)
S2—P1—C10—C11	178.1 (3)	C11—C12—C13—C14	-0.5 (6)
S1—P1—C10—C11	60.1 (3)	O2—C13—C14—C15	-178.8 (4)

O1—P1—C10—C15	125.9 (3)	C12—C13—C14—C15	1.5 (6)
S2—P1—C10—C15	-3.8 (4)	C13—C14—C15—C10	-1.2 (7)
S1—P1—C10—C15	-121.9 (3)	C11—C10—C15—C14	0.1 (6)
C15—C10—C11—C12	0.9 (6)	P1—C10—C15—C14	-178.1 (3)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1A \cdots O2 ⁱⁱ	0.97	2.60	3.4843 (2)	151
C14—H14 \cdots O1 ⁱⁱⁱ	0.93	2.55	3.4548 (2)	163

Symmetry codes: (ii) $x+1/2, -y+1/2, -z+1/4$; (iii) $x-1/2, -y+1/2, -z+1/4$.

2,5-Bis(4-methoxyphenyl)-1,6,3,4,2 λ^5 ,5 λ^5 -dioxadithiadiphospecane-2,5-dithione (2)

Crystal data

$C_{18}H_{22}O_4P_2S_4$

$M_r = 492.53$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.4262$ (6) \AA

$b = 13.3761$ (8) \AA

$c = 17.7998$ (13) \AA

$\beta = 90.068$ (7) $^\circ$

$V = 2244.3$ (3) \AA^3

$Z = 4$

$F(000) = 1024$

$D_x = 1.458$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ \AA

Cell parameters from 5521 reflections

$\theta = 1.9\text{--}28.8^\circ$

$\mu = 0.59$ mm^{-1}

$T = 120$ K

Prism, colourless

$0.21 \times 0.20 \times 0.14$ mm

Data collection

Oxford Diffraction KM-4 CCD
diffractometer

Graphite monochromator

Detector resolution: 8.19 pixels mm^{-1}

ω scans

Absorption correction: analytical

[CrysAlis PRO (Agilent, 2011) based on
expressions derived by Clark & Reid (1995)]

$T_{\min} = 0.893$, $T_{\max} = 0.929$

9496 measured reflections

4047 independent reflections

3309 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$

$\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 1.9^\circ$

$h = -6 \rightarrow 11$

$k = -13 \rightarrow 16$

$l = -21 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.082$

$wR(F^2) = 0.241$

$S = 1.05$

4047 reflections

256 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1828P)^2 + 0.8642P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 2.27$ e \AA^{-3}

$\Delta\rho_{\min} = -0.84$ e \AA^{-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.

Refinement. Refined as a 2-component twin.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.34560 (19)	0.44281 (13)	0.17369 (10)	0.0294 (4)
P2	0.74857 (19)	0.25294 (12)	0.13780 (10)	0.0288 (4)
S1	0.40975 (19)	0.31382 (12)	0.11377 (10)	0.0318 (4)
S2	0.5483 (2)	0.24408 (12)	0.18767 (10)	0.0325 (4)
S3	0.1939 (2)	0.49814 (14)	0.11372 (11)	0.0384 (5)
S4	0.8724 (2)	0.19798 (14)	0.21394 (12)	0.0413 (5)
O1	0.4806 (6)	0.5097 (3)	0.1892 (3)	0.0342 (11)
O2	0.7721 (6)	0.3641 (3)	0.1101 (3)	0.0348 (11)
O3	0.2227 (5)	0.2847 (4)	0.4774 (3)	0.0344 (11)
O4	0.7677 (6)	0.0419 (4)	-0.1545 (3)	0.0377 (12)
C1	0.5242 (8)	0.5920 (5)	0.1389 (4)	0.0356 (16)
H1A	0.5618	0.6477	0.1696	0.043*
H1B	0.4399	0.6169	0.1114	0.043*
C2	0.6350 (8)	0.5604 (5)	0.0833 (4)	0.0357 (16)
H2A	0.6429	0.613	0.0444	0.043*
H2B	0.6019	0.4987	0.0581	0.043*
C3	0.7811 (9)	0.5412 (5)	0.1152 (5)	0.0392 (17)
H3A	0.8083	0.5991	0.1467	0.047*
H3B	0.8493	0.5376	0.073	0.047*
C4	0.7962 (8)	0.4456 (5)	0.1626 (4)	0.0335 (15)
H4A	0.8923	0.441	0.1849	0.04*
H4B	0.7252	0.4443	0.2035	0.04*
C10	0.3054 (7)	0.3994 (5)	0.2668 (4)	0.0303 (15)
C11	0.4091 (7)	0.3973 (5)	0.3230 (4)	0.0288 (14)
H11	0.501	0.4233	0.3132	0.035*
C12	0.3784 (7)	0.3576 (5)	0.3929 (4)	0.0311 (15)
H12	0.4494	0.3549	0.4307	0.037*
C13	0.2422 (8)	0.3216 (5)	0.4072 (4)	0.0302 (15)
C14	0.1390 (8)	0.3237 (5)	0.3512 (4)	0.0343 (16)
H14	0.0465	0.2985	0.3607	0.041*
C15	0.1728 (8)	0.3631 (5)	0.2814 (4)	0.0341 (15)
H15	0.1026	0.3649	0.2431	0.041*
C16	0.0843 (8)	0.2559 (5)	0.4988 (5)	0.0377 (17)
H16A	0.0202	0.3133	0.4942	0.057*
H16B	0.0854	0.2327	0.5511	0.057*
H16C	0.0513	0.2017	0.4661	0.057*
C20	0.7453 (7)	0.1893 (5)	0.0491 (4)	0.0291 (14)
C21	0.7321 (8)	0.2409 (5)	-0.0182 (4)	0.0302 (15)

H21	0.7173	0.3112	-0.018	0.036*
C22	0.7406 (8)	0.1894 (5)	-0.0860 (4)	0.0320 (15)
H22	0.7333	0.2242	-0.1323	0.038*
C23	0.7602 (7)	0.0850 (5)	-0.0852 (4)	0.0302 (14)
C24	0.7703 (9)	0.0347 (5)	-0.0185 (4)	0.0372 (17)
H24	0.7819	-0.0359	-0.0183	0.045*
C25	0.7636 (9)	0.0867 (5)	0.0488 (4)	0.0368 (16)
H25	0.7716	0.0517	0.0951	0.044*
C26	0.7930 (13)	-0.0639 (6)	-0.1555 (5)	0.058 (3)
H26A	0.8789	-0.0789	-0.1266	0.087*
H26B	0.8055	-0.0863	-0.2075	0.087*
H26C	0.7119	-0.0986	-0.1331	0.087*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0321 (9)	0.0271 (9)	0.0289 (9)	0.0023 (7)	0.0031 (7)	0.0019 (7)
P2	0.0358 (9)	0.0216 (8)	0.0292 (10)	0.0003 (7)	0.0061 (8)	-0.0005 (6)
S1	0.0380 (9)	0.0291 (8)	0.0285 (9)	-0.0009 (7)	0.0034 (8)	-0.0020 (7)
S2	0.0407 (9)	0.0271 (8)	0.0299 (9)	0.0032 (7)	0.0086 (8)	0.0039 (7)
S3	0.0399 (9)	0.0405 (10)	0.0348 (10)	0.0079 (7)	0.0008 (8)	0.0061 (8)
S4	0.0486 (11)	0.0347 (10)	0.0404 (11)	0.0097 (8)	-0.0055 (9)	-0.0018 (8)
O1	0.047 (3)	0.025 (2)	0.030 (3)	-0.001 (2)	0.007 (2)	0.005 (2)
O2	0.051 (3)	0.021 (2)	0.033 (3)	-0.005 (2)	0.005 (2)	-0.002 (2)
O3	0.040 (3)	0.031 (2)	0.032 (3)	0.004 (2)	0.008 (2)	0.007 (2)
O4	0.051 (3)	0.030 (3)	0.032 (3)	0.008 (2)	0.000 (2)	-0.003 (2)
C1	0.043 (4)	0.021 (3)	0.043 (4)	-0.001 (3)	0.010 (3)	0.008 (3)
C2	0.047 (4)	0.026 (3)	0.034 (4)	-0.007 (3)	0.002 (3)	0.006 (3)
C3	0.052 (4)	0.023 (3)	0.042 (4)	-0.007 (3)	0.001 (4)	0.000 (3)
C4	0.040 (4)	0.025 (3)	0.035 (4)	-0.001 (3)	0.001 (3)	-0.002 (3)
C10	0.036 (3)	0.022 (3)	0.032 (4)	0.006 (3)	-0.003 (3)	0.001 (3)
C11	0.025 (3)	0.027 (3)	0.035 (4)	0.003 (2)	0.008 (3)	0.000 (3)
C12	0.033 (4)	0.029 (3)	0.031 (4)	0.003 (3)	0.001 (3)	-0.003 (3)
C13	0.040 (4)	0.020 (3)	0.030 (4)	0.008 (3)	0.007 (3)	-0.003 (3)
C14	0.038 (4)	0.027 (3)	0.038 (4)	-0.004 (3)	0.005 (3)	0.004 (3)
C15	0.034 (4)	0.037 (4)	0.032 (4)	0.003 (3)	0.002 (3)	-0.001 (3)
C16	0.042 (4)	0.033 (4)	0.038 (4)	0.006 (3)	0.010 (3)	0.001 (3)
C20	0.030 (3)	0.026 (3)	0.031 (4)	-0.002 (3)	0.009 (3)	-0.002 (3)
C21	0.037 (4)	0.024 (3)	0.030 (4)	0.001 (3)	0.001 (3)	0.000 (3)
C22	0.035 (4)	0.028 (3)	0.033 (4)	0.000 (3)	-0.001 (3)	0.001 (3)
C23	0.028 (3)	0.029 (3)	0.034 (4)	0.003 (3)	0.003 (3)	-0.005 (3)
C24	0.057 (5)	0.020 (3)	0.035 (4)	-0.005 (3)	0.009 (4)	-0.003 (3)
C25	0.051 (4)	0.029 (4)	0.031 (4)	-0.003 (3)	0.009 (3)	0.004 (3)
C26	0.106 (8)	0.030 (4)	0.037 (5)	0.024 (5)	0.000 (5)	-0.010 (3)

Geometric parameters (Å, °)

P1—O1	1.580 (5)	C10—C15	1.366 (10)
P1—C10	1.798 (7)	C10—C11	1.398 (9)
P1—S3	1.931 (3)	C11—C12	1.383 (10)
P1—S1	2.117 (2)	C11—H11	0.95
P2—O2	1.582 (5)	C12—C13	1.395 (10)
P2—C20	1.794 (7)	C12—H12	0.95
P2—S4	1.933 (3)	C13—C14	1.393 (10)
P2—S2	2.091 (3)	C14—C15	1.387 (10)
S1—S2	2.074 (3)	C14—H14	0.95
O1—C1	1.477 (8)	C15—H15	0.95
O2—C4	1.453 (8)	C16—H16A	0.98
O3—C13	1.357 (8)	C16—H16B	0.98
O3—C16	1.413 (9)	C16—H16C	0.98
O4—C23	1.364 (8)	C20—C25	1.383 (10)
O4—C26	1.435 (9)	C20—C21	1.389 (10)
C1—C2	1.501 (11)	C21—C22	1.391 (10)
C1—H1A	0.99	C21—H21	0.95
C1—H1B	0.99	C22—C23	1.408 (10)
C2—C3	1.511 (11)	C22—H22	0.95
C2—H2A	0.99	C23—C24	1.368 (10)
C2—H2B	0.99	C24—C25	1.388 (10)
C3—C4	1.539 (10)	C24—H24	0.95
C3—H3A	0.99	C25—H25	0.95
C3—H3B	0.99	C26—H26A	0.98
C4—H4A	0.99	C26—H26B	0.98
C4—H4B	0.99	C26—H26C	0.98
O1—P1—C10	101.1 (3)	C12—C11—C10	120.3 (6)
O1—P1—S3	118.4 (2)	C12—C11—H11	119.9
C10—P1—S3	118.5 (2)	C10—C11—H11	119.9
O1—P1—S1	108.6 (2)	C11—C12—C13	119.4 (6)
C10—P1—S1	105.2 (2)	C11—C12—H12	120.3
S3—P1—S1	104.22 (11)	C13—C12—H12	120.3
O2—P2—C20	100.0 (3)	O3—C13—C14	124.9 (7)
O2—P2—S4	119.4 (2)	O3—C13—C12	114.8 (6)
C20—P2—S4	116.5 (2)	C14—C13—C12	120.3 (7)
O2—P2—S2	108.2 (2)	C15—C14—C13	119.2 (7)
C20—P2—S2	109.4 (2)	C15—C14—H14	120.4
S4—P2—S2	103.04 (11)	C13—C14—H14	120.4
S2—S1—P1	103.13 (10)	C10—C15—C14	121.1 (7)
S1—S2—P2	105.87 (10)	C10—C15—H15	119.5
C1—O1—P1	122.7 (5)	C14—C15—H15	119.5
C4—O2—P2	121.7 (4)	O3—C16—H16A	109.5
C13—O3—C16	118.3 (6)	O3—C16—H16B	109.5
C23—O4—C26	115.8 (6)	H16A—C16—H16B	109.5
O1—C1—C2	112.6 (6)	O3—C16—H16C	109.5

O1—C1—H1A	109.1	H16A—C16—H16C	109.5
C2—C1—H1A	109.1	H16B—C16—H16C	109.5
O1—C1—H1B	109.1	C25—C20—C21	120.1 (7)
C2—C1—H1B	109.1	C25—C20—P2	118.2 (6)
H1A—C1—H1B	107.8	C21—C20—P2	121.7 (5)
C1—C2—C3	115.8 (6)	C20—C21—C22	119.8 (6)
C1—C2—H2A	108.3	C20—C21—H21	120.1
C3—C2—H2A	108.3	C22—C21—H21	120.1
C1—C2—H2B	108.3	C21—C22—C23	119.4 (6)
C3—C2—H2B	108.3	C21—C22—H22	120.3
H2A—C2—H2B	107.4	C23—C22—H22	120.3
C2—C3—C4	115.5 (6)	O4—C23—C24	125.0 (6)
C2—C3—H3A	108.4	O4—C23—C22	114.6 (6)
C4—C3—H3A	108.4	C24—C23—C22	120.3 (6)
C2—C3—H3B	108.4	C23—C24—C25	120.0 (6)
C4—C3—H3B	108.4	C23—C24—H24	120
H3A—C3—H3B	107.5	C25—C24—H24	120
O2—C4—C3	104.8 (6)	C20—C25—C24	120.4 (7)
O2—C4—H4A	110.8	C20—C25—H25	119.8
C3—C4—H4A	110.8	C24—C25—H25	119.8
O2—C4—H4B	110.8	O4—C26—H26A	109.5
C3—C4—H4B	110.8	O4—C26—H26B	109.5
H4A—C4—H4B	108.9	H26A—C26—H26B	109.5
C15—C10—C11	119.8 (7)	O4—C26—H26C	109.5
C15—C10—P1	118.9 (5)	H26A—C26—H26C	109.5
C11—C10—P1	121.2 (5)	H26B—C26—H26C	109.5
C10—P1—O1—C1	-156.0 (5)	O3—C13—C14—C15	-179.6 (6)
S3—P1—O1—C1	-24.8 (6)	C12—C13—C14—C15	-0.7 (10)
S1—P1—O1—C1	93.6 (5)	C11—C10—C15—C14	-0.2 (10)
C20—P2—O2—C4	-173.9 (5)	P1—C10—C15—C14	176.4 (5)
S4—P2—O2—C4	-45.6 (6)	C13—C14—C15—C10	0.2 (11)
S2—P2—O2—C4	71.7 (5)	O2—P2—C20—C25	163.4 (6)
P1—O1—C1—C2	-95.9 (7)	S4—P2—C20—C25	33.1 (7)
O1—C1—C2—C3	-71.3 (8)	S2—P2—C20—C25	-83.2 (6)
C1—C2—C3—C4	72.0 (9)	O2—P2—C20—C21	-13.4 (6)
P2—O2—C4—C3	-168.1 (5)	S4—P2—C20—C21	-143.7 (5)
C2—C3—C4—O2	65.1 (8)	S2—P2—C20—C21	100.0 (6)
O1—P1—C10—C15	161.1 (5)	C25—C20—C21—C22	-1.5 (11)
S3—P1—C10—C15	29.9 (6)	P2—C20—C21—C22	175.3 (6)
S1—P1—C10—C15	-86.0 (6)	C20—C21—C22—C23	1.1 (11)
O1—P1—C10—C11	-22.4 (6)	C26—O4—C23—C24	-2.2 (11)
S3—P1—C10—C11	-153.5 (5)	C26—O4—C23—C22	177.7 (8)
S1—P1—C10—C11	90.6 (5)	C21—C22—C23—O4	-179.7 (6)
C15—C10—C11—C12	0.8 (10)	C21—C22—C23—C24	0.2 (11)
P1—C10—C11—C12	-175.8 (5)	O4—C23—C24—C25	178.9 (7)
C10—C11—C12—C13	-1.3 (10)	C22—C23—C24—C25	-1.0 (11)
C16—O3—C13—C14	-7.2 (9)	C21—C20—C25—C24	0.6 (12)

C16—O3—C13—C12	173.8 (6)	P2—C20—C25—C24	-176.2 (6)
C11—C12—C13—O3	-179.7 (6)	C23—C24—C25—C20	0.6 (12)
C11—C12—C13—C14	1.3 (10)		

Hydrogen-bond geometry (Å, °)

Cg is the centroid of the C20–C25 ring.

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C1—H1 <i>B</i> \cdots S3	0.99	2.81	3.3883 (2)	118
C4—H4 <i>B</i> \cdots O1	0.99	2.48	3.1308 (2)	123
C4—H4 <i>B</i> \cdots O4 ⁱ	0.99	2.56	3.2708 (2)	128
C11—H11 \cdots O4 ⁱ	0.95	2.62	3.4951 (3)	154
C24—H24 \cdots O3 ⁱⁱ	0.95	2.51	3.4240 (3)	162
C16—H16 <i>A</i> \cdots Cg ⁱⁱⁱ	0.98	2.62	3.454 (8)	143

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